-FINAL-

2014 UPDATE

UNIFORM FEDERAL POLICY QUALITY ASSURANCE PROJECT PLAN

FORMER GRIFFISS AIR FORCE BASE ROME, NEW YORK

Prepared for:

Air Force Civil Engineer Center
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Acronyms and Abbreviations

%D	Percent Difference
AECOM	AECOM Technical Services
AFB	Air Force Base
AFCEC	Air Force Civil Engineer Center
AFCEE	Air Force Center for Engineering and the Environment
AOC	Area of Concern
AOI	Area of Interest
BRAC	Base Realignment and Closure
BOD	Biological Oxygen Demand
°C	degrees Celsius
CA	Corrective Action
CAPE	CAPE, Inc.
CAS	Chemical Abstracts Service
CCC	Calibration Check Compounds
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CV	Calibration Verification
CFR	Code of Federal Regulations
CLLE	Continuous Liquid/Liquid Extraction
COC	Chain of Custody
COD	Chemical Oxygen Demand
DL	Detection Limit
DoD	Department of Defense
DQI	Data Quality Indicator
DQO	Data Quality Objective
EICP	Extracted ion current profile
ERPIMS	Environmental Restoration Program Information Management System
EXC	Capital Investment Execution Division
FDA	Fire Demonstration Area
FFA	Federal Facilities Agreement
FPM	FPM Remediations, Inc.
FPTA	Fire Protection Training Area
GC	Gas Chromatography
GC/MS	Gas Chromatography and Mass Spectrometer
GLDC	Griffiss Local Development Corporation
HCl	Hydrochloric Acid

H&S	Health and Safety
ICV	Initial Calibration verification
ID	Identification
IRP	Installation Restoration Program
IS	Internal Standard
kg	kilogram(s)
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
LTM	Long Term Monitoring
LUC/IC	Land use Control/Institutional Control
mg	milligram(s)
mL	milliliter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
N/A	Not Available
No.	Number
NPL	National Priorities List
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
O&M	Operation and Maintenance
oz	ounce
PAH	Polynuclear Aromatic Hydrocarbon
PCBs	Polychlorinated Biphenyls
PID	photoionization detector
PGM	Program Manager
pH	Measure of the acidity or basicity of a solution (pH = -log[hydrogen ion concentration])
PM	Project Manager
PMP	Project Management Plan
POC	point of contact
POP	Period of Performance
ppm	parts per million
PQL	Practical Quantitation Limit
PQO	Practical Quality Objective
PVC	Polyvinyl Chloride

QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
r	Correlation Coefficient
r^2	Coefficient of Determination
RACR	Remedial Action Completion Report
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RL	Reporting Limit
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
STD	Standard
SVE	Soil Vapor Extraction
SVI	Soil Vapor Intrusion
SVOC	Semi-Volatile Organic Compound
TA	Test America Laboratories, Inc.
TAL	Target Analyte List
TBD	To Be Determined
TDS	Total Dissolved Solids
TIC	Tentatively Identified Compound
TKN	Total Kjehldahl nitrogen
TOC	Total Organic Carbon
UFP-QAPP	Uniform Federal Policy Quality Assurance Project Plan
UST	underground storage tank
USEPA	United States Environmental Protection Agency
VES	Vapor Extraction System
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
µg/L	microgram per liter

INTRODUCTION

FPM Remediations, Inc. (FPM), in association with CAPE Inc. (CAPE) and AECOM Technical Services (AECOM), under contract with the Air Force Civil Engineer Center (AFCEC), is performing long term monitoring (LTM), site remediation, and site investigations at the former Griffiss Air Force Base (AFB), Rome, New York (Figure 1-1).

The former Griffiss AFB covered approximately 3,552 contiguous acres in the lowlands of the Mohawk River Valley in Rome, Oneida County, New York. Topography within the valley is relatively flat, with elevations on the former Griffiss AFB ranging 435-595 feet above mean sea level. Three Mile Creek, Six Mile Creek (both of which drain into the New York State Barge Canal, located to the south of the base), and several state-designated wetlands are located on the former Griffiss AFB, which is bordered by the Mohawk River on the west. Due to its high average precipitation and predominantly silty sands, the former Griffiss AFB is considered a groundwater recharge zone.

The scope of work to be completed for this project is summarized in Table 1-1.

TABLE 1-1
GRIFFISS PERFORMANCE BASES REMEDIATION SCOPE OF WORK

Group	Sites	Work Element	Monitoring Matrix	Site Objective	Site Update Year 2014
CERCLA Sites – Landfill Areas of Concern (AOCs)	LF001, LF002, LF003, LF007, and LF009	LTM	Groundwater and Surface Water	Optimization	Optimization
CERCLA Sites - SD052 SVI System Sites	Buildings 774, 776, 785, and 786	SVI System Operation and Monitoring	Indoor/Outdoor Air and Sub-Slab Vapor	Optimization	Optimization
NYSDEC Petroleum Spill Sites – Site Closures	SD041, SS064, SS066, and SS067,	Groundwater/ Soil Remediation* and LTM	Groundwater Groundwater and Surface Water at SS064	Site Closure	Site Closure
NYSDEC Petroleum Spill Sites – Site Optimization	SS054 and SS068	Groundwater/ Soil Remediation and Long Term Monitoring	Groundwater	Optimization	Optimization

Group	Sites	Work Element	Monitoring Matrix	Site Objective	Site Update Year 2014
CERCLA Sites – SVI Land use Control/Institutional Control (LUC/IC) Sites	ST006	Sampling	Indoor/Outdoor Air, Sub-Slab Vapor, and Soil Vapor	Site Closure	Site part of PBR initially for LUC/ICs inspections only. Objective changed to Site Closure in 2012 Contract Modification
CERCLA Sites – LUC/IC Sites	DP012, DP013, AOI 72, and Building 211	Sampling	Soil	Site Closure	Sites part of PBR initially for LUC/ICs inspections only. Objective changed to Site Closure in 2012 Contract Modification

Note:
* - Groundwater and Soil Remediation will not occur at SS070.
NYSDEC = New York State Department of Environmental Conservation
CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act
SVI = Soil Vapor Intrusion

This Uniform Federal Policy Quality Assurance Project Plan (UFP/QAPP) has been prepared in conjunction with the tasks described in the Former Griffiss AFB 2013 Updated Project Management Plan (PMP) (CAPE, October 2013) and Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Sites and Petroleum Spill Sites Optimization Plans.

Quality Assurance Project Plan (QAPP) Worksheet #1 – Title and Approval Page

UFP-QAPP
Document Title

Air Force Civil Engineering Center
Lead Organization

Daniel Baldyga, FPM Remediations, Inc.
Preparer's Name and Organizational Affiliation

584 Phoenix Drive, Rome, NY, 13441, (315)336-7721, d.baldyga@fpm-remediations.com
Preparer's Address, Telephone Number, and E-mail Address

03/05/2014
Preparation Date (Day/Month/Year)

Investigative Organization's Project Manager: Phil Dula, Project Manager (PM)
Printed Name/Title

Signature/Date

Investigative Organization's Project Quality Assurance (QA) Officer:
Henry Vaca, Chem. Quality System Manager
Printed Name/Title

Signature/Date

Lead Organization's Project Manager: David Farnsworth, AFCEC PM
Printed Name/Title

Signature/Date

Laboratory QA Manager: Elaine Walker, Test America (TA)
Printed Name/Title

Signature/Date

Other Approval Authority: Mike Healy/General Operations Manager
Printed Name/Title

Signature/Date

QAPP Worksheet #2 – QAPP Identifying Information

Site Number/Code: None

Operable Unit: None

Contractor Name: CAPE/FPM/AECOM

Contractor Number: FA8903-10-D-8595

Contract Title: Performance Based Remediation

Work Assignment Number: Task Order

1. Identify guidance used to prepare QAPPs:
   UFP QAPP and Air Force Center for Engineering and the Environment (AFCEE) QAPP Version 4.0.02, May 2006.

2. Identify regulatory program:
   CERCLA, Resource Conservation and Recovery Act, United States Environmental Protection Agency (USEPA) Region II, and New York State Department of Environmental Conservation (NYSDEC)

3. Identify approval entity:
   AFCEC, USEPA, NYSDEC

4. This is a project-specific QAPP. This document was prepared in conjunction with the Former Griffiss AFB 2013 Updated PMP (CAPE, October 2013) and CERCLA Sites and Petroleum Spill Sites Optimization Plans.

5. List dates of scoping sessions that were held: March 30, 2011 and March 31, 2011.

6. List dates and titles of QAPP documents written for previous site work, if applicable:
   Basewide AFCEE QAPP for the former Griffiss AFB, October 2006.

7. List organizational partners (stakeholders) and connection with lead organization: USEPA, regulator; NYSDEC, regulator; New York State Department of Health (NYSDOH), regulator; AFCEC, representative party; CAPE, prime contractor; FPM, sub-contractor; AECOM, sub-contractor; and Property Owners/Occupants (including the Griffiss Local Development Corporation (GLDC), Mohawk Valley EDGE, and Oneida County Department of Aviation).

8. List data users:
   AFCEC, USEPA, NYSDEC, and NYSDOH

If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion: Not applicable.

QAPP Worksheet #3 – Distribution List

QAPP Recipients	Title	Organization / Address	Telephone Number	E-mail Address	Document Control Number
David Farnsworth	Griffiss BRAC Manager	Air Force Civil Engineer Center	518-563-2871	david.farnsworth@us.af.mil	
Robert Morse	Remedial PM	United States Environmental Protection Agency	212-637-4331	Morse.Bob@epamail.epa.gov	
Heather Bishop	Environmental Engineer	New York State Department of the Environmental Conservation	518-402-9692	hlbishop@gw.dec.state.ny.us	
Mark Tibbe	NYSDEC Petroleum Spills	New York State Department of the Environmental Conservation	315-793-2554	mctibbe@gw.dec.state.ny.us	
Kristin Kulow	NYSDOH	New York State Department of Health	607-432-3911	kxk07@health.state.ny.us	
Mike Healy	General Manger of Operations	CAPE Inc.	847-548-5994	mhealy@cape-inc.com	
Phil Dula	PM	CAPE Inc.	913-327-8300	pdula@cape-inc.com	
Gaby Atik	FPM Task Manager	FPM Remediations, Inc.	315-336-7721	g.atik@fpm-remediations.com	
Mike Niederreither	AECOM Task Manager	AECOM	717-790-3404	mike.niederreither@aecom.com	
Elaine Walker	Test America PM	Test America (Denver Laboratory)	303-736-0156	Elaine.walker@testamericainc.com	

QAPP Worksheet #4 – Project Personnel Sign-Off Sheet

Organization: AFCEC

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
David Farnsworth	AFCEC/CIBE Plattsburgh	315-356-0810		
Cathy Jerrard	AFCEC/ CIBE Griffiss	315-356-0810		

Organization: CAPE

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Kurt Gates	Program Manager (PGM)	210-377-2008		
Mike Healy	General Manager of Operations	847-548-5994		
Phil Dula	Project Manager	913-327-8300		
Merle Miller	Senior Engineer	210-377-2008		
Glen Mayekawa	Health and Safety (H&S) Manager	714-599-9071		
Henry Vaca	Quality System Manager	770-908-7200		

Organization: FPM

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Gaby Atik	FPM Task Manager	315-336-7721		
Daniel Baldyga	FPM Technical Lead	315-336-7721		
Connie van Hoesel	Chemical Quality Control (QC) Coordinator	315-336-7721		

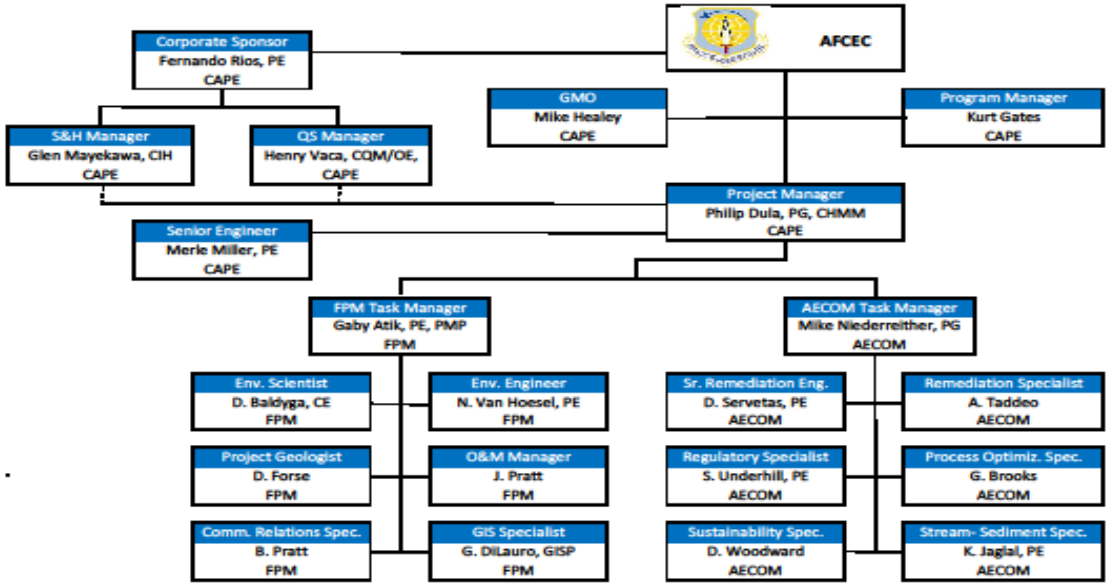
Organization: AECOM

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Mike Niederreither	AECOM Task Manager	717-790-3404		
John Santacroce	AECOM Technical Lead	518-951-2265		

Organization: Laboratory (Test America)

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Elaine Walker	TA PM	303-736-0156		

QAPP Worksheet #5 – Project Organizational Chart



QAPP Worksheet #6 – Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, pathways, etc.)
Point of Contact (POC) with USEPA, NYSDEC, and NYSDOH	AFCEC/ CIBE Plattsburgh	David Farnsworth	518-563-2871	David Farnsworth is the Griffiss BRAC Base Environmental Coordinator (BEC)
	AFCEC/ CIBE Griffiss	Cathy Jerrard	315-356-0810	Ms. Jerrard is an Environmental Engineer for Griffiss Support and an alternate point of contact to David Farnsworth.
Overall Project Management	AFCEC/ CIBE Griffiss	David Farnsworth	518-563-2871	David Farnsworth is the Griffiss BEC
Manage Program	CAPE PGM	Kurt Gates	210-377-2008	Is the single POC with authority for the program organization.
Manage entire project	CAPE PM	Phil Dula	913-327-8300	Is the primary interface with AFCEC and ensures performance objectives are met.
Manage project team	General Manager of Operations	Mike Healy	847-548-5994	Develops and maintains alliances with Team subcontractors.
Manage FPM Tasks	FPM Task Manager	Gaby Atik	315-336-7721	Communicates with CAPE PM and provides monthly reports and schedule updates to CAPE PM
Manage AECOM Tasks	AECOM Task Manager	Mike Niederreither	717-790-3404	Communicates with CAPE PM and provides monthly reports and schedule updates to CAPE PM
QAPP Changes in the Field	FPM Technical Lead	Daniel Baldyga	315-336-7721	Supervises field sampling and Operation and Maintenance (O&M) activities.
Daily Field Progress Reports	FPM Technical Lead	Daniel Baldyga	315-336-7721	Supervises field sampling and O&M activities. Authors status and completion reports.
Sampling, Drilling, and Monitoring Well Installation	AECOM Technical Lead	Dan Servetas	518-951-2378	Responsible for all drilling, sampling, and monitoring well installation activities to assure goals of the field investigations are attained.

QAPP Worksheet #6 – Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Reporting Lab Data Quality Issues	Chemical Quality Control Coordinator	Connie van Hoesel	315-336-7721	Will determine corrective actions (CAs) for lab data quality issues
Field and Analytical CAs	Chemical Quality Control Coordinator	Connie van Hoesel	315-336-7721	Will determine CAs for field and analytical issues
Release of Analytical Data	Chemical Quality Control Coordinator	Connie van Hoesel	315-336-7721	No analytical data can be released until validation is completed and has approved the release.
QAPP Amendments	AFCEC/CIBE Griffiss	Michael McDermott	315-356-0810	Any major changes to the QAPP must be approved by Michael McDermott before the changes can be implemented.

QAPP Worksheet #7 – Personnel Responsibilities and Qualifications Table

Name	Title	Organization	Responsibilities	Education and Experience Qualifications
David Farnsworth	Griffiss BEC	AFCEC/CIBE Plattsburgh	Support role as on site Air Force representative.	B.S. Civil Engineering, 32 years environmental experience with the federal government.
Cathy Jerrard	Environmental Engineer	AFCEC/CIBE Griffiss	Support role as on site Air Force representative.	B.S. Mechanical Engineering, 16 years of environmental experience.
Kurt Gates	PGM	CAPE	PGM	B.S., Safety Science Engineering, over 20 years of experience as PGM.
Phil Dula	PM	CAPE	Manages project – coordinates between lead agency and subcontractor.	M.B.A., M.S. Geology, B.A. Biology, 28 years environmental experience.
Mike Healy	General Manager of Operations	CAPE	Develops and maintains alliances with Team subcontractors.'	M.S. and B.S. Geology, 23 years environmental experience.
Merle Miller	Project Engineer	CAPE	Ensures all environmental, civil, and process engineering support goals are achieved.	B.S. Civil Engineering, 13 years of civil engineering experience.
Gaby Atik	FPM Task Manager	FPM	Communicates with CAPE PM and directs site work to ensure exact compliance with approved PWS, work plan, SSHP, CQC Plan, and applicable regulations.	M.S. Environmental Management, 20+ years environmental experience.
Mike Niederreither	AECOM Task Manager	AECOM	Communicates with CAPE PM and directs site work to ensure exact compliance with approved PWS, work plan, SSHP, CQC Plan, and applicable regulations.	M.S. Hydrology, B.S. Geology, 20+ years of environmental experience.
Daniel Baldyga	FPM Technical Lead	FPM	Supervises field sampling and coordinates all field activities.	B.S. Biology, 10+ years of environmental experience.

QAPP Worksheet #7 – Personnel Responsibilities and Qualifications Table

Name	Title	Organization	Responsibilities	Education and Experience Qualifications
Dan Servetas	AECOM Technical Lead	AECOM	Coordinates AECOM's internal resources, ensures all process engineering goals are achieved, and directs staff efforts.	M.S. Environmental Engineering, B.S Civil Engineering, 20+ years of environmental experience.
Glen Mayekawa	H&S Officer	CAPE	Oversees H&S for field activities.	M.S. and B.S. Health Science, 30+ years of comprehensive industrial hygiene experience and health and safety management.
Henry Vaca	Quality System Manager	CAPE	Quality System Manager	M.S. Quality Assurance, B.S. Mechanical Engineering, 25+ years of Quality Management Experience
Connie van Hoesel	Team Chemist/Data Specialist	FPM	Manages analytical laboratory (soil and groundwater samples).	M.S. Environmental Engineering, B.A. Chemistry, 11 years experience
Connie van Hoesel	Chemical Quality Control Coordinator	FPM	Determines need for CA for field and analytical issues. Performs data verification.	M.S. Environmental Engineering, B.A. Chemistry, 11 years of experience.

QAPP Worksheet #8 – Special Personnel Training Requirements Table

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training[1]	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates[2]
Field chemistry and sampling	H&S Training per 29 Code of Federal Regulations (CFR) 1910.120 Confined Space Entry Training – 8-hour per 29 CFR 1910.146 Tailgate meeting to discuss sampling plan and procedures	Various	Various Start of fieldwork	All	FPM Technical Lead, FPM	Onsite office Safety File, office electronic backup

1 All sampling personnel will be trained using sampling techniques described in the SOP (Appendix A).

2 All field personnel (including drilling sub-contractors) certifications will be retained electronic by the CAPE team for review.

QAPP Worksheet #9 – Project Scoping Session Participants Sheet

Project Name: Former Griffiss AFB PBR **Projected Date(s) of Sampling:** January 1, 2011 through December 31, 2015 **Project Manager:** Phil Dula, CAPE	**Site Name:** Former Griffiss AFB Petroleum Spill sites and CERCLA AOCs **Site Location:** Former Griffiss AFB, Rome, New York

Date of Session: March 30 and 31, 2011

Scoping Session Purpose: Kickoff meetings with Regulatory agencies to discuss site remediation/monitoring approaches and site objectives.

March 30, 2011

Name	Title	Affiliation	Phone #	E-mail Address
Michael McDermott	Former AFCEC PM			
Cathy Jerrard	AFCEC-Griffiss	AFCEC	315-356-0810	catherine.jerrard@us.af.mil
Scott Reichinger	NYSDEC Petroleum Spills	NYSDEC	315-793-2554	screichi@gw.dec.state.ny.us
Mark Tibbe	NYSDEC Petroleum Spills	NYSDEC	315-793-2747	mctibbe@gw.dec.state.ny.us
Phil Dula	PM	CAPE	913-327-8300	pdula@cape-inc.com
Gaby Atik	FPM Task Manager	FPM	315-336-7721	g.atik@fpm-remediations.com
Daniel Baldyga	FPM Technical Lead	FPM	315-336-7721	d.baldyga@fpm-remediations.com
Dan Servetas	AECOM Technical Lead	AECOM	518-951-2378	daniel.servetas@aecom.com
John Santacroce	AECOM	AECOM	518-951-2265	john.santacroce@aecom.com

March 31, 2011

Name	Title	Affiliation	Phone #	E-mail Address
Doug Pocze	Remedial PM	USEPA	212-637-4432	Pocze.Doug@epamail.epa.gov
Heather Bishop	Environmental Engineer	NYSDEC	518-402-9692	hlbishop@gw.dec.state.ny.us
John Swartout	NYSDEC	NYSDEC	518-402-9620	jbswarto@gw.dec.state.ny.us
Jim Harrington	NYSDEC	NYSDEC	518-402-9624	jbharrin@gw.dec.state.ny.us
Phil Dula	PM	CAPE	913-327-8300	pdula@cape-inc.com
Gaby Atik	FPM Task Manager	FPM	315-336-7721	g.atik@fpm-remediations.com
Daniel Baldyga	FPM Technical Lead	FPM	315-336-7721	d.baldyga@fpm-remediations.com
Dan Servetas	AECOM Technical Lead	AECOM	518-951-2378	daniel.servetas@aecom.com

Consensus Decisions:

Kick-off Meetings

The former Griffiss AFB PBR kickoff meetings were held to discuss all proposed site activities and site objectives. The discussions were held for 2 days to obtain feedback from the regulatory agencies. The March 30, 2011, meeting addressed planned strategies for the Griffiss Petroleum Spill Sites and the March 31, 2011, meeting was held to discuss planned approaches to achieve eventual site closures for the Griffiss CERCLA Sites.

March 30, 2011 – AECOM, CAPE, FPM, AFCEC, and NYSDEC

For the petroleum spill sites, LTM will continue in conjunction with additional groundwater and soil remediation through biosparging, bioventing, and oxidant injections. Remediation will be conducted at all sites except for SS070 (LTM only), and all sites are anticipated to achieve site and spill closure by the end of the period of performance (POP) except for SS054 and SS068.

March 31, 2011 – AECOM, CAPE, FPM, AFCEC, USEPA, and NYSDEC

For the CERCLA AOCs, LTM will continue at the Creek AOCs, Landfill AOCs, and SS060 (On-Base Groundwater AOCs). Groundwater remediation is also anticipated for SS060 via the use of emulsified vegetable oil injections. It is anticipated that SS060 will be closed with restricted use. LTM optimization is anticipated for the Landfill AOCs, and site closure is anticipated for the Creek AOCs. The site objectives will be supported through addition LTM.

Vapor sampling will be performed at the SD052 SVI system sites to monitor the effectiveness of the SVI systems during the POP. System and sampling optimization are the objectives at these systems.

Three Mile Creek AOC and Six Mile Creek AOC Meeting

A meeting between the Air Force, USEPA, and NYSDEC held on March 26, 2012 to discuss the Three Mile Creek and Six Mile Creek AOCs. Based on this meeting, a Remedial Action Completion Report (RACR) was completed submitted to EPA and DEC in August 2012. No additional sampling is recommended at the sites and sampling information pertaining to these sites has been deleted from this revision.

QAPP Worksheet #10 – Problem Definition

Problem Definition

As a result of the various national defense missions carried out at the former Griffiss AFB since 1942, hazardous and toxic substances were used and hazardous wastes were generated, stored, or disposed of at various sites on the installation. The defense missions involved, among others, procurement, storage, maintenance, and shipping of war material; research and development; and aircraft O&M.

Numerous studies, investigations, and remedial actions under the U.S. Department of Defense (DOD) Installation Restoration Program (IRP) have been performed to locate, assess, quantify, and remove contaminant sources at the past toxic and hazardous waste storage, disposal, and spill sites. Pursuant to Section 105 of CERCLA, Griffiss AFB was included on the National Priorities List (NPL) on July 15, 1987. On March 20, 2009, 2,897.2 acres were deleted from the NPL. On August 21, 1990, the Air Force, USEPA, and NYSDEC entered into a Federal Facilities Agreement (FFA) under Section 120 of CERCLA.

Starting in 2002, LTM was implemented at the former Griffiss AFB. LTM is currently conducted at several petroleum spill sites, including: petroleum source removal AOCs, on-base groundwater AOCs, landfill AOCs, and creek AOCs.

Groundwater and surface water monitoring is conducted at five landfill AOCs for landfill leachate indicators and volatile organic compounds (VOCs).

Groundwater sampling is conducted at five petroleum spill sites for VOCs. Surface water will be sampled at one site for VOCs. Two of the petroleum spill sites are associated with permanent biosparging systems. At the remaining petroleum spill sites, temporary biosparging and groundwater extraction will be conducted periodically.

Soil Vapor Intrusion (SVI) sampling is conducted at four SVI mitigation sites. Sampling consists of indoor, outdoor, and sub-slab vapor sampling for VOCs at each site.

SVI sampling is conducted at one Soil Vapor Extraction (SVE) system site. Sampling consists of indoor, outdoor, and sub-slab vapor sampling for VOCs.

Soil sampling will be initiated at three LUC/IC sites. Samples will analyzed for VOCs, pesticides, and metals including mercury and hexavalent chromium.

QAPP Worksheet #10 – Problem Definition

Project Description

Additional LTM for groundwater will be conducted at the petroleum spill sites, on-base groundwater AOC, and landfill AOCs. These sites and site objectives are provided in Table 1-1.

Groundwater:

- Analysis will include VOCs for quarterly, semi-annual, and annual monitoring performed at the petroleum spill sites (SS054, SS063, SS064, SS067, and SS068) at sampling locations illustrated in Figures 17-9 through 17-13.
- Analysis will include VOCs and landfill leachate indicators (including anions, Total Kjehldahl nitrogen (TKN), ammonia, COD, Biological Oxygen Demand (BOD), TOC, TDS, alkalinity, hardness, and color) for annual monitoring performed at the five Landfill AOCs. The sampling locations are illustrated on Figures 17-2 through 17-6.

Surface Water:

- Analysis of VOCs for quarterly monitoring at one petroleum spill site at three locations [SS064 (Figure 17-11)]. Analysis of VOCs and landfill leachate indicators for annual monitoring at two Landfill AOCs. Three sampling locations are present at LF001 (Figure 17-2) and six sampling locations are present at LF009 (Figure 17-6). Analysis of landfill leachate indicators will be conducted for annual sampling at three Landfill AOCs (LF002, LF003, and LF007). The sampling locations are illustrated on Figures 17-3, 17-4, and 17-5.

SVI Sampling:

- One indoor air sample, one outdoor air sample, and three sub-slab vapor samples will be collected semi-annually at each SVI mitigation system. The samples will be analyzed for VOCs. The vapor sampling locations are illustrated on Figures 17-7 and 17-8.
- Two indoor air samples, one outdoor air sample, and three sub-slab vapor samples will be collected quarterly at ST006 Building 101 AOC. The samples will be analyzed for VOCs. The vapor sampling locations are illustrated on Figure 17-14.

Soil:

QAPP Worksheet #10 – Problem Definition

- Sampling will be conducted at DP012 Building 301 AOC (five soil samples), at DP013 Building 255 AOC (2 soil samples), at Area of Interest (AOI) 72 (two soil samples), and at Building 211 (one composite concrete (treated as a soil) sample). The samples will be analyzed for pesticides at DP012 Building 301 AOC, hexavalent chromium at DP013 Building 255 AOC, VOCs at AOI 72, and mercury at Building 211. The sampling locations are illustrated in Figures 17-15, 17-16, 17-17, and 17-18.

Project Decision Condition:

In order to achieve the goals stated in the project description, the following data inputs have been identified:

- The confirmation and delineation of contamination at petroleum spill sites and AOCs throughout the former Griffiss AFB.

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements

Who will use the data? Data will be used by USEPA, NYSDEC, AFCEC, CAPE, FPM, and AECOM.

What will the data be used for?

- Groundwater analytical results will be used to assess groundwater contamination data trends and to support site closure or monitoring optimization recommendations
- Surface water analytical results will be used to assess contamination data trends and the potential impacts from upgradient sites, and to support site closure or monitoring optimization recommendations
- Indoor air, outdoor air, and sub-slab vapor analytical results will be used to assess the performance of the SVI mitigation systems and the performance of the SVE system.
- Soil analytical results will be used to delineate and quantify residual contamination at the petroleum sites. Soil analytical results will be used to support site closure with residential use at four LUC/IC sites

What types of data are needed?

- Groundwater samples analyzed for VOCs, SVOCs, and landfill leachate indicators
- Surface water samples analyzed for VOCs and landfill leachate indicators
- Soil samples analyzed for VOCs, metals including mercury and hexavalent chromium, and pesticides
- Indoor, outdoor, and sub-slab vapor samples analyzed for VOCs.

How much data are needed?

Landfill AOCs (LF001, LF002, LF003, LF007, and LF009) – Groundwater and surface water sampling will be required until 2040. Annual monitoring rounds will be used to support any optimization recommendations.

SVI mitigation system sites (SD052 SVI systems at Buildings 774, 776, 785, and 786) – Indoor, outdoor, and sub-slab vapor samples

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements

will be required as long as the systems are in operation. The semi-annual sampling will be used to assess the effectiveness of the SVI mitigation systems.

Petroleum spill sites (SS054, SS063, SS064, SS067, and SS068) – At least four quarterly sampling rounds of data is required to determine contamination trends or to support site closure. Data will include groundwater and surface water sampling results.

SVE system (ST006 Building 101) – two indoor air samples, one outdoor air sample, and three sub-slab vapor samples will be collected. The quarterly sampling will be used to assess the effectiveness of the SVE system.

LUC/IC AOCs – At DP012 Building 301 AOC, five endpoint samples will be used to confirm the absence of contamination above residential values following a removal action. Four samples will be collected in the walls of the excavation and one sample will be collected from the bottom of the excavation. At DP013 Building 255 AOC, two soil samples will be used to confirm the absence of chromium contamination above residential values from 0 to 2 ft bgs. At AOI 72, two endpoint samples from the bottom of the proposed excavation will be used to confirm the absence of contamination above residential values. At Building 211, one concrete (treated as soil) sample collected from the vault floor will be used to confirm the absence or presence of contamination above residential values.

How good does the data need to be? All analytical data will be generated from groundwater, surface water, vapor, and soil samples sent to TA. Samples will be duplicated in the field at a rate of 10% and analyzed by TA to assess sampling precision. Matrix spike/ matrix spike duplicates (MS/MSD) will be collected at a rate of 5%. Additional QC/ quality assurance (QA) protocols (field and lab) are provided in Worksheets # 20 and #22 through #33.

When will data be collected? Please refer to the CERCLA Sites and Petroleum Spill Sites Optimization Plans for this information.

QAPP Worksheet #12 – Measurement Performance Criteria Table

Matrix	Groundwater and soil	Full data verification and validation criteria are listed in Table 12-3			
Analytical Group	VOCs, SVOCs				
Conc. Level	Low-to-Medium				
Sampling Procedure[1]	**Analytical Method/ Standard Operating Procedure (SOP)**[2]	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), and/or Analytical (A)**
SOPs No. 1, No. 2, No. 3, and No. 4	EPA 8260B/ DV-MS-0010 EPA 8270D/ DV-MS-0012	Precision – Lab	Relative Percent Difference (RPD) < 30%	MS/MSD and/or Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)	S&A
		Precision – Field/Laboratory	If both the parent and duplicate values are > 5x Reporting Limit (RL, considered equivalent to the limit of quantitation [LOQ]), then 30% RPD for aqueous, 50% soil. If either the parent or duplicate value is < 5X the RL, then the difference between the parent and duplicate must be < 2X the RL.	Field Duplicates	S&A
		Accuracy/Bias	See Tables 12-1 and 12-2	LCS, MS/MSD and surrogate recoveries	A
		Accuracy/Bias Contamination	No target compounds > ½ LOQ	Method blanks	A

QAPP Worksheet #12 – Measurement Performance Criteria Table

Sampling Procedure[1]	Analytical Method/ SOP[2]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), and/or Analytical (A)
SOPs No. 1, No. 2, No. 3, and No. 4 (continued)	EPA 8260B/ DV-MS-0010 EPA 8270D/ DV-MS-0012 (continued)	Quantitation Limit	Limit of Quantitation (LOQ) > Limit of Detection (LOD) LOD & LOQ must be verified quarterly.	Standard that is at or below the LOQ as the lowest point on the calibration curve.	A
		Sensitivity	Sample results will be reported to the Detection Limit (DL).	Sample results that are less than the LOQ, but greater than the DL, will be reported with a J-flag.	A
		Completeness	90 and 95% for soil and groundwater, respectively	Data Completeness Check	S&A
		Properly Decontaminated Equipment	Detections < LOQ	Equipment Blank	S
		External Contamination of Samples	Detections < RL	Ambient Blank	S
		Sample Contamination check during transport	Detections < LOQ	Trip Blank	S

[1]Reference number from QAPP Worksheet #21

[2]Reference number from QAPP Worksheet #23

QAPP Worksheet #12 – Measurement Performance Criteria Table

Matrix	Soil	Full data verification and validation criteria are listed in Table 12-4			
Analytical Group	Pesticides, PCBs				
Conc. Level	Low-to-Medium				
Sampling Procedure[1]	**Analytical Method/ SOP[2]**	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), and/or Analytical (A)**
SOPs No. 1, No. 2, No. 3, No. 4, and No. 5	EPA 8081B/ DV-GC-0020/ DV-GC-0026 EPA 8082A/ DV-GC-0021/ DV-GC-0030	Precision – Lab	RPD < 20%	MS/MSD and/or LCS/LCSD	S&A
		Precision – Field/Laboratory	If both the parent and duplicate values are > 5x the RL (considered equivalent to the LOQ), then 50% for soil. If either the parent or duplicate value is < 5X the RL, then the difference between the parent and duplicate must be < 2X the RL.	Field Duplicates	S&A
		Accuracy/Bias	See Tables 12-1 and 12-2	LCS, MS/MSD and surrogate recoveries	A
		Accuracy/Bias Contamination	No target compounds > ½ LOQ	Method blanks	A

QAPP Worksheet #12 – Measurement Performance Criteria Table

Sampling Procedure[1]	Analytical Method/ SOP[2]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), and/or Analytical (A)
SOPs No. 1, No. 2, No. 3, No. 4, and No. 5 (continued)	EPA 8081B/ DV-GC-0020/ DV-GC-0026 EPA 8082A/ DV-GC-0021/ DV-GC-0030 (continued)	Quantitation Limit	LOQ > LOD LOD & LOQ must be verified quarterly.	Standard that is at or below the LOQ as the lowest point on the calibration curve.	A
		Sensitivity	Sample results will be reported to the DL.	Sample results that are less than the LOQ, but greater than the DL, will be reported with a J-flag.	A
		Completeness	> 90% laboratory analysis	Data Completeness Check	S&A
		Properly Decontaminated Equipment	Detections < LOQ	Equipment Blank	S

[1]Reference number from QAPP Worksheet #21

[2]Reference number from QAPP Worksheet #23

QAPP Worksheet #12 – Measurement Performance Criteria Table

Matrix	Groundwater and soil	Full data verification and validation criteria are listed in Table 12-5			
Analytical Group	Metals, including Hg and hexavalent chromium				
Conc. Level	Low-to-Medium				
Sampling Procedure[1]	**Analytical Method/ SOP[2]**	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), and/or Analytical (A)**
SOPs No. 1, No. 2, No. 3, No. 4, and No. 5	EPA 6010C/ DV-MT-0021 EPA 7470/7471B/ DV-MT-0016/ DV-MT-0017/ DV-MT-0023 EPA 7196A SA-GE-001	Precision – Lab	RPD < 20%	MS/MSD and/or LCS/LCSD	S&A
		Precision – Field/Laboratory	If both the parent and duplicate values are > 5x RL (considered equivalent to the LOQ), then 20% RPD for aqueous samples, 30% for soil. If either the parent or duplicate value is < 5X the RL, then the difference between the parent and duplicate must be < 2X the RL.	Field Duplicates	S&A
		Accuracy/Bias	See Tables 12-1 and 12-2	LCS, MS/MSD and surrogate recoveries	A
		Accuracy/Bias Contamination	No target compounds > ½ LOQ	Method blanks	A

QAPP Worksheet #12 – Measurement Performance Criteria Table

Sampling Procedure[1]	Analytical Method/ SOP[2]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), and/or Analytical (A)
SOPs No. 1, No. 2, No. 3, No. 4, and No. 5 (continued)	EPA 6010C/ DV-MT-0021 EPA 7470/7471B/ DV-MT-0016/ DV-MT-0017/ DV-MT-0023 EPA 7196A SA-GE-001 (continued)	Quantitation Limit	LOQ > LOD LOD & LOQ must be verified quarterly.	Standard that is at or below the LOQ as the lowest point on the calibration curve.	A
		Sensitivity	Sample results will be reported to the DL.	Sample results that are less than the LOQ, but greater than the DL, will be reported with a J-flag.	A
		Completeness	90 and 95% for soil and groundwater, respectively	Data Completeness Check	S&A
		Properly Decontaminated Equipment	Detections < LOQ	Equipment Blank	S

[1]Reference number from QAPP Worksheet #21

[2]Reference number from QAPP Worksheet #23

QAPP Worksheet #12 – Measurement Performance Criteria Table

Matrix	Groundwater	Full data verification and validation criteria are listed in Table 12-6			
Analytical Group	Wet chemistry analytes				
Conc. Level	Low-to-Medium				
Sampling Procedure[1]	**Analytical Method/ SOP[2]**	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), and/or Analytical (A)**
SOPs No. 1, No. 2, and No. 3	SW9056A, EPA 351.2, 350.1, 410.4, SM5210B, SM2340C, SM2120B, SW9060A, SW9066, SW9012B, SM2540C, SM2320B DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	Precision – Lab	RPD < 20%	MS/MSD and/or LCS/LCSD	S&A
		Precision – Field/Laboratory	If both the parent and duplicate values are > 5x RL (considered equivalent to the LOQ), then 20% RPD for aqueous samples, 30% for soil. If either the parent or duplicate value is < 5X the RL, then the difference between the parent and duplicate must be < 2X the RL.	Field Duplicates	S&A
		Accuracy/Bias	See Tables 12-1 and 12-2	LCS, MS/MSD and surrogate recoveries	A
		Accuracy/Bias Contamination	No target compounds > ½ LOQ	Method blanks	A

QAPP Worksheet #12 – Measurement Performance Criteria Table

Sampling Procedure[1]	Analytical Method/ SOP[2]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), and/or Analytical (A)
SOPs No. 1, No. 2, and No. 3 (continued)	SW9056A, EPA 351.2, 350.1, 410.4, SM5210B, SM2340C, SM2120B, SW9060A, SW9066, SW9012B, SM2540C, SM2320B DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060 (continued)	Quantitation Limit	LOQ > LOD LOD & LOQ must be verified quarterly.	Standard that is at or below the LOQ as the lowest point on the calibration curve.	A
		Sensitivity	Sample results will be reported to the DL.	Sample results that are less than the LOQ, but greater than the DL, will be reported with a J-flag.	A
		Completeness	> 95% laboratory analysis	Data Completeness Check	S&A
		Properly Decontaminated Equipment	Detections < LOQ	Equipment Blank	S

[1]Reference number from QAPP Worksheet #21

[2]Reference number from QAPP Worksheet #23

QAPP Worksheet #12 – Measurement Performance Criteria Table

Matrix	Soil gas	Full data verification and validation criteria are listed in Table 12-7			
Analytical Group	VOCs				
Conc. Level	Low-to-Medium				
Sampling Procedure[1]	**Analytical Method/ SOP[2]**	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), and/or Analytical (A)**
SOP No. 6	TO-15 BR-AT-004	Precision – Lab	RPD < 20%	MS/MSD and/or LCS/LCSD	S&A
		Precision – Field/Laboratory	If both the parent and duplicate values are > 5x RL (considered equivalent to the LOQ), then 25% RPD. If either the parent or duplicate value is < 5X the RL, then the difference between the parent and duplicate must be < 2X the RL.	Field Duplicates	S&A
		Accuracy/Bias	See Tables 12-1 and 12-2	LCS, MS/MSD and surrogate recoveries	A
		Accuracy/Bias Contamination	No target compounds > ½ LOQ	Method blanks	A

QAPP Worksheet #12 – Measurement Performance Criteria Table

Sampling Procedure[1]	Analytical Method/ SOP[2]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), and/or Analytical (A)
SOP No. 6 (continued)	TO-15 (continued) BR-AT-004	Quantitation Limit	LOQ > LOD LOD & LOQ must be verified quarterly.	Standard that is at or below the LOQ at the lowest point on the calibration curve.	A
		Sensitivity	Sample results will be reported to the DL.	Sample results that are less than the LOQ, but greater than the DL, will be reported with a J-flag.	A
		Completeness	> 95% laboratory analysis	Data Completeness Check	S&A
		Properly Decontaminated Equipment	Detections < LOQ	Equipment Blank	S

[1]Reference number from QAPP Worksheet #21

[2]Reference number from QAPP Worksheet #23

QAPP Worksheet #13 – Secondary Data Criteria and Limitations Table

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
Background information and historic levels of petroleum impact at the site.	Documents pertaining to past work at the former Griffiss AFB petroleum spill sites and AOCs.	Collection of groundwater, surface water, soil, soil gas, indoor vapor, outdoor vapor, and sub-slab vapor samples.	Data will be used to make project decisions and determine if closure requirements and clean-up goals are met.	None

QAPP Worksheet #14 – Summary of Project Tasks

Sampling Tasks:

General

1. Landfill AOCs:

- Sampling locations are shown on Figures 17-2 through 17-6. Discussion of the sampling approach and sampling design and rationale is provided in Worksheet #17.
- Groundwater sampling to assess the contaminant absence/presence and data trends.
- Surface water sampling to assess if groundwater contamination is migrating to creek environments.

2. SVI Mitigation System Sites:

- Sampling locations are shown on Figures 17-7 and 17-8. Discussion of the sampling approach and sampling design and rationale is provided in Worksheet #17.
- Indoor air sampling to evaluate the effectiveness of the SVI mitigation systems and assess data trends.
- Outdoor air sampling to be used as a reference for indoor sampling results.
- Sub-slab vapor sampling to evaluate the effectiveness of the SVI mitigation systems and assess data trends.

3. Petroleum Spill Sites:

- Sampling locations are shown on Figures 17-9 through 17-13. Discussion of the sampling approach and sampling design and rationale is provided in Worksheet #17.
- Groundwater sampling to evaluate contamination trends and the effectiveness of the groundwater remediation systems.

QAPP Worksheet #14 – Summary of Project Tasks

5. SVE System Sites:

- Sampling locations are shown on Figure 17-14. Discussion of the sampling approach and sampling design and rationale is provided in Worksheet #17.
- Sub-slab vapor sampling to evaluate the effectiveness of the SVE system and assess data trends.

6. LUC/IC Sites:

- Sampling locations are shown on Figures 17-15, 17-16, 17-17, and 17-18. Discussion of the sampling approach and sampling design and rationale is provided in Worksheet #17.
- Soil sampling to determine the presence/absence of residual contamination at site. If contamination is present, determine if levels are below residential site levels.

Samples will be collected using the SOPs attached as Appendix A of this UFP-QAPP.

Analysis Tasks:

1. Landfill AOCs

- TA will analyze groundwater samples for VOCs using USEPA Method SW8260B (AFCEE QAPP 4.0 List), metals using USEPA Method SW6010, and Landfill leachate indicators using USEPA methods SW9056A (anions), 351.2 (nitrogen), 350.1 (ammonia), 410.4 [chemical oxygen demand (COD)], SM5210B [biological oxygen demand (BOD)], SW9060A [total organic carbon (TOC)], SM2540C [total dissolved solids (TDS)], SM 2320B (alkalinity), SM2340C (hardness), 110.2 (color), SW9066 (phenols), SW9012B (cyanide), and SW 6010B (metals including boron).

2. SVI Mitigation System Sites

- TA will analyze vapor samples for VOCs using Method TO-15 (AFCEE QAPP 4.0 List).

3. On-base Groundwater AOC

QAPP Worksheet #14 – Summary of Project Tasks

- TA will analyze groundwater samples for VOCs using USEPA Method SW8260B (AFCEE QAPP 4.0 List).

4. Petroleum Spill Sites.
   - TA will analyze groundwater and surface water samples for VOCs using USEPA Method SW8260B (AFCEE QAPP 4.0 List and STARS List) and SVOCs using USEPA Method SW8270 (AFCEE QAPP 4.0 List).
5. SVE System Site
   - TA will analyze vapor samples for VOCs using Method TO-15 (AFCEE QAPP 4.0 List).
6. LUC/IC Sites
   - TA will analyze soil samples for VOCs using USEPA Method SW8260B, pesticides using USEPA Method SW8081B, metals using USEPA Method SW6010C, mercury using USEPA Method 7471B, and hexavalent chromium using USEPA Method 7196A.

Quality Control Tasks:

1. MS/MSDs will be collected at an approximate frequency of 5%.
2. Duplicates will be collected at a rate of 10% and analyzed by TA to assess field and laboratory precision.
3. Trip blank samples will be included in each cooler containing samples for VOC analysis.
4. Ambient blanks will be collected each day that VOC samples are collected.
5. Equipment blanks will be collected from each type of non-disposable, decontaminated sampling device.

QAPP Worksheet #14 – Summary of Project Tasks

Secondary Data:

Previously collected data will be evaluated. See Worksheet #13.

Data Management Tasks:

Data will be delivered in an Environmental Restoration Program Information Management System (ERPIMS) database compatible format after data verification/ validation have been performed and data qualifiers have been added.

Documentation and Records:

1. All field documentation will be recorded in indelible ink in bound field books. These will summarize all daily field activities, weather conditions, personnel present, visitors, etc. All samples collected will be documented as to their location, which will be measured from the closest two perpendicular walls. Each day's samples and associated field measurements shall be recorded on field sampling forms. Chain of Custody (COC) forms, airbills, and sample logs will be prepared and retained for each sample.

2. A copy of the final UFP QAPP will be retained in central project file (electronically on a server) and in print form in the onsite office.

Data Packages:

TA will complete analytical data packages in accordance with the AFCEC approved forms or similar and will provide ERPIMS X file.

Assessment / Audit Tasks:

Field Sample Collection and Documentation Audits: to be determined.

Data Review Tasks

1. For the samples, TA will verify that all data are complete for samples received. All data package deliverable requirements will be met. Data will be 100% verified by FPM in accordance with this UFP-QAPP. A data verification report will be prepared for each lab work order (lab data package).

2. Verified and validated data and all related field logbooks/notes/records will be reviewed to assess total measurement error and determine overall usability of the data for project purposes. Data limitations will be determined and data will be compared to Project Quality Objectives and required

QAPP Worksheet #14 – Summary of Project Tasks

Action Limits. CA will be initiated as necessary. Final data are placed in the ERPIMS database, with any necessary qualifiers and tables, charts and graphs generated.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOCs (SW8620B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
1,1,1,2-Tetrachloroethane	630-20-6	5	0.17	0.2	1.0
1,1,1-Trichloroethane	71-55-6	5	0.16	0.2	1.0
1,1,2,2-Tetrachloroethane	79-34-5	5	0.20	0.4	1.0
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5	0.79	2.8	3.0
1,1,2-Trichloroethane	79-00-5	1	0.32	0.4	1.0
1,1-Dichloroethane	75-34-3	5	0.16	0.2	1.0
1,1-Dichloroethene	75-35-4	5	0.14	0.2	1.0
1,1-Dichloropropene	563-58-6	5	0.15	0.4	1.0
1,2,3-Trichlorobenzene	87-61-6	5	0.18	0.4	1.0
1,2,3-Trichloropropane	96-18-4	0.04	0.77	0.8	3.0
1,2,4-Trichlorobenzene	120-82-1	5	0.32	0.8	1.0
1,2,4-Trimethylbenzene	95-63-6	5	0.14	0.2	1.0
1,2-Dibromo-3-Chloropropane	96-12-8	0.04	0.81	1.6	5.0
1,2-Dichlorobenzene	95-50-1	3	0.13	0.2	1.0
1,2-Dichloroethane	107-06-2	0.6	0.13	0.2	1.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOCs (SW8620B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
1,2-Dichloropropane	78-87-5	1	0.13	0.2	1.0
1,3,5-Trimethylbenzene	108-67-8	5	0.14	0.4	1.0
1,3-Dichlorobenzene	541-73-1	3	0.16	0.2	1.0
1,3-Dichloropropane	142-28-9	5	0.15	0.2	1.0
1,4-Dichlorobenzene	106-46-7	3	0.16	0.4	1.0
1,4-Dioxane	123-91-1	6.7[3]	71	80	220
1-Chlorohexane	544-10-5	NA	0.17	0.2	1.0
2,2-Dichloropropane	594-20-7	5	0.20	0.4	1.0
2-Butanone (MEK)	78-93-3	49,000[3]	1.83	3.2	6.0
2-Hexanone	591-78-6	50	1.4	3.2	5.0
4-Chlorotoluene	106-43-4	5	0.17	0.4	1.0
4-Methyl-2-pentanone (MIBK)	108-10-1	10,000[3]	1.04	3.2	5.0
Acetone	67-64-1	50	1.9	6.4	10
Benzene	71-43-2	1	0.16	0.2	1.0
Bromobenzene	108-86-1	5	0.17	0.2	1.0
Bromochloromethane	74-97-5	5	0.10	0.2	1.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOCs (SW8620B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Bromodichloromethane	75-27-4	50	0.17	0.2	1.0
Bromoform	75-25-2	50	0.19	0.4	1.0
Bromomethane	74-83-9	5	0.21	0.4	2.0
Carbon disulfide	75-15-0	15,000[3]	0.45	0.8	2.0
Carbon tetrachloride	56-23-5	5	0.19	0.4	2.0
Chlorobenzene	108-90-7	5	0.17	0.2	1.0
Chlorobromomethane	74-97-5	830[3]	0.10	0.2	1.0
Chlorodibromomethane	124-48-1	50	0.17	0.4	1.0
Chloroethane	75-00-3	5	0.41	1.6	2.0
Chloroform	67-66-3	7	0.16	0.2	1.0
Chloromethane	74-87-3	1,900[3]	0.30	0.8	2.0
cis-1,2-Dichloroethene	156-59-2	5	0.15	0.2	1.0
cis-1,3-Dichloropropene	10061-01-5	5	0.16	0.2	1.0
Cyclohexane	110-82-7	130,000[3]	0.28	0.4	2.0
Dibromomethane	74-95-3	5	0.17	0.4	1.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOCs (SW8620B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Dichlorodifluoromethane	75-71-8	5	0.31	0.8	2.0
Ethylbenzene	100-41-4	5	0.16	0.2	1.0
Hexachlorobutadiene	87-68-3	0.5	0.36	0.4	1.0
Isopropylbenzene	98-82-8	5	0.19	0.4	1.0
Methyl acetate	79-20-9	160,000[3]	1.64	2	5.0
Methyl tert-butyl ether	1634-04-4	10	0.25	0.4	5.0
Methylcyclohexane	108-87-2	NA	0.36	0.4	2.0
Methylene Chloride	75-09-2	5	0.32	0.4	5.0
m-Xylene & p-Xylene	179601-23-1	5	0.34	0.8	2.0
Naphthalene	91-20-3	10	0.22	0.8	1.0
n-Butylbenzene	104-51-8	5	0.32	0.4	1.0
N-Propylbenzene	103-65-1	5	0.16	0.2	1.0
o-Xylene	95-47-6	5	0.19	0.4	1.0
p-Isopropyltoluene	99-87-6	5	0.17	0.4	1.0
sec-Butylbenzene	135-98-8	5	0.17	0.4	1.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOCs (SW8620B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Styrene	100-42-5	5	0.17	0.4	1.0
tert-Butylbenzene	98-06-6	5	0.16	0.4	1.0
Tetrachloroethene	127-18-4	5	0.20	0.4	1.0
Toluene	108-88-3	5	0.17	0.4	1.0
trans-1,2-Dichloroethene	156-60-5	5	0.15	0.2	1.0
trans-1,3-Dichloropropene	10061-02-6	0.4	0.19	0.4	1.0
Trichloroethene	79-01-6	5	0.16	0.2	1.0
Trichlorofluoromethane	75-69-4	5	0.29	0.8	2.0
Vinyl chloride	75-01-4	2	0.10	0.4	1.5
Xylenes, Total	1330-20-7	5	0.53	1.2	3.0

1. New York State Department of Environmental Conservation (NYSDEC) Division of Water Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations, 6 NYCRR Part 703, NYSDEC, August 1999
2. Laboratory-specific DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method. DLs may be subject to update.
[3]EPA Regional Screening Levels Tapwater Supporting Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non cancer hazardous index
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: VOCs (SW8260B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
1,1,1,2-Tetrachloroethane	630-20-6	19,000[4]	0.56	1.0	5.0
1,1,1-Trichloroethane	71-55-6	500,000/100,000[1]	0.52	1.0	5.0
1,1,2,2-Tetrachloroethane	79-34-5	NA/35,000[3]	0.61	1.0	5.0
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	NA/100,000[3]	0.45	20	20
1,1,2-Trichloroethane	79-00-5	11,000[4]	0.88	1.0	5.0
1,1-Dichloroethane	75-34-3	240,000/19,000[1]	0.21	0.8	5.0
1,1-Dichloroethene	75-35-4	500,000/100,000[1]	0.59	1.0	5.0
1,1-Dichloropropene	563-58-6	NA/NA	0.54	1.0	5.0
1,2,3-Trichlorobenzene	87-61-6	490,000[4]	0.75	1.0	5.0
1,2,3-Trichloropropane	96-18-4	NA/80,000[3]	0.81	1.0	5.0
1,2,4-Trichlorobenzene	120-82-1	220,000[4]	0.73	1.0	5.0
1,2,4-Trimethylbenzene	95-63-6	190,000/47,000[1]	0.58	1.0	5.0
1,2-Dibromo-3-Chloropropane	96-12-8	54[4]	0.60	1.0	10
1,2-Dichlorobenzene	95-50-1	500,000/100,000[1]	0.45	1.0	5.0
1,2-Dichloroethane	107-06-2	30,000/2,300[1]	0.70	1.0	5.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: VOCs (SW8260B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
1,2-Dichloropropane	78-87-5	9,400[4]	0.55	1.0	5.0
1,3,5-Trimethylbenzene	108-67-8	190,000/47,000[1]	0.57	1.0	5.0
1,3-Dichlorobenzene	541-73-1	280,000/17,000[1]	0.48	1.0	5.0
1,3-Dichloropropane	142-28-9	16,000,000[4]	0.51	1.0	5.0
1,4-Dichlorobenzene	106-46-7	130,000/9,800[1]	0.78	1.0	5.0
1,4-Dioxane	123-91-1	130,000/9,800[1]	56.1	80	500
1-Chlorohexane	544-10-5	NA/NA	0.63	0.80	5.0
2,2-Dichloropropane	594-20-7	NA/NA	0.44	1.0	5.0
2-Butanone (MEK)	78-93-3	500,000/100,000[1]	1.83	6.4	20
2-Hexanone	591-78-6	2,100,000[4]	4.89	10	20
4-Chlorotoluene	106-43-4	NA/NA	0.78	1.0	5.0
4-Methyl-2-pentanone (MIBK)	108-10-1	53,000,000[4]	4.36	10	20
Acetone	67-64-1	500,000/100,000[1]	5.38	10	20
Benzene	71-43-2	44,000/2,900[1]	0.47	1.0	5.0
Bromobenzene	108-86-1	3,000,000[4]	0.49	1.0	5.0
Bromochloromethane	74-97-5	1,600,000[4]	0.30	1.0	5.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: VOCs (SW8260B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Bromodichloromethane	75-27-4	2,700[4]	0.22	0.8	5.0
Bromoform	75-25-2	620,000[4]	0.23	0.8	5.0
Bromomethane	74-83-9	73,000[4]	0.50	1.0	10
Carbon disulfide	75-15-0	NA/100,000[3]	0.42	1.0	5.0
Carbon tetrachloride	56-23-5	22,000/1,400[1]	0.63	1.0	5.0
Chlorobenzene	108-90-7	500,000/100,000[1]	0.54	1.0	5.0
Chlorodibromomethane	124-48-1	NA/NA	0.57	1.0	5.0
Chloroethane	75-00-3	NA/NA	0.89	1.0	10
Chloroform	67-66-3	350,000/10,000[1]	0.29	1.0	10
Chloromethane	74-87-3	1,200,000[4]	0.77	1.0	10
cis-1,2-Dichloroethene	156-59-2	500,000/59,000[1]	0.56	1.0	5.0
cis-1,3-Dichloropropene	10061-01-5	NA/NA	1.29	2.0	5.0
Cyclohexane	110-82-7	70,000,000[4]	0.40	1.6	5.0
Dibromomethane	74-95-3	250,000[4]	0.84	1.0	5.0
Dichlorodifluoromethane	75-71-8	940,000[4]	0.52	1.0	10
Ethylbenzene	100-41-4	390,000/30,000[1]	0.67	1.0	5.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: VOCs (SW8260B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Hexachlorobutadiene	87-68-3	62,000[4]	0.55	1.0	5.0
Isopropylbenzene	98-82-8	NA/100,000[3]	0.59	1.0	5.0
Methyl acetate	79-20-9	780,000,000[4]	2.75	4.0	8.5
Methyl tert-butyl ether	1634-04-4	500,000/62,000[1]	0.34	1.0	20
Methylcyclohexane	108-87-2	NA/NA	0.42	0.80	5.0
Methylene Chloride	75-09-2	500,000/51,000[1]	1.6	3.2	5.0
m-Xylene & p-Xylene	179601-23-1	NA/NA	1.04	2.0	3.2
Naphthalene	91-20-3	500,000/100,000[1]	0.63	1.0	5.0
n-Butylbenzene	104-51-8	500,000/100,000[1]	0.56	1.0	5.0
n-Propylbenzene	103-65-1	500,000/100,000[1]	0.58	1.0	5.0
o-Xylene	95-47-6	6,900,000[4]	0.61	1.0	5.0
p-Isopropyltoluene	99-87-6	NA/NA	0.49	1.0	5.0
sec-Butylbenzene	135-98-8	500,000/100,000[1]	0.77	1.0	5.0
Styrene	100-42-5	100,000,000[4]	0.63	1.0	5.0
tert-Butylbenzene	98-06-6	500,000/100,000[1]	0.50	1.0	5.0
Tetrachloroethene	127-18-4	150,000/5,500[1]	0.59	1.0	5.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: VOCs (SW8260B)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Toluene	108-88-3	500,000/100,000[1]	0.69	1.0	5.0
trans-1,2-Dichloroethene	156-60-5	500,000/100,000[1]	0.39	1.0	5.0
trans-1,3-Dichloropropene	10061-02-6	NA/NA	0.67	1.0	5.0
Trichloroethene	79-01-6	200,000/10,000[1]	0.23	0.8	5.0
Trichlorofluoromethane	75-69-4	7,900,000[4]	1.04	2.0	10
Vinyl chloride	75-01-4	13,000/210[1]	1.34	2.0	5.0
Xylenes, Total	1330-20-7	500,000/100,000[1]	1.65	3.0	8.2

1 - NYSDEC 60 NYCRR Part 375 Restricted and UnrestrictedUse Soil Cleanup Objectives, December 2006. Commercial clean-up objectives from Table 375-6.8(b) and Unrestricted clean-up objectives from Table 375-6.8(a).
2 - Achievable DLs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.
3 - CP-51/ Soil Cleanup Guidance, October 2010.
4 - EPA Regional Screening Levels Resident Soil Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non cancer hazardous index
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
1,1'-Biphenyl	92-52-4	5	1.75	2.0	10
1,2,4,5-Tetrachlorobenzene	95-94-3	5	1.73	2.0	10
1,2,4-Trichlorobenzene	120-82-1	5	0.28	1.0	10
1,2-Dichlorobenzene	95-50-1	3	0.23	1.0	10
1,3-Dichlorobenzene	541-73-1	3	0.30	1.0	10
1,4-Dichlorobenzene	106-46-7	3	0.32	1.0	10
2,2'-oxybis[1-chloropropane]	108-60-1	5	0.28	1.0	10
2,3,4,6-Tetrachlorophenol	58-90-2	1,700[3]	2.0	2.0	50
2,4,5-Trichlorophenol	95-95-4	8,900[3]	0.45	1.0	20
2,4,6-Trichlorophenol	88-06-2	35[3]	0.29	1.0	20
2,4-Dichlorophenol	120-83-2	1	0.64	2.0	10
2,4-Dimethylphenol	105-67-9	1	0.58	4.0	10
2,4-Dinitrophenol	51-28-5	1	10	20	80
2,4-Dinitrotoluene	121-14-2	5	1.66	4.0	20
2,6-Dinitrotoluene	606-20-2	5	1.89	4.0	20

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
2-Chloroaniline	106-47-8	5	2.14	5.0	25
2-Chloronaphthalene	91-58-7	10	0.26	1.0	10
2-Chlorophenol	95-57-8	710[3]	2.0	4.0	10
2-Methylnaphthalene	91-57-6	270[3]	0.29	1.0	10
2-Methylphenol	95-48-7	1	0.98	4.0	10
2-Nitroaniline	88-74-4	5	1.73	4.0	50
2-Nitrophenol	88-75-5	NA	0.39	1.0	20
3,3'-Dichlorobenzidine	91-94-1	5	2.0	10	50
3-Nitroaniline	99-09-2	5	2.0	2.0	50
4,6-Dinitro-2-methylphenol	534-52-1	12[3]	4.0	10	80
4-Bromophenyl phenyl ether	101-55-3	NA	0.43	1.0	10
4-Chloro-3-methylphenol	59-50-7	NA	2.41	5.0	20
4-Chloroaniline	106-47-8	5	2.14	5.0	25
4-Chlorophenyl phenyl ether	7005-72-3	NA	1.66	4.0	10
4-Methylphenol	106-44-5	NA	0.25	1.0	20
4-Nitroaniline	100-01-6	5	2.0	4.0	50

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
4-Nitrophenol	100-02-7	NA	1.23	10	50
Acenaphthene	83-32-9	20	0.28	1.0	10
Acenaphthylene	208-96-8	NA	0.49	1.0	10
Acetophenone	98-86-2	15,000[3]	0.24	5.0	10
Anthracene	120-12-7	50	0.42	1.0	10
Atrazine	1912-24-9	7.5	0.73	10	50
Benzaldehyde	100-52-7	15,000[3]	2.0	2.0	10
Benzo[a]anthracene	56-55-3	0.002[4*]	0.35	1.0	10
Benzo[a]pyrene	50-32-8	Not Detectable	0.31	1.0	10
Benzo[b]fluoranthene	205-99-2	0.002[4*]	0.531	1.0	10
Benzo[g,h,i]perylene	191-24-2	NA	0.50	1.0	10
Benzo[k]fluoranthene	207-08-9	0.002[4*]	0.46	1.0	10
Benzoic acid	65-85-0	580,000[3]	10	50	80
Benzyl alcohol	100-51-6	15,000[3]	0.23	1.0	25
Bis(2-chloroethoxy)methane	111-91-1	5	0.97	4.0	10
Bis(2-chloroethyl)ether	111-44-4	1	0.41	1.0	20

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Bis(2-ethylhexyl) phthalate	117-81-7	5	0.56	1.0	10
Butyl benzyl phthalate	85-68-7	50	1.0	4.0	20
Caprolactam	105-60-2	77,000[3]	5.0	10	35
Carbazole	86-74-8	NA	0.43	1.0	10
Chrysene	218-01-9	0.002[4*]	0.54	1.0	10
Dibenz(a,h)anthracene	53-70-3	0.029[3*]	0.51	1.0	10
Dibenzofuran	132-64-9	58[3]	0.29	1.0	10
Diethyl phthalate	84-66-2	50	0.38	1.0	20
Dimethyl phthalate	131-11-3	50	0.21	1.0	20
Di-n-butyl phthalate	84-74-2	50	1.16	4.0	20
Di-n-octyl phthalate	117-84-0	50	0.35	1.0	20
Fluoranthene	206-44-0	50	0.20	1.0	20
Fluorene	86-73-7	50	0.31	1.0	10
Hexachlorobenzene	118-74-1	0.04*	0.66	1.0	10
Hexachlorobutadiene	87-68-3	0.5*	3.3	10	30
Hexachlorocyclopentadiene	77-47-4	5	10	20	50

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[2]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Hexachloroethane	67-72-1	5	2.1	4.0	10
Indeno[1,2,3-cd]pyrene	193-39-5[4]	0.002*	0.65	1.0	10
Isophorone	78-59-1	50	0.21	1.0	10
Naphthalene	91-20-3	10	0.29	1.0	10
Nitrobenzene	98-95-3	0.4	0.81	2.0	20
N-Nitrosodi-n-propylamine	621-64-7	0.093[3*]	0.35	1.0	20
N-Nitrosodiphenylamine	86-30-6	50[3]	0.44	1.0	10
Pentachlorophenol	87-86-5	1	20	40	80
Phenanthrene	85-01-8	50	0.26	1.0	10
Phenol	108-95-2	1	2.0	5.0	10
Pyrene	129-00-0	50	0.37	1.0	10

* - Analyte has DL greater than PA is below the DL. If the analyte is not detected, it will be considered below the standard.
1 - New York State Department of Environmental Conservation (NYSDEC) Division of Water Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations, 6 NYCRR Part 703, NYSDEC, August 1999
2 - Laboratory-specific DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method. DLs may be subject to update.
3 - EPA Regional Screening Levels Tapwater Supporting Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non cancer hazardous index
4 - New York State Department of Environmental Conservation (NYSDEC) Division of Water Technical and Operational Guidance Series, Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations, NYSDEC, June 1998
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
1,1'-Biphenyl	92-52-4	510,000[4]	50	167	330
1,2,4,5-Tetrachlorobenzene	95-94-3	180,000[4]	49	67	330
1,2,4-Trichlorobenzene	120-82-1	220,000[4]	28	33	330
1,2-Dichlorobenzene	95-50-1	500,000/100,000[1]	22	33	330
1,3-Dichlorobenzene	541-73-1	280,000/17,000[1]	12	33	330
1,4-Dichlorobenzene	106-46-7	130,000/9,800[1]	13.6	33	330
2,2'-oxybis[1-chloropropane]	108-60-1	NA/NA	23	33	330
2,3,4,6-Tetrachlorophenol	58-90-2	18,000,000[4]	137	167	1600
2,4,5-Trichlorophenol	95-95-4	NA/100,000[3]	10	130	330
2,4,6-Trichlorophenol	88-06-2	440,000[4]	10	66	330
2,4-Dichlorophenol	120-83-2	NA/100,000[3]	10	66	330
2,4-Dimethylphenol	105-67-9	12,000,000[4]	66	130	330
2,4-Dinitrophenol	51-28-5	NA/100,000[3]	333	670	1600
2,4-Dinitrotoluene	121-14-2	16,000[4]	66	130	330
2,6-Dinitrotoluene	606-20-2	NA/1,030[3]	28	66	330

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
2-Chloroaniline	95-51-2	NA/NA	81.9	130	330
2-Chloronaphthalene	91-58-7	NA/NA	10	33	330
2-Chlorophenol	95-57-8	NA/100,000[3]	21	33	330
2-Methylnaphthalene	91-57-6	NA/410[3]	19	33	330
2-Methylphenol	95-48-7	500,000/100,000[1]	13	33	330
2-Nitroaniline	88-74-4	6,100,000[4]	50	66	1600
2-Nitrophenol	88-75-5	NA/NA	10	66	330
3,3'-Dichlorobenzidine	91-94-1	11,000[4]	90	330	1600
3-Nitroaniline	99-09-2	NA/NA	73	133	1600
4,6-Dinitro-2-methylphenol	534-52-1	49,000[4]	330	660	1600
4-Bromophenyl phenyl ether	101-55-3	NA/NA	19	33	330
4-Chloro-3-methylphenol	59-50-7	NA/NA	66	130	330
4-Chloroaniline	106-47-8	NA/100,000[3]	81.9	130	330
4-Chlorophenyl phenyl ether	7005-72-3	NA/NA	21	66	330
4-Methylphenol	106-44-5	500,000/34,000[3]	33	66	330
4-Nitroaniline	100-01-6	240,000[4]	72.5	130	1600

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
4-Nitrophenol	100-02-7	NA/NA	97	330	1600
Acenaphthene	83-32-9	500,000/100,000[1]	10.3	17	330
Acenaphthylene	208-96-8	500,000/100,000[1]	17	33	330
Acetophenone	98-86-2	78,000,000[4]	20	33	330
Anthracene	120-12-7	500,000/100,000[1]	17	33	330
Atrazine	1912-24-9	21,000[4]	37	130	330
Benzaldehyde	100-52-7	78,000,000[4]	39	67	330
Benzo[a]anthracene	56-55-3	5,600/1,000[1]	20	33	330
Benzo[a]pyrene	50-32-8	1,000/1,000[1]	20	33	330
Benzo[b]fluoranthene	205-99-2	5,600/1,000[1]	26.2	33	330
Benzo[g,h,i]perylene	191-24-2	500,000/100,000[1]	16	33	330
Benzo[k]fluoranthene	207-08-9	56,000/1,000[1]	40	66	330
Benzoic acid	65-85-0	NA/100,000[3]	330	660	1600
Benzyl alcohol	100-51-6	61,000,000[4]	10	33	330
Bis(2-chloroethoxy)methane	111-91-1	1,800,000[4]	23	66	330
Bis(2-chloroethyl)ether	111-44-4	6,900[4]	16.6	33	330

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Bis(2-ethylhexyl) phthalate	117-81-7	NA/50,000[3]	46	66	330
Butyl benzyl phthalate	85-68-7	NA/100,000[3]	43	66	330
Caprolactam	105-60-2	310,000,000[4]	106	330	1600
Carbazole	86-74-8	NA/NA	36	67	330
Chrysene	218-01-9	56,000/1,000[1]	27	33	330
Dibenz(a,h)anthracene	53-70-3	560/330[1]	19	33	330
Dibenzofuran	132-64-9	350,000/14,000[1]	20	33	330
Diethyl phthalate	84-66-2	NA/100,000[3]	26	33	660
Dimethyl phthalate	131-11-3	NA/100,000[3]	23	33	330
Di-n-butyl phthalate	84-74-2	NA/100,000[3]	29	33	330
Di-n-octyl phthalate	117-84-0	100,000[3]	14.4	66	330
Fluoranthene	206-44-0	500,000/100,000[1]	36	66	330
Fluorene	86-73-7	500,000/100,000[1]	18	33	330
Hexachlorobenzene	118-74-1	6,000/330[1]	29	66	330
Hexachlorobutadiene	87-68-3	62,000[4]	10	66	330
Hexachlorocyclopentadiene	77-47-4	3,700,000[4]	50	66	1700

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: SVOCs including PAH (SW8270D)

Concentration Level: Low-to-Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Hexachloroethane	67-72-1	120,000[4]	21.3	33	330
Indeno[1,2,3-cd]pyrene	193-39-5	5,600/500[1]	22	33	330
Isophorone	78-59-1	NA/100,000[3]	17	33	330
Naphthalene	91-20-3	500,000/100,000[1]	31	66	330
Nitrobenzene	98-95-3	69,000/3,700[3]	22	33	330
N-Nitrosodi-n-propylamine	621-64-7	690[4]	31	66	330
N-Nitrosodiphenylamine	86-30-6	990,000[4]	21	33	330
Pentachlorophenol	87-86-5	6,700/2,400[1]	330	670	1600
Phenanthrene	85-01-8	500,000/100,000[1]	17	33	330
Phenol	108-95-2	500,000/100,000[1]	18	33	330
Pyrene	129-00-0	500,000/100,000[1]	12.1	33	400

1 - NYSDEC 60 NYCRR Part 375 Restricted and UnrestrictedUse Soil Cleanup Objectives, December 2006. Commercial clean-up objectives from Table 375-6.8(b) and Residential clean-up objectives from Table 375-6.8(a).
2 - Achievable DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.
3 - CP-51/ Soil Cleanup Guidance, October 2010.
4 - EPA Regional Screening Levels Resident Soil Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non-cancer hazardous index

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: Metals (SW6010C and SW7471B)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[1]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Aluminum	7429-90-5	2,000	18	31	300
Antimony	7440-36-0	3	3.14	8	20
Arsenic	7440-38-2	25	4.41	12	25
Barium	7440-39-3	1,000	0.576	1.5	10
Beryllium	7440-41-7	3[4]	0.474	0.5	1.5
Boron	7440-42-8	1,000	4.37	7	100
Cadmium	7440-43-9	5	0.452	0.8	5
Calcium	7440-70-2	NA	34.5	80	1000
Chromium	7440-47-3	50	0.663	1.5	15
Cobalt	7440-48-4	47[3]	1.23	2.0	15
Copper	7440-50-8	200	1.36	3.5	15
Iron	7439-89-6	300	22	30	100
Lead	7439-92-1	25	2.61	5.0	15
Magnesium	7439-95-4	35,000	10.7	25	500

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: Metals (SW6010C and SW7471B)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (µg/L)[1]	Achievable Laboratory Limits[1]		
			DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Manganese	7439-96-5	300	0.253	0.50	10
Molybdenum	7439-98-7	780[3]	3.13	7.0	30
Nickel	7440-02-0	100	1.29	3.0	40
Potassium	7440-09-7	NA	237	250	3000
Selenium	7782-49-2	10	4.86	12	22
Silver	7440-22-4	50	0.933	2.0	15
Sodium	7440-23-5	20,000	91.6	250	5000
Thallium	7440-28-0	0.5[4]	4.91	12	40
Vanadium	7440-62-2	NA	1.11	2.5	15
Zinc	7440-67-7	2,000[4]	4.53	13	150
Mercury	7439-97-6	0.7	0.027	0.080	0.2

1 - New York State Department of Environmental Conservation (NYSDEC) Division of Water Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations, 6 NYCRR Part 703, NYSDEC, August 1999.
2 - Laboratory-specific DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method. DLs may be subject to update.
3 - EPA Regional Screening Levels Tap-water Supporting Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non-cancer hazardous index.
4 - New York State Department of Environmental Conservation (NYSDEC) Division of Water Technical and Operational Guidance Series, Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations, NYSDEC, June 1998.
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: Metals (SW6010C, SW7471B, and 7196A)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Aluminum	7429-90-5	770,000[4]	1.55	3.0	50
Antimony	7440-36-0	310[4]	0.38	0.60	2
Arsenic	7440-38-2	16/16[1]	0.66	1.0	2.5
Barium	7440-39-3	400/350[1]	0.076	0.18	2
Beryllium	7440-41-7	590/14[1]	0.033	0.070	0.5
Boron	7440-42-8	160,000[4]	980	1,000	10,000
Cadmium	7440-43-9	9.3/2.5[1]	0.041	0.10	0.5
Calcium	7440-70-2	NA/NA	14.1	20	100
Chromium	7440-47-3	36	0.058	0.15	3.5
Hexavalent Chromium	7440-47-3	22[1]	0.058	0.15	3.5
Cobalt	7440-48-4	NA/30[3]	0.1	0.20	1
Copper	7440-50-8	270/270[1]	0.217	0.50	5
Iron	7439-89-6	NA/2,000[3]	3.8	5.0	80
Lead	7439-92-1	1,000/400[1]	0.27	0.80	0.9
Magnesium	7439-95-4	NA/NA	3.7	5.0	30
Manganese	7439-96-5	10,000/2,000[1]	0.1	0.15	4.5

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: Metals (SW6010C, SW7471B, and 7196A)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Molybdenum	7439-98-7	3,900[4]	0.26	0.50	2.5
Nickel	7440-02-0	310/140[1]	0.123	0.20	4
Potassium	7440-09-7	NA/NA	41	50	300
Selenium	7782-49-2	1,500/36[1]	0.86	1.2	3
Silver	7440-22-4	1,500/36[1]	0.16	0.20	1.5
Sodium	7440-23-5	NA/NA	59	100	500
Thallium	7440-28-0	7.8[4]	0.65	1.2	3
Vanadium	7440-62-2	NA/100[3]	0.094	0.20	2
Zinc	7440-67-7	10,000/2,200	0.398	0.80	8
Mercury	7439-97-6	2.8/0.81[1]	5.53	13.33	17

1 - NYSDEC 60 NYCRR Part 375 Restricted and UnrestrictedUse Soil Cleanup Objectives, December 2006. Commercial clean-up objectives from Table 375-6.8(b) and Residential clean-up objectives from Table 375-6.8(a).
2 - Achievable DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.
3 - Determination of Soil Cleanup Objectives and Cleanup Levels, Technical and Administrative Guidance Memorandum (TAGM) #4046, NYSDEC, January 1994.
4 - EPA Regional Screening Levels Resident Soil Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non-cancer hazardous index.
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: Pesticides (SW8081B)

Concentration Level: Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[1]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Aldrin	309-00-2	680/19[1]	0.251	0.46	1.7
alpha-BHC	319-84-6	3,400/97[1]	0.214	0.46	1.7
beta-BHC	319-85-7	3,000/72[1]	0.664	0.69	1.7
delta-BHC	319-86-8	500,000/100,000[1]	0.401	0.69	1.7
gamma-BHC (Lindane)	58-89-9	9,200/280[1]	0.464	0.69	1.7
alpha-Chlordane	5103-71-9	24,000/910[1]	0.323	0.46	1.7
gamma-Chlordane	5103-74-2	NA/540[3]	0.266	0.69	1.7
4,4'-DDD	72-54-8	92,000/2,600[1]	0.546	0.69	1.7
4,4'-DDE	72-55-9	62,000/1,800[1]	0.238	0.46	1.7
4,4'-DDT	50-29-3	47,000/1,700[1]	0.59	0.69	2.0
Dieldrin	60-57-1	1,400/39[1]	0.21	0.46	1.7
Endosulfan I	959-98-8	200,000/4,800[1]	0.176	0.46	1.7
Endosulfan II	33213-65-9	200,000/4,800[1]	0.287	0.46	1.7
Endosulfan sulfate	1031-07-8	200,000/4,800[1]	0.276	0.46	1.7

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: Pesticides (SW8081B)

Concentration Level: Medium

Analyte	CAS Number	Project Action Limit (µg/kg) Commercial Use/Residential Use	Achievable Laboratory Limits[1]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
Endrin	72-20-8	89,000/2,200[1]	0.306	0.46	1.7
Endrin aldehyde	7421-93-4	NA/NA	0.171	0.46	1.7
Endrin ketone	53494-70-5	NA/NA	0.489	0.69	1.7
Heptachlor	76-44-8	15,000/420[1]	0.214	0.46	1.7
Heptachlor epoxide	1024-57-3	NA/77[3]	0.426	0.69	1.7
Methoxychlor	72-43-5	NA/100,000[3]	0.45	0.69	3.3
Toxaphene	8001-35-2	4.4[4]	15.8	27	170

1 - NYSDEC 60 NYCRR Part 375 Restricted and Unrestricted Use Soil Cleanup Objectives, December 2006. Commercial clean-up objectives from Table 375-6.8(b) and Residential clean-up objectives from Table 375-6.8(a).
2 - Achievable DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.
3 - CP-51/ Soil Cleanup Guidance, October 2010.
4 - Determination of Soil Cleanup Objectives and Cleanup Levels, Technical and Administrative Guidance Memorandum (TAGM) #4046, NYSDEC, January 1994.
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: PCBs (SW8082A)

Concentration Level: Medium

Analyte	CAS Number	Project Action Limit (µg/kg)[1] Commercial Use/Residential Use	Achievable Laboratory Limits[2]		
			DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
PCB – 1016	12674-11-2	1,000/1,000	5.09	10	33
PCB – 1221	11104-28-2	1,000/1,000	15.6	20	33
PCB – 1232	11141-16-5	1,000/1,000	5.12	10	33
PCB – 1242	53469-21-9	1,000/1,000	9.12	10	33
PCB – 1248	12672-29-6	1,000/1,000	5.61	10	33
PCB – 1254	11097-69-1	1,000/1,000	5.52	10	33
PCB – 1260	11096-82-5	1,000/1,000	2.65	10	33

1 - NYSDEC 60 NYCRR Part 375 Restricted and Unrestricted Use Soil Cleanup Objectives, December 2006. Commercial clean-up objectives from Table 375-6.8(b) and Residential clean-up objectives from Table 375-6.8(a).
2 - Achievable DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: Landfill Leachate Indicators (SW9056, 351.2, 350.2, 410.4, 405.1, SW9060, SM2540C, SM2320B, 130.2, 110.2, SW9066, SW9012, and SW6010B)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/L)[1]	Achievable Laboratory Limits[1]		
			DL (mg/L)	LOD (mg/L)	LOQ (mg/L)
Bromide	24959-67-9	2[4]	0.113	0.20	0.5
Chloride	16887-00-6	250[4]	0.254	0.50	3
Fluoride	16984-48-8	1.5	0.06	0.10	1
Nitrate as N	14797-55-8	10	0.042	0.10	0.5
Nitrate + Nitrite as N	STL00217	10	0.042	0.10	0.5
Nitrite as N	14797-65-0	1	0.049	0.10	0.5
Orthophosphate as P	14265-44-2	NA	0.187	NA	0.5
Sulfate	14808-79-8	250	0.232	0.50	5
Total Organic Carbon	7440-44-0	NA	0.155	0.40	1.0
Total Phenols	64743-03-9	NA	0.009	0.005	0.02
Cyanide	57-12-5	0.2	0.002	0.002	0.010
Total Dissolved Solids	STL00242	500	4.70	10	10
Hardness	STL00009	NA	1.3	1.5	5.0
Alkalinity	STL00171	NA	1.07	2.0	5.0

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: Landfill Leachate Indicators (SW9056, 351.2, 350.2, 410.4, 405.1, SW9060, SM2540C, SM2320B, 130.2, 110.2, SW9066, SW9012, and SW6010B)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/L)[1]	Achievable Laboratory Limits[1]		
			DL (mg/L)	LOD (mg/L)	LOQ (mg/L)
BOD	STL00311	NA	0.236	0.6	2.0
Total Kjeldahl Nitrogen (TKN)	STL00296	1	0.18	0.5	1.0
Color	STL00153	15	N/A	5 pcu	5 pcu
COD	STL00070	NA	4.06	10	20

1 - New York State Department of Environmental Conservation (NYSDEC) Division of Water Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations, 6 NYCRR Part 703, NYSDEC, August 1999
2 - Laboratory-specific DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method. DLs may be subject to update.
3 - EPA Regional Screening Levels Tap-water Supporting Table, November 2012. Value used reflects most stringent value of either the carcinogenic target risk or non-cancer hazardous index
4 - New York State Department of Environmental Conservation (NYSDEC) Division of Water Technical and Operational Guidance Series, Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations, NYSDEC, June 1998
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[2]		
			DL ($\mu g/m^3$)	LOD ($\mu g/m^3$)	LOQ ($\mu g/m^3$)
Dichlorodifluoromethane	75-71-8	NA	0.099	0.64	2.5
Freon 22	75-45-6	NA	0.081	0.46	1.8
1,2-Dichlorotetrafluoroethane	76-14-2	NA	0.14	0.56	1.4
Chloromethane	74-87-3	818/263	0.070	0.08	1.0
n-Butane	106-97-8	NA	0.052	0.1	1.2
Vinyl chloride	75-01-4	NA	0.023	0.2	0.51
1,3-Butadiene	106-99-0	NA	0.055	0.09	0.44
Bromomethane	74-83-9	NA	0.10	0.16	0.78
Chloroethane	75-00-3	NA	0.087	0.11	1.3
Bromoethene(Vinyl Bromide)	593-60-2	NA	0.083	0.17	0.87

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[1]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
Trichlorofluoromethane	75-69-4	NA	0.12	0.73	1.1
Freon TF	76-13-1	NA	0.15	0.31	1.5
1,1-Dichloroethene	75-35-4	NA	0.34	0.32	0.79
Acetone	67-64-1	NA	0.95	0.31	12
Isopropyl alcohol	67-63-0	NA	0.19	0.32	12
Carbon disulfide	75-15-0	20,440/2,440	0.062	0.62	1.6
3-Chloropropene	107-05-1	NA	0.15	0.13	1.6
Methylene Chloride	75-09-2	1,740/1,740	0.080	0.14	1.7
tert-Butyl alcohol	75-65-0	NA	0.12	0.61	15
Methyl tert-butyl ether	1634-04-4	87,600/8,760	0.054	0.14	0.72
trans-1,2-Dichloroethene	156-60-5	NA	0.091	0.32	0.79
n-Hexane	110-54-3	20,440/2,044	0.070	0.28	0.70

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[1]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
1,1-Dichloroethane	75-34-3	NA	0.093	0.53	0.81
Methyl Ethyl Ketone	78-93-3	146,000/14,600	0.074	0.12	1.5
cis-1,2-Dichloroethene	156-59-2	1,022/102	0.33	0.16	0.79
1,2-Dichloroethene, Total	540-59-0	NA	0.091	0.16	0.79
Chloroform	67-66-3	36/36	0.12	0.39	0.98
Tetrahydrofuran	109-99-9	NA	0.086	0.12	15
1,1,1-Trichloroethane	71-55-6	146,000/14,600	0.11	0.71	1.1
Cyclohexane	110-82-7	175,200/17,520	0.065	0.45	0.69
Carbon tetrachloride	56-23-5	55/55	0.082	0.82	1.3
2,2,4-Trimethylpentane	540-84-1	NA	0.070	0.61	0.93
Benzene	71-43-2	105/88	0.058	0.13	0.64
1,2-Dichloroethane	107-06-2	31/31	0.073	0.32	0.81

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[1]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
n-Heptane	142-82-5	NA	0.070	0.16	0.82
Trichloroethene	79-01-6	409/41	0.049	0.43	1.1
Methyl methacrylate	80-62-6	NA	0.066	0.16	2.0
1,2-Dichloropropane	78-87-5	NA	0.11	0.18	0.92
1,4-Dioxane	123-91-1	NA	0.25	1.2	18
Bromodichloromethane	75-27-4	NA	0.080	0.54	1.3
cis-1,3-Dichloropropene	10061-01-5	NA	0.059	0.18	0.91
methyl isobutyl ketone	108-10-1	87,600/8,760	0.14	0.33	2.0
Toluene	108-88-3	146,000/14,600	0.053	0.15	0.75
trans-1,3-Dichloropropene	10061-02-6	NA	0.068	0.09	0.91
1,1,2-Trichloroethane	79-00-5	NA	0.087	0.22	1.1
Tetrachloroethene	127-18-4	139/102	0.10	0.27	1.4

QAPP Worksheet #15 -- Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[1]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
Methyl Butyl Ketone (2-Hexanone)	591-78-6	NA	0.16	0.53	2.0
Dibromochloromethane	124-48-1	NA	0.094	0.68	1.7
1,2-Dibromoethane	106-93-4	NA	0.11	0.31	1.5
Chlorobenzene	108-90-7	NA	0.060	0.37	0.92
Ethylbenzene	100-41-4	743/743	0.065	0.35	0.87
m,p-Xylene	179601-23-1	2,920/292	0.096	0.69	2.2
Xylene, o-	95-47-6	2,920/292	0.069	0.35	0.87
Xylene (total)	1330-20-7	2,920/292	0.069	0.35	0.87
Styrene	100-42-5	29,200/2,920	0.047	0.34	0.85
Bromoform	75-25-2	NA	0.074	0.41	2.1

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[2]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
Cumene	98-82-8	NA	0.054	0.39	0.98
1,1,2,2-Tetrachloroethane	79-34-5	NA	0.076	0.89	1.4
n-Propylbenzene	103-65-1	NA	0.064	0.98	0.98
4-Ethyltoluene	622-96-8	NA	0.074	0.64	0.98
1,3,5-Trimethylbenzene	108-67-8	175/18	0.093	0.64	0.98
2-Chlorotoluene	95-49-8	NA	0.067	0.67	1.0
tert-Butylbenzene	98-06-6	NA	0.060	0.71	1.1
1,2,4-Trimethylbenzene	95-63-6	175/18	0.10	0.64	0.98
sec-Butylbenzene	135-98-8	NA	0.082	0.71	1.1
4-Isopropyltoluene	99-87-6	NA	0.11	0.71	1.1
1,3-Dichlorobenzene	541-73-1	3,212/321	0.11	0.78	1.2

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Matrix: Vapor

Analytical Group: VOCs (TO-15)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ($\mu g/m^3$)[1] Sub-slab vapor/indoor vapor screening values	Achievable Laboratory Limits[2]		
			DL ($\mu g/m^3$)	LOD (ppbv)	LOQ ($\mu g/m^3$)
1,4-Dichlorobenzene	106-46-7	23,360/2,336	0.11	0.78	1.2
Benzyl chloride	100-44-7	NA	0.11	0.67	1.0
n-Butylbenzene	104-51-8	NA	0.12	0.71	1.1
1,2-Dichlorobenzene	95-50-1	31/31	0.16	0.78	1.2
1,2,4-Trichlorobenzene	120-82-1	NA	0.22	0.96	3.7
Hexachlorobutadiene	87-68-3	NA	0.31	2.1	2.1
Naphthalene	91-20-3	NA	0.20	1.7	2.6

[1]Air Force calculated sub-slab screening values/indoor screening values.
[2] Laboratory-specific DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method. DLs may be subject to update.
NA – PAL is not available, any detection of an analyte with no PAL will be evaluated when applicable.

QAPP Worksheet #16 – Project Schedule / Timeline Table

The project schedule is provided in Appendix C.

QAPP Worksheet #17 – Sampling Design and Rationale

Sampling Approach:

The former Griffiss AFB site location map is provided on Figure 17-1. Sample locations for the Landfill AOCs, SVI Mitigation sites, On-base Groundwater AOCs, SVE System Site, LUC/IC Site Closure AOCs, and Petroleum Spill Sites are illustrated in Figures 17-2 through 17-17.

Activities at the sites listed above will include the collection of groundwater, surface water, indoor air, outdoor air, sub-slab vapor, soil gas, and soil sampling.

Sampling Design and Rationale

Additional LTM for groundwater will be conducted at the petroleum spill sites, on-base groundwater AOC, and landfill AOCs. Groundwater samples will be collected at existing monitoring wells at the Landfill AOCs (Figures 17-2 through 17-6). Groundwater samples will also be collected at existing monitoring wells and newly installed monitoring wells at the petroleum spill sites (Figures 17-9 through 17-13). Collection of the groundwater samples will be conducted using the low flow sampling and bailer sampling methods. The sampling will be conducted at a network of monitoring wells that achieves the optimal coverage of the potential and residual groundwater contamination. All field parameters collected during sampling (Appendix A) will also be used to assess and document groundwater flow and water quality parameters. The groundwater flow will also be used to generate contamination plume figures provided in the future LTM Reports

Surface water sampling will be conducted at one petroleum spill site, and five landfill AOCs. Surface water will be collected at three locations at the Apron 2 petroleum spill site (Figure 17-11). Samples will be analyzed for VOCs. Surface water will also be collected at six locations for Landfill 6 (including two leachate sampling locations), three locations for Landfill 1, 2/3, and 5, and two locations for Landfill 7. Sample analysis includes VOCs, and landfill leachate indicators. Samples will be collected using the grab sampling method. The sample results will be used to confirm contamination data trends and to delineate the potential impacts from upgradient sites.

Two indoor air samples, one outdoor air sample, and three sub-slab vapor sample will be collected semi-annually at each SVI mitigation system (Figures 17-7 and 17-8). The samples will be analyzed for VOCs to evaluate the effectiveness of the SVI mitigation systems in mitigating the potential for SVI.

QAPP Worksheet #17 – Sampling Design and Rationale

Two indoor air samples, one outdoor air sample, and three sub-slab vapor samples will be collected quarterly at the SVE system associated with ST006 Building 101 AOC (Figure 17-14). The samples will be analyzed for VOCs

Soil sampling will be conducted at DP012 Building 301 AOC (Figure 17-15) and AOI 72 (Figure 17-16) to confirm that the removal actions conducted at the sites removed all contamination above residential use soil cleanup objectives. Soil sampling will be conducted at DP013 Building 255 AOC (Figure 17-17) to confirm that all residual hexavalent chromium levels below above residential use soil cleanup objectives from 0 to 2 ft bgs. Concrete sampling will be conducted at Building 211 (Figure 17-18) to determine if the site can be closed with no restrictions. The samples will be analyzed for pesticides and metals at DP012 Building 301 AOC (5 samples), VOCs at AOI 72 (2 samples), and DP013 Building 255 AOC (2 samples). Concrete samples will be analyzed for mercury at Building 211 (1 sample). All field parameter measurements will be documented in the daily chemical QC reports issued as part of the LTM/Sampling Reports. At monitoring wells, the groundwater elevations will be used to delineate the contaminant plumes and groundwater flows for figure product and recommendation support.

QAPP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table for LTM and O&M Samples

Sampling Location	Matrix	Depth (feet)	Analytical Group	Concentration Level	Number of Samples	Sampling SOP Reference[1]	Rationale for Sampling Location
LF001 (Landfill 1)	Groundwater	0 - 30	VOCs	Low-to-Medium	35	SOP No. 2	See Worksheet #17
LF001 (Landfill 1)	Groundwater	0 - 30	Landfill leachate indicators	Low-to-Medium	55	SOP No. 2	See Worksheet #17
LF001 (Landfill 1)	Surface Water	N/A	VOCs/Landfill leachate indicators	Low-to-Medium	15	SOP No. 3	See Worksheet #17
LF002 (Landfill 2/3)	Groundwater	5 - 35	Landfill leachate indicators	Low-to-Medium	20	SOP No. 2	See Worksheet #17
LF002 (Landfill 2/3)	Surface Water	N/A	Landfill leachate indicators	Low-to-Medium	15	SOP No. 3	See Worksheet #17
LF003 (Landfill 7)	Groundwater	20 - 30	Landfill leachate indicators	Low-to-Medium	20	SOP No. 2	See Worksheet #17
LF003 (Landfill 7)	Surface Water	N/A	Landfill leachate indicators	Low-to-Medium	6	SOP No. 3	See Worksheet #17
LF007 (Landfill 5)	Groundwater	15 - 20	Landfill leachate indicators	Low-to-Medium	15	SOP No. 2	See Worksheet #17
LF007 (Landfill 5)	Surface Water	N/A	Landfill leachate indicators	Low-to-Medium	9	SOP No. 3	See Worksheet #17
LF009 (Landfill 6)	Groundwater	0 - 100	VOCs	Low-to-Medium	35	SOP No. 2	See Worksheet #17
LF009 (Landfill 6)	Groundwater	0 - 100	Landfill leachate indicators	Low-to-Medium	95	SOP No. 2	See Worksheet #17

QAPP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table for LTM and O&M Samples

Sampling Location	Matrix	Depth (feet)	Analytical Group	Concentration Level	Number of Samples	Sampling SOP Reference[1]	Rationale for Sampling Location
LF009 (Landfill 6)	Surface Water	N/A	VOCs /Landfill leachate indicators	Low-to-Medium	15	SOP No. 3	See Worksheet #17
LF009 (Landfill 6)	Surface Water	N/A	Landfill leachate indicators	Low-to-Medium	15	SOP No. 3	See Worksheet #17
SD052 (SVI Systems -Building 774)	Indoor air, outdoor air, and sub-slab vapor	N/A	VOCs	Low-to-Medium	22	SOP No. 6	See Worksheet #17
SD052 (SVI Systems -Building 776)	Indoor air, outdoor air, and sub-slab vapor	N/A	VOCs	Low-to-Medium	22	SOP No. 6	See Worksheet #17
SD052 (SVI Systems -Building 785)	Indoor air, outdoor air, and sub-slab vapor	N/A	VOCs	Low-to-Medium	22	SOP No. 6	See Worksheet #17
SD052 (SVI Systems -Building 786)	Indoor air, outdoor air, and sub-slab vapor	N/A	VOCs	Low-to-Medium	22	SOP No. 6	See Worksheet #17
SS054 (Building 781)	Groundwater	40 - 60	VOCs	Medium-to-High	170	SOP No. 2	See Worksheet #17
SS063 (Apron 1)	Groundwater	20 – 30	VOCs	Medium-to-High	54	SOP No. 2	See Worksheet #17

QAPP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table for LTM and O&M Samples

Sampling Location	Matrix	Depth (feet)	Analytical Group	Concentration Level	Number of Samples	Sampling SOP Reference[1]	Rationale for Sampling Location
SS064 (Apron 2)	Groundwater	20 - 30	VOCs	Medium-to-High	84	SOP No. 2	See Worksheet #17
SS064 (Apron 2)	Surface water	0 - 1	VOCs	Low	48	SOP No. 3	See Worksheet #17
SS067 (Building 7001)	Groundwater	20 - 30	VOCs	Medium-to-High	32	SOP No. 2	See Worksheet #17
SS068 (Building 789)	Groundwater	20 - 30	VOCs	Medium-to-High	120	SOP No. 2	See Worksheet #17
ST006 Building 101 AOC	Indoor air, outdoor air, and sub-slab vapor	N/A	VOCs	Low	40	SOP No. 6	See Worksheet #17
DP012 Building 301 AOC	Soil	0-4	Pesticides and metals	Low	7	SOP No. 1	See Worksheet #17
DP013 Building 255 AOC	Soil	0-2	Hexavalent chromium and metals	Low	2	SOP No. 1	See Worksheet #17
AOI 72	Soil	6	VOCs	Low	3	SOP No. 1	See Worksheet #17
Building 211	Soil	0-1	Mercury	Low	1	SOP No. 1	See Worksheet #17

[1]Specify the appropriate letter or number from the Project Sampling SOP References table (Worksheet #21).

QAPP Worksheet #19 – Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Preparation and Analytical Method / SOP Reference[1]	Sample Volume	Containers	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation, analysis)
Water	Metals	Low	DV-MT-0019 & DV-MT-0021 / DV-IP-0010	1, 250mL, HDPE	100 mL	HNO_3, pH < 2; Cool < 6°C	180 days
Soil	Metals	Low	DV-MT-0019 & DV-MT-0021 / DV-IP-0015 / SA-GE-001	1, 4oz, glass jar	20 grams	Cool < 6°C	180 days
Water	Metals	Low	DV-MT-0017	1, 250mL, HDPE	40 mL	HNO_3, pH < 2; Cool < 6°C	28 days
Soil	Metals	Low	DV-MT-0016 & DV-MT-0023	1, 4oz, glass jar	5 grams	Cool < 6°C	28 days
Water	General Chemistry	Low	DV-WC-0006	1, 250mL amber glass jar	100 mL	H_2SO_4, pH < 2; Cool < 6°C	28 days
Water	General Chemistry	Low	DV-WC-0040	1 Liter amber glass jar	1000 mL	H_2SO_4, pH < 2; Cool < 6°C	28 days
Water	General Chemistry	Low	DV-WC-0020	1, 50mL, HDPE	15 mL	Cool < 6°C	48 Hours (NO_2, NO_3, & OPO_4) / 28 Days (Fl, Cl, Br, SO_4)
Water	General Chemistry	Low	DV-WC-0082	1, 250mL, HDPE	100 mL	NaOH, pH >12; Cool < 6°C	14 Days

QAPP Worksheet #19 – Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Preparation and Analytical Method / SOP Reference[1]	Sample Volume	Containers	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation, analysis)
Water	General Chemistry	Low	DV-WC-0060	One 100mL, HDPE	25 mL	HNO_3, pH < 2; Cool < 6°C	180 days
Water	MS VOA	Low-to-Medium	DV-MS-0010	Three 40mL glass VOA Vials	40 ml glass VOA vial	<6°C; adjust pH <2; 0.008% Na2S2O3[4]	14 days, Preserved; 7 days, Unpreserved
Soil	MS VOA	Low-to-Medium	DV-MS-0010	Two 25g EnCore™	50 grams	DI water/frozen or Methanol; <6°C	14 days, Preserved; 7 days, Unpreserved
Water	MS Semi VOA	Low	DV-MS-0012 / DV-OP-0006, DV-OP-0007, & DV-OP-0008	Two 1 liter, amber	2000 mL	Cool < 6°C	7 days to extract; 40 days from extract
Soil	MS Semi VOA	Low	DV-MS-0012 / DV-OP-0007 & DV-OP-0010	One 4oz, glass jar	60 grams	Cool < 6°C	14 days to extract; 40 days from extract
Soil	GC Semi VOA	Low	DV-GC-0020 & DV-GC-0026 / DV-OP-0007	One 4oz, glass jar	60 grams	Cool < 6°C	14 days to extract; 40 days from extract
Soil	GC Semi VOA	Low	DV-GC-0021 & DV-GC-0030 / DV-OP-0007	One 4oz, glass jar	60 grams	Cool < 6°C	14 days to extract; 40 days from extract

QAPP Worksheet #19 – Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Preparation and Analytical Method / SOP Reference[1]	Sample Volume	Containers	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation, analysis)
Soil Vapor	VOCs	Low-to-Medium	TO-15/005(BR-AT-004)	200 mL	6 Liter Summa Canister	NA	30 days

[1] Refer to the Analytical SOP References table (Worksheet #23).
[2] Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.
[3] The minimum sample size is based on analysis allowing for sufficient sample for reanalysis. Additional volume is needed for the laboratory Matrix Spike/Matrix Spike Duplicate sample analysis.
[4] Free Chlorine must be removed by the appropriate addition of Na2S2O3. This preservation is not necessary if free chlorine is not present in the groundwater.
[5] If hydrocarbons within the boiling point range of nC6 and nC12 are suspected, soil samples should be collected in pre-weighed VOA vials with PFTE lined caps.

QAPP Worksheet #20 – Field Quality Control Sample Summary Table for Groundwater/Soil Samples

Sample Location	Matrix	Analytical Group	Conc. Level	Preparation and Analytical SOP[1]	No. of Samples[2]	No. of Field Duplicate Samples[3]	No. of MS/ MSD[4]	No. of Blanks (Trip)[5]	Total No. of Samples
LF001 (Landfill 1)	Groundwater	VOCs	Low-to-Medium	DV-MS-0010	28	2	1	4	
LF001 (Landfill 1)	Groundwater	Landfill leachate indicators	Low-to-Medium	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	62	6	3	0	
LF001 (Landfill 1)	Surface Water	VOCs /Landfill leachate indicators	Low	DV-MS-0010, DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	15	0 - 1	0	4	
LF002 (Landfill 2/3)	Groundwater	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	18	1- 2	1	0	
LF002 (Landfill 2/3)	Surface Water	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	9	0 - 1	1	0	

QAPP Worksheet #20 – Field Quality Control Sample Summary Table for Groundwater/Soil Samples

Sample Location	Matrix	Analytical Group	Conc. Level	Preparation and Analytical SOP[1]	No. of Samples[2]	No. of Field Duplicate Samples[3]	No. of MS/ MSD[4]	No. of Blanks (Trip)[5]	Total No. of Samples
LF003 (Landfill 7)	Groundwater	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	24	2	1	0	
LF003 (Landfill 7)	Surface Water	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	9	0 – 1	0	0	
LF007 (Landfill 5)	Groundwater	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	15	0 - 1	1	0	
LF007 (Landfill 5)	Surface Water	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	9	0 - 1	0	0	
LF009 (Landfill 6)	Groundwater	VOCs	Low-to-Medium	DV-MS-0010	28	1 – 2	1	4	
LF009 (Landfill 6)	Groundwater	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020,	76	7 - 8	3	0	

QAPP Worksheet #20 – Field Quality Control Sample Summary Table for Groundwater/Soil Samples

Sample Location	Matrix	Analytical Group	Conc. Level	Preparation and Analytical SOP[1]	No. of Samples[2]	No. of Field Duplicate Samples[3]	No. of MS/ MSD[4]	No. of Blanks (Trip)[5]	Total No. of Samples
				DV-WC-0040, DV-WC-0082, DV-WC-0060					
LF009 (Landfill 6)	Surface Water	VOCs/Landfill leachate indicators	Low	DV-MS-0010, DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	15	0 – 1	0	4	
LF009 (Landfill 6)	Surface Water	Landfill leachate indicators	Low	DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060	15	0 - 1	0	0	
SD052 (SVI Systems - Building 774)	SVI Vapor	VOCs	Low	BR-AT-004	22	2	1	5	
SD052 (SVI Systems - Building 776)	SVI Vapor	VOCs	Low	BR-AT-004	22	2	1	5	
SD052 (SVI Systems - Building 785)	SVI Vapor	VOCs	Low-to-Medium	BR-AT-004	22	2	1	5	

QAPP Worksheet #20 – Field Quality Control Sample Summary Table for Groundwater/Soil Samples

Sample Location	Matrix	Analytical Group	Conc. Level	Preparation and Analytical SOP[1]	No. of Samples[2]	No. of Field Duplicate Samples[3]	No. of MS/ MSD[4]	No. of Blanks (Trip)[5]	Total No. of Samples
SD052 (SVI Systems - Building 786)	SVI Vapor	VOCs	Low-to-Medium	BR-AT-004	22	2	1	1	
SS054 (Building 781)	Groundwater	VOCs	High	DV-MS-0010	170	17	8	20	
SS063 (Apron 1)	Groundwater	VOCs	High	DV-MS-0010	54	5 - 6	3	12	
SS064 (Apron 2)	Groundwater	VOCs	High	DV-MS-0010	84	8 - 9	4	16	
SS064 (Apron 2)	Surface water	VOCs	High	DV-MS-0010	48	4 - 5	2 - 3	16	
SS067 (Building 7001)	Groundwater	VOCs	High	DV-MS-0010	165	16 - 17	9	20	
SS068 (Building 789)	Groundwater	VOCs	High	DV-MS-0010	120	12	6	20	
ST006 Building 101 AOC	SVI Vapor	VOCs	Low	BR-AT-004	40	8	4	5	52
DP012 Building 301 AOC	Soil	Pesticides and Metals	Low	DV-GC-0020 / DV-MT-0019 / DV-MT-0021 / DV-IP-0010	5	1	0	0	6
DP013 Building 255	Soil	Hexavalent chromium and Metals	Low	SA-GE-001 / DV-MT-0019 /	2	0	0	1	3

QAPP Worksheet #20 – Field Quality Control Sample Summary Table for Groundwater/Soil Samples

Sample Location	Matrix	Analytical Group	Conc. Level	Preparation and Analytical SOP[1]	No. of Samples[2]	No. of Field Duplicate Samples[3]	No. of MS/ MSD[4]	No. of Blanks (Trip)[5]	Total No. of Samples
AOC				DV-MT-0021 / DV-IP-0010					
AOI 72	Soil	VOCs	Low	DV-MS-0010	2	1	0	1	4
Building 211	Soil	Mercury	Low	DV-MT-0023	1	0	0	0	0

[1] Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

[2] The number of samples collected may vary depending on field conditions.

[3] Total numbers of Field Duplicate Samples will meet project goal of 10%.

[4] Total MS/MSD Samples will meet project goal of 5%.

[5] Trip blank samples will be included in each cooler containing aqueous VOCs.

Ambient blanks will be collected each day that VOC samples are collected.

Equipment blanks will be collected from each type of non-disposable, decontaminated sampling device.

QAPP Worksheet #21 – Project Sampling SOP References Table

SOPs are located in Appendix A.

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP No. 1	Soil Sampling	FPM	Hand Auger or Direct Push Rig	N	Includes descriptions and procedures for surface and subsurface soil sampling.
SOP No. 2	Groundwater Sampling	FPM	Bailer/ peristaltic pump/low flow	N	Includes descriptions and procedures for groundwater sampling.
SOP No. 3	Surface water	FPM	Grab	N	Includes descriptions and procedures for surface water sampling.
SOP No. 4	Surface Soil and Sediment	FPM	Grab/hand auger	N	Includes descriptions and procedures for sediment sampling.
SOP No. 5	Vapor	FPM	Vacuum sample canister with regulator	N	Includes descriptions and procedures for SVI and soil vapor sampling.
SOP No. 6	Sample Handling, Documentation, and Tracking	FPM	N/A	N	Includes sample packaging, shipping, and chain-of-custody requirements.
SOP No. 7	Decontamination	FPM	N/A	N	Includes descriptions and procedures for decontamination of personnel and equipment.
SOP No. 8	Monitoring Well Installation and Development	FPM	N/A	N	Includes description for the drilling, completion and development of

QAPP Worksheet #21 – Project Sampling SOP References Table

SOPs are located in Appendix A.

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
					monitoring wells.
SOP No. 9	Monitoring Well and Boring Abandonment	FPM	N/A	N	Includes description for the plugging and abandonment of soil borings and monitoring wells.

QAPP Worksheet #22 – Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maint. Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP [1]
Photoionization detector (PID)	Calibrated to 100 parts per million (ppm) using 100 ppm isobutylene	Clean unit weekly	Check response with marking pen	Observe pump and PID response	Daily	Within 3%	Clean Lamp	Field personnel	SOP No. 1, 2 and 3.
Water Quality Measure System (YSI 556 MPS, Horiba U-52, or similar)	Daily DO with tap water. Weekly conductivity with 1.413 mS/cm conductivity standard. Weekly pH with pH 4 and 7 pH standards. Weekly ORP with 240 mV ORP solution.	Clean unit daily with Simple Green or similar and distilled water rinse.	Check response to calibration.	Daily for damaged cord, probe or controller.	Daily Weekly	95-105% 1.35-1.45 mS/cm 3.98-4.02, 6.98-7.02 230 -250 mV	Clean Probe, repeat calibration procedure	Field personnel	SOP No. 3.

[1] The Project Sampling SOP References table is found on Worksheet #21.

QAPP Worksheet #23 – Analytical SOP References Table

Laboratory SOPs are located in Appendix B

SOP Reference Number [1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
DV-IP-0010	Revision 4.7, 07/18/2012 Acid Digestion of Aqueous Samples for Metals Analysis by ICP (SW3005A)	Preparation	Metals	N/A	TA Denver	N
DV-IP-0014	Revision 3.4, 09/01/2010 Acid digestion of Aqueous Samples for Analysis by ICP-MS (SW-846 3005A, 3020A, 3050B, and EPA 200.8)	Preparation	Metals	N/A	TA Denver	N
DV-IP-0015	Revision 4, 02/3/2012 Acid Digestion of Solids (EPA 3050B)	Preparation	Metals	N/A	TA Denver	N
DV-MT-0019	Revision 1.1, 03/12/2010 ICP Analysis for Trace Elements by SW-846 Method 6010B	Definitive	Metals	ICP	TA Denver	N
DV-MT-0021	Revision 0.3, 07/13/2012 ICP Analysis for Trace Elements by SW-846 Method 6010C	Definitive	Metals	ICP	TA Denver	N

QAPP Worksheet #23 – Analytical SOP References Table

Laboratory SOPs are located in Appendix B

SOP Reference Number [1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
DV-MT-0016	Revision 2.3 09/01/2010 Mercury in Solids by Cold Vapor Atomic Asorption (SW-846 7471A)	Definitive	Metals (Mercury)	CVAA	TA Denver	N
DV-MT-0017	Revision 1.2 07/13/2012 Mercury in Water by Cold Vapor Atomic Asorption (CVAA) (SW-846 7470A)	Definitive	Metals (Mercury)	CVAA	TA Denver	N
DV-MT-0023	Revision 1.2 07/13/2012 Mercury in Solids by Cold Vapor Atomic Asorption (SW-846 7471B)	Definitive	Metals (Mercury)	CVAA	TA Denver	N
DV-GC-0020	Revision 7, 10/12/2012 Chlorinated Pesticides (SW846 Method 8081A)	Definitive	Chlorinated Pesticides	GC	TA Denver	N
DV-GC-0026	Revision 0.1, 01/26/2010 Chlorinated Pesticides (SW846 Method 8081B)	Definitive	Chlorinated Pesticides	GC	TA Denver	N
DV-GC-0021	Revision 6.0, 06/15/2012	Definitive	PCBs	GC	TA Denver	N

QAPP Worksheet #23 – Analytical SOP References Table

Laboratory SOPs are located in Appendix B

SOP Reference Number [1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	Polychlorinated Biphenyls (PCBs) by GC/ECD (SW846 Method 8082)					
DV-GC-0030	Revision 0.1, 06/11/2010 Polychlorinated Biphenyls (PCBs) by GC/ECD (SW846 Method 8082A)	Definitive	PCBs	GC	TA Denver	N
DV-MS-0009	Revision 3.4, 05/31/2012 Screening for VOCs by headspace GC/FID	Definitive	Volatiles	GCMS	TA Denver	N
DV-MS-0010	Revision 9.0, 01/4/2013 Determination of Volatile Organics by Gas Chromatography and Mass Spectrometer (GC/MS) (SW846 8260B and EPA 624)	Definitive	Volatiles	GCMS	TA Denver	N
DV-MS-0011	Revision 5.2, 05/04/2010 GC/MS Analysis Based on Method 8270C and 625	Definitive	Semi-Volatiles	GCMS	TA Denver	N

QAPP Worksheet #23 – Analytical SOP References Table

Laboratory SOPs are located in Appendix B

SOP Reference Number [1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
DV-MS-0012	Revision 3, 01/4/2013 GC/MS Analysis Based on Method 8270D	Definitive	Semi-Volatiles	GCMS	TA Denver	N
DV-WC-0006	Revision 7.1, 04/19/2010 Carbon in Water (TOC, TIC, DOC, and TC) [EPA 415.1, SM 5310B & SW 9060A]	Definitive	TOC, water	Shimadzu	TA Denver	N
DV-WC-0020	Revision 7.1, 12/04/2009 Anions by Ion Chromatography (EPA 300.0, SW 9056A)	Definitive	Anions	IC	TA Denver	N
DV-WC-0023	Revision 3.2, 03/01/2010 Percent Moisture in Soils and Wastes [EPA 160.3, ASTM D2216, CLP ILM05.3]	Definitive	Moisture, soils	NA	TA Denver	N
DV-WC-0082	Revision 0.2, 06/11/2010 Total and Amenable Cyanide by SW-846 9010C, 9012B, and 9013	Definitive	Cyanide	Colorimetric	TA Denver	N
DV-WC-0040	Revision 5.2, 11/19/2010	Definitive	Ammonia	Colorimetric	TA Denver	N

QAPP Worksheet #23 – Analytical SOP References Table

Laboratory SOPs are located in Appendix B

SOP Reference Number [1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	Ammonia Nitrogen by Autoanalyzer [EPA 350.1]					
DV-WC-0060	Revision 3.2, 05/15/2010 Hardness by Titration [EPA SM2340C and SM 2340C]	Definitive	Hardness, Water	Titration	TA Denver	N
DV-OP-0006	Revision 9.0, 01/15/2013 Extraction of Aqueous Samples by Separatory Funnel, SW-846 3510C and EPA 600 Series	Preparation	Organic Prep	N/A	TA Denver	N
DV-OP-0007	Revision 7.0, 12/5/2012 Concentration and Clean-up of Organic Extracts (SW-846 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A, and EPA 600 series)	Preparation	Organic Prep	N/A	TA Denver	N
DV-OP-0008	Revision 5, 08/02/2010 Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction	Preparation	Organic Prep	N/A	TA Denver	N

QAPP Worksheet #23 – Analytical SOP References Table

Laboratory SOPs are located in Appendix B

SOP Reference Number [1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	(CLLE) by Method SW-846 3520C and Methods 625 and 607					
DV-OP-0010	Revision 3, 06/23/2010 Soxhlet Extraction of Solid Samples (SW-846 3540C)	Preparation	Organic Prep	N/A	TA Denver	N
DV-OP-0015	08/1/2012 Microwave Extraction of Solid Samples (SW-846 3546)	Preparation	Organic Prep	N/A	TA Denver	N
DV-OP-0016	12/5/2012 Ultrasonic Extraction of Solid Samples (SW-846 3550 B and C)	Preparation	Organic Prep	N/A	TA Denver	N
DV-OP-0023	Revision 1, 01/31/2012 Extraction of Aqueous Samples by Microextraction, (SW-846 3511) VOCs/SVOCs	Preparation	Organic Prep	N/A	TA Denver	N
001	Mercury by CVAA, SW-846 7471A, BR-ME-004, Rev.12,	Definitive	Mercury	CVAA	TA Burlington	No

QAPP Worksheet #23 – Analytical SOP References Table

Laboratory SOPs are located in Appendix B

SOP Reference Number [1]	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	03/05/10					
002	Metals by ICP-OES, SW846 6010B, BR-ME-005, Rev.11, 03/05/10	Definitive	Metals	ICP-OES	TA Burlington	No
003	Polychlorinated Biphenyls (PCBs) by GC/ECD, SW846 8082, BR-GC-005, Rev.9, 11/12/08	Definitive	PCB as Aroclors	GC/ECD	TA Burlington	No
004	Chlorinated Pesticides by GC/ECD, SW846 8081A, BR-GC-006, Rev.9, 05/12/09	Definitive	Pesticides	GC/ECD	TA Burlington	No
005	VOCs in Ambient Air, EPA TO-15, BR-AT-004, Rev. 7, 08/16/2012	Definitive	VOCs	GC/MS	TA Burlington	No
SA-GE-001	Measurements of using the Konelab Analyzer, Rev 2B. 02/05/2014	Definitive	Hexavalent chromium	ICP	TA Savannah	No

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
GCMS - 8260	Check of mass spectral ion intensities, i.e., Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours	Refer to criteria listed in the method SOP for Tune criteria.	Retune the instrument and verify (instrument maintenance may be needed).	Lab Manager / Analyst	DV-MS-0010
	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	SPCCs average RF ≥ 0.30 or 0.1 depending on the compound and %RSD for RFs for Calibration Check Compounds (CCCs) ≤ 30% and all other target analytes %RSD for RF < 15%.	Correct problem then repeat initial calibration	Lab Manager / Analyst	DV-MS-0010
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following five-point initial calibration	All analytes within 25% of expected value	Correct problem then repeat initial calibration	Lab Manager / Analyst	DV-MS-0010
	Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.30 or 0.1 depending on the compound; and CCCs: ≤20% difference (when using RFs) or drift (when using least squares	Correct problem then repeat initial calibration and re-analyze all samples since last	Lab Manager / Analyst	DV-MS-0010

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
			regression).	successful CCV.		
	Continuing calibration check		CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). All other target compounds ≤20%, up to 5 non-CCC target compounds may fail this requirement provided the % difference is ≤ 40%.	Continuing calibration check	Lab Manager / Analyst	DV-MS-0010
	Internal Standards (IS)	Every sample/standard and blank	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). Extracted ion current profile (EICP) area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples. (See federal programs SOP DV-QA-024P for program specific requirements)	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the technical director for advice).	Lab Manager / Analyst	DV-MS-0010

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
GCMS - 8270	Check of mass spectral ion intensities, i.e., Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours	Refer to criteria listed in the method SOP for Tune criteria, including DDT, Benzidine and Pentachlorophenol requirements.	Retune the instrument and verify (instrument maintenance may be needed).	Lab Manager / Analyst	DV-MS-0011
	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF < 15%. option (if %RSD is > 15%)– linear regression r 2 > 0.99, r ≥ 0.995.	Correct problem then repeat initial calibration If the calibration is not considered linear by either %RSD or linear regression, then correct the problem and re-calibrate.	Lab Manager / Analyst	DV-MS-0011

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP[1]
	ICV must be from a second source.	Immediately following five-point initial calibration	All analytes within 25% of expected value	Correct problem then repeat initial calibration	Lab Manager / Analyst	DV-MS-0011
	CCV	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.050; and	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.	Lab Manager / Analyst	DV-MS-0011
	Continuing calibration check		CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). All other target compounds ≤20%, up to 5 non-CCC target compounds may fail this requirement provided the % difference is ≤ 40%.	Continuing calibration check	Lab Manager / Analyst	DV-MS-0011
	Internal Standards	Every sample/standard and blank	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL	Inspect mass spectrometer and GC for malfunctions;	Lab Manager / Analyst	DV-MS-0011

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
			(sample/standard). EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples. (See federal programs SOP DV-QA-024P for program specific requirements)	mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the technical director for advice).		
ICP – 6010 and 7196	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	N/A	N/A	Lab Manager/ Analyst	DV-MT-0019, DV-MT-0021, & SA-GE-001
	ICV must be from a second source.	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration	Lab Manager/ Analyst	DV-MT-0019, DV-MT-0021, & SA-GE-001
	Calibration blank (CB)	After every continuing calibration verification	Must be <3 times the IDL or the average of 3 CB must be <3 times the IDL.	Correct problem then analyze calibration blank and previous 10 samples	Lab Manager/ Analyst	DV-MT-0019, DV-MT-0021, & SA-GE-001
	CCV	Before sample analysis, after	All analytes within 10% of expected value and RSD of	Repeat calibration and re-analyze all	Lab Manager/	DV-MT-0019, DV-MT-0021,

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
		every 10 samples, and at the end of the analysis sequence	replicate integrations <5%	samples since last successful calibration	Analyst	& SA-GE-001
	Interference check solution (ICS)	At the beginning of an analytical run	Within 20% of expected value	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples	Lab Manager/ Analyst	DV-MT-0019, DV-MT-0021, & SA-GE-001
CVAA – 7470/7471	Initial calibration (minimum five standards and a blank)	Daily initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration. If calibration fails again, re-digest the entire digestion batch.	Lab Manager/ Analyst	DV-MT-0016, DV-MT-0017 & DV-MT-0023
	ICV must be from a second source.	Immediately following initial daily calibration	Analytes within 10% of expected value	Correct problem then repeat initial calibration. If calibration fails again, re-digest the entire digestion	Lab Manager/ Analyst	DV-MT-0016, DV-MT-0017 & DV-MT-0023

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
				batch.		
	CB	Once per initial daily calibration	No analytes detected ≥ LOD	Correct problem then re-digest and re-analyze calibration and entire digestion batch	Lab Manager/ Analyst	DV-MT-0016, DV-MT-0017 & DV-MT-0023
	CCV	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value	Repeat calibration and re-analyze all samples since last successful calibration	Lab Manager/ Analyst	DV-MT-0016, DV-MT-0017 & DV-MT-0023
GC – 8081/8082/8141/8151	Minimum five-point initial calibration for all target analytes[2]	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	Linear regression correlation coefficient $r^2 \geq 0.99$, $r \geq 0.995$. RSD of CF ≤ 20%	Correct problem then repeat initial calibration	Lab Manager/ Analyst	DV-GC-0020, DV-GC-0026, DV-GC-0021, DV-GC-0030
	ICV must be from a second	Once immediately	All target analytes within 15% of expected value	Correct problem then repeat initial	Lab Manager/	DV-GC-0020, DV-GC-0026,

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
	source	following initial calibration		calibration	Analyst	DV-GC-0021, DV-GC-0030
	CCV	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value and within the RT Window[3].	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.	Lab Manager/ Analyst	DV-GC-0020, DV-GC-0026, DV-GC-0021, DV-GC-0030
	8081 Only - Breakdown check (Endrin and DDT)	Before sample analysis	Degradation ≤15% for either Endrin or DDT.	Inlet/column maintenance; repeat breakdown check and re-analyze all samples since last successful breakdown check.	Lab Manager/ Analyst	DV-GC-0020, DV-GC-0026, DV-GC-0021, DV-GC-0030
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW at the start of the run or	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. If questions, see the supervisor or technical director.	Lab Manager/ Analyst	DV-GC-0020, DV-GC-0026, DV-GC-0021, DV-GC-0030

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
		daily.				
Total Organic - 9060A	Calibration Curve – Minimum 5-point calibration	Initial calibration. Perform instrument re-calibration once per year minimum.	$r \geq 0.995$.	Recalibrate	Lab Manager/ Analyst	DV-WC-0006
	ICV must be from a second source.	Immediately following initial calibration	±10%	Recalibrate	Lab Manager/ Analyst	DV-WC-0006
	CCV	Each use, beginning, every 10 samples, end of batch	± 10%	Rerun affected samples	Lab Manager/ Analyst	DV-WC-0006
Ion Chromatograph - 9056A	Calibration Curve – Minimum 5-point calibration	Initial calibration. Perform instrument re-calibration once per year minimum.	RSD ± 10%, $r^2 \geq 0.99$, $r \geq 0.995$.	Recalibrate	Lab Manager/ Analyst	DV-WC-0020
	ICV, second	Immediately	±10%	Recalibrate	Lab	DV-WC-0020

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
	source	following initial calibration			Manager/ Analyst	
	CCV	Each use, beginning, every 10 samples, end of batch	± 10%	Rerun affected samples	Lab Manager/ Analyst	DV-WC-0020
Colorimetric Analyzer - 9012B	Initial calibration (six-point calibration standards)	Initial daily calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration	Lab Manager / Analyst	DV-WC-0082
	High and Low Distilled standard	Prepared per batch.	±10%	Re-distill and re-analyze all associated samples	Lab Manager / Analyst	DV-WC-0082
	ICV must be from a second source.	Immediately following initial daily calibration	±10%	Correct problem then repeat initial calibration	Lab Manager / Analyst	DV-WC-0082

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP[1]
Titrimetric Analyzer - SM 2340C	Standardization of titrant	Initial daily standardization prior to sample analysis.	N/A – See method SOP for standardization procedure	N/A	Lab Manager / Analyst	DV-WC-0060
	LCS/LCSD	Prepared per batch.	±10%	Re-titrate all associated samples	Lab Manager / Analyst	DV-WC-0060
Titrimetric Analyzer - SM 2320B	Standardization of titrant	Initial daily standardization prior to sample analysis.	N/A – See method SOP for standardization procedure	N/A	Lab Manager / Analyst	DV-WC-0025
	CCV	Analyzed every 10 samples.	±10%	Re-titrate all associated samples	Lab Manager / Analyst	DV-WC-0025
	LCS/LCSD	Prepared per batch.	±10%	Re-titrate all associated samples	Lab Manager / Analyst	DV-WC-0025
Colorimetric Analyzer - 350.1	Calibration Curve – Minimum five-point calibration	Initial calibration. Perform instrument re-calibration once per year minimum.	RSD ± 10%, $r^2 \geq 0.99$, $r \geq 0.995$.	Recalibrate	Lab Manager/ Analyst	DV-WC-0040

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
	ICV must be from a second source.	Immediately following initial calibration	±10%	Recalibrate	Lab Manager/ Analyst	DV-WC-0040
	CCV	Each use, beginning, every 10 samples, end of batch	± 10%	Rerun affected samples	Lab Manager/ Analyst	DV-WC-0040
GC/ECD HP5890 HP6890/5973	Initial multi-point calibration with verification, daily calibration check	Prior to sample analysis, then as required	Initial RSD ≤20% Linear Regression r ≥ 0.995 ICV 80-120 % Recovery CCV % D or drift ≤20%	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	003, 004
Leeman Labs Hydra AA	Initial multi-point calibration with verification, daily calibration check	Prior to sample analysis, then as required	Linear Regression r ≥ 0.995 ICV 90-110 % Recovery CCV % D ≤20%	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	001
Thermo ICAP 6500	Initial multi-point calibration with verification, daily calibration	Prior to sample analysis, then as required	ICAL NA ICV 90-110 % Recovery CCV % D ≤10%, % RSD between replicate integrations <	Perform Maintenance, Check Standards, Recalibrate,	Assigned Lab personnel	002

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsi-ble for CA	SOP[1]
	check		5%	Reanalyze		
GC: Agilent 6890 MS: Agilent 5973 or 5972 MSD	Initial multi-point calibration with verification, daily calibration check	Prior to sample analysis, then as required	RSD for each analyte ≤ 30% with two exceptions up to 40%	Correct problem and repeat calibration	Assigned Lab personnel	005

The Analytical SOP References table is found on Worksheet #23
1- This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2 – Method 8082, a five-point calibration is only analyzed for Aroclors 1016 and 1260.
3 - The mean of all calibrated compounds may be used, but all compounds above the 15% must be documented in a NCM

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
GC	Change septum, clean injection port, change or clip column, install new liner, replace column, filters and seals	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	CCV passes criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst	QA Manual – Section 20
GC-MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	Analyst	QA Manual – Section 20
GC-MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Response factors and chromatogram review	Instrument performance and sensitivity	As needed	Tune and CCV pass criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst	QA Manual – Section 20

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
ICP	Replace disposables, flush lines, clean injector and torch, Perform Hg alignment, check purge windows	Intensity of 1PPM Manganese STD within criteria	Check connections	Daily or as needed	Intensity of 1PPM Manganese STD within criteria	Replace, investigate injector, reanalyze	Analyst	QA Manual – Section 20
ICP	Replace pump windings and gas tanks, check standard and sample flow	Monitor ISTD counts for variation	Instrument performance and sensitivity	As needed	Monitor ISTD counts for variation	Replace windings, recalibrate and reanalyze	Analyst	QA Manual – Section 20
ICPMS	Replace disposables, clean/change nebulizer, torch, and cones	Tuning	Instrument performance and sensitivity	Daily or as needed	Tune and CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
CVAA	Replace disposables, flush lines, check lamp current and gas flow	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
Colorimetric	Replace disposable, flush lines, clean autosampler and pump rollers	Analytical standards	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
Spectrophotometer	Replace disposable, flush lines, and clean autosampler.	Analytical standards	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
Ion Chromatograph	Replace disposables, check for leaks and eluent levels, change columns and bed supports as needed, clean conductivity cell	Analytical standards	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
Shimadzu	Replace disposables, check for leaks, change copper and tin as needed, clean purging cell	Analytical standards	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
LECO	Replace Disposables and check gas flow.	Analytical standards	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
HPLC	Replace columns, DAD flow cell windows and ball-valve cartridges as needed, clean/change filters, check eluent	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
	reservoirs							
LC/MS & IC/MS/MS	Replace columns as needed, change filters and seals, clean lenses and needles, check eluent reservoirs	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	Analyst	QA Manual – Section 20
GC/ECD HP5890 HP6890/5973	Change Septa, Clean/Replace Injection Port Liner, Clip/Replace column	SW846 8081A SW846 8082 SW846 8151 SW846 8015	Pass breakdown Pass Continuing Calibration	Daily or as needed	< 15% breakdown CCV < 15%	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	003, 004
Leeman Labs Hydra AA	Torch, Nebulizer, Spray Chamber, Pump Tubing	SW846 7470A SW846 7471A	Pass calibration checks	Daily or as needed	Corr Coeff $\geq$ 0.995 90-110% Recovery	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	001
Thermo ICAP 6500	Torch, Nebulizer, Spray	SW846 6010B	Pass calibration checks	Daily or as needed	Corr Coeff $\geq$ 0.995 90-110%	Perform Maintenance, Check	Assigned Lab personnel	002

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
	Chamber, Pump Tubing				Recovery	Standards, Recalibrate, Reanalyze		
GC: Agilent 6890 MS: Agilent 5973 or 5972 MSD	Check GC / Entech Column Interface Check Nitrogen Tank Volume Check Nitrogen Valves Software and Valves Cut 2-3 inches from GC Column	TO-15	Pass Tune Pass Continuing Calibration	Daily or as needed	Tune: See TO-15 Method CCV: %D ≤ 30	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	005

QAPP Worksheet #26 – Sample Handling System

Sample Collection, Packaging, and Shipment Sample Collection (Personnel/Organization): Field Personnel / FPM and AECOM Sample Packaging (Personnel/Organization): Field Personnel / FPM and AECOM Coordination of Shipment (Personnel/Organization): FPM Technical Lead / FPM and AECOM Field Personnel Type of Shipment/Carrier: Laboratory courier/Overnight FedEx or UPS
Sample Receipt and Analysis Sample Receipt (Personnel/Organization): TBD / Test America Sample Custody and Storage (Personnel/Organization): TBD / Test America Sample Preparation (Personnel/Organization): TBD / Test America Sample Determinative Analysis (Personnel/Organization): TBD / Test America
Sample Archiving Field Sample Storage (No. of days from sample collection): 30 days Sample Extract/Digestate Storage (No. of days from extraction/digestion): 90 days Biological Sample Storage (No. of days from sample collection): N/A
Sample Disposal Personnel/Organization: TBD / Test America Number of Days from Analysis: 30 days

QAPP Worksheet #27 – Sample Custody Requirements Table

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):

See SOP No. 7.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):

See the following SOPs:

SOP #LOGIN01 Sample Receiving and Login

SOP #SOP33 Laboratory Waste Management

Sample Identification Procedures:

See SOP No. 7 and Supplemental WP.

Chain of Custody Procedures:

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	VOCs and SVOCs
Analytical Method / SOP Reference	EPA 8260B/8270C/8270D DV-MS-0010, DV-MS-0011

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>1/2RL; no common lab contaminants >RL.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>½RL; no common lab contaminants >RL.
LCS	1/Batch (20 samples)	See Table 12-1	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
MS/MSD	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits
Surrogates	Every sample	See Table 12-2	Check calculations and instrument performance; recalculate, reanalyze.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	Metals
Analytical Method / SOP Reference	EPA 6010B/6010C DV-MT-0019 & DV-MT-0021

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>½RL	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>½RL
LCS	1/Batch (20 samples)	See Table 12-1	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
MS/MSC	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits
Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition	Analyst / Section Supervisor	Accuracy/Bias/ Precision	N/A

Matrix		Water / Soil				
Analytical Group		Metals				
Analytical Method / SOP Reference		EPA 6010B/6010C DV-MT-0019 & DV-MT-0021				
Post digestion spike addition	When dilution test fails	Recovery within 25% of expected results	Correct problem then re-analyze post digestion spike addition	Analyst / Section Supervisor	Accuracy/Bias	N/A

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	Metals
Analytical Method / SOP Reference	EPA 7470A/7471A/7471B DV-MT-0016, DV-MT-0017 & DV-MT-0023

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>½RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>½RL
LCS	1/Batch (20 samples)	See Table 12-1	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
MS/MSD	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits
Dilution test; five-fold	Each preparatory batch	Five times dilution sample result must	Perform post digestion spike addition	Analyst / Section	Accuracy/Bias/ Precision	N/A

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	Metals
Analytical Method / SOP Reference	EPA 7470A/7471A/7471B DV-MT-0016, DV-MT-0017 & DV-MT-0023

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
dilution test		be ±10% of the undiluted sample result		Supervisor		
Recovery test	When dilution test fails	Recovery within 85-115% of expected results	Dilute the sample; re-analyze post digestion spike addition	Analyst / Section Supervisor	Accuracy/Bias	N/A

QAPP Worksheet #28 – QC Samples Table

Matrix	Water/ Soil
Analytical Group	SVOCs
Analytical Method / SOP Reference	EPA 8081A/8081B/8082/8082A DV-GC-0020, DV-GC-0026, DV-GC-0021, DV-GC-0030

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>½RL; no common lab contaminants >RL.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>½RL; no common lab contaminants >RL.
LSC	1/Batch (20 samples)	See Table 12-1	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
MS/MSD	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits
Surrogates	Every sample	See Table 12-2	Check calculations and instrument performance; recalculate, reanalyze.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits

QAPP Worksheet #28 – QC Samples Table

Matrix	Water/ Soil
Analytical Group	SVOCs
Analytical Method / SOP Reference	EPA 8081A/8081B/8082/8082A DV-GC-0020, DV-GC-0026, DV-GC-0021, DV-GC-0030

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Second-column confirmation	100% for all positive results Only applies to 8082 for specific programs	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.	Analyst / Section Supervisor	Accuracy/Bias	Same as for initial or primary column analysis

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	General Chemistry
Analytical Method / SOP Reference	EPA 9060A DV-WC-0006

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>½RL	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>½RL
LCS	1/Batch (20 samples)	See Table 12-1	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
MS/MSD	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits

[1] This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	General Chemistry
Analytical Method / SOP Reference	EPA 9056A DV-WC-0020

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>½RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>½RL
LCS	1/Batch (20 samples)	See Table 12-1	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
Duplicate	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag duplicate data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	±30%, historical or client specific limits
MS/MSD	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag MS/MSD data; discuss in	Analyst / Section	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	General Chemistry
Analytical Method / SOP Reference	EPA 9056A DV-WC-0020

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			narrative.	Supervisor		Control Limits

[1] This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.

QAPP Worksheet #28 – QC Samples Table

Matrix	Water / Soil
Analytical Group	General Chemistry
Analytical Method / SOP Reference	SM 2340C DV-WC-0060

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>½RL; no common lab contaminants >RL.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>½RL; no common lab contaminants >RL.
LCS	1/Batch (20 samples)	See Table 12-1	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
MS/MSD	1/Batch (20 samples)	See Table 12-1	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits

[1] This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.

QAPP Worksheet #28 – QC Samples Table

Matrix	Groundwater / Soil
Analytical Group	PCBs
Analytical Method / SOP Reference	U.S. EPA Method SW 8082

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch	No target compounds ≥ ½ RL.	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Laboratory Analyst	Contamination	No target compounds ≥ ½ RL
LCS	One per preparation/analytical batch	See Table 12-1	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst	Accuracy/ Bias	See Table 12-3
MS/MSD	Each group of field samples in an SDG or each SDG, whichever is most frequent	See Table 12-1	Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Examine the project-specific Data	Laboratory Analyst	Accuracy/Bias and Precision	See Table 12-3

QAPP Worksheet #28 – QC Samples Table

Matrix	Groundwater / Soil
Analytical Group	PCBs
Analytical Method / SOP Reference	U.S. EPA Method SW 8082

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			Quality Objectives (DQOs). Contact the client as to additional measures to be taken.			
Surrogates	Each sample, standard, blank	See Table 12-2	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If matrix effect is verified, discuss in case narrative.	Laboratory Analyst	Accuracy/Bias	See Table 12-3
LODs	Annual	Per Laboratory SOP	Reanalyze LOD	TestAmerica Laboratory	Sensitivity	Low enough to support RLs

QAPP Worksheet #28 – QC Samples Table

Matrix	Groundwater / Soil
Analytical Group	Pesticides
Analytical Method / SOP Reference	U.S. EPA Method SW 8081

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch	No target compounds ≥ ½ RL.	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Laboratory Analyst	Contamination	No target compounds ≥ ½ RL
LCS	One per preparation/analytical batch	See Table 12-1	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst	Accuracy/ Bias	See Table 12-3
MS/MSD	Each group of field samples in an SDG or each SDG, whichever is most frequent	See Table 12-1	Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Examine the project-specific DQOs.	Laboratory Analyst	Accuracy/Bias and Precision	See Table 12-3

QAPP Worksheet #28 – QC Samples Table

Matrix	Groundwater / Soil
Analytical Group	Pesticides
Analytical Method / SOP Reference	U.S. EPA Method SW 8081

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			Contact the client as to additional measures to be taken.			
Surrogates	Each sample, standard, blank	See Table 12-2	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If matrix effect is verified, discuss in case narrative.	Laboratory Analyst	Accuracy/Bias	See Table 12-3
LODs	Annual	Per Laboratory SOP	Reanalyze LOD	TestAmerica Laboratory	Sensitivity	Low enough to support RLs

QAPP Worksheet #28 – QC Samples Table

Matrix	Groundwater
Analytical Group	Landfill Leachate Indicators
Analytical Method / SOP Reference	U.S. EPA Method SW 9056A (anions), 351.2 (nitrogen), 350.1 (ammonia), 410.4 [chemical oxygen demand (COD)], SM5210B [biological oxygen demand (BOD)], SW 9060A [total organic carbon (TOC)], SM 2540C [total dissolved solids (TDS)], SM 2320B (alkalinity), SM2340C (hardness), 110.2 (color), SW 9066 (phenols), SW 9012B (cyanide), and SW 6010B (boron)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch	No target compounds ≥ ½ RL. For common laboratory contaminants, no analytes detected > RL.	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Laboratory Analyst	Accuracy/ Bias Contamination	No target compounds ≥ ½ RL
LCS	One per preparation/analytical batch	See Table 12-1	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Analyst	Accuracy/ Bias	See Table 12-3
MS/MSD	Each group of field samples in an SDG	See Table 12-1	Assess data to determine whether there is a matrix	Laboratory Analyst	Accuracy/Bias and Precision	See Table 12-3

QAPP Worksheet #28 – QC Samples Table

Matrix	Groundwater
Analytical Group	Landfill Leachate Indicators
Analytical Method / SOP Reference	U.S. EPA Method SW 9056A (anions), 351.2 (nitrogen), 350.1 (ammonia), 410.4 [chemical oxygen demand (COD)], SM5210B [biological oxygen demand (BOD)], SW 9060A [total organic carbon (TOC)], SM 2540C [total dissolved solids (TDS)], SM 2320B (alkalinity), SM2340C (hardness), 110.2 (color), SW 9066 (phenols), SW 9012B (cyanide), and SW 6010B (boron)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
	or each SDG, whichever is most frequent		effect or analytical error. Review LCS for failed target analytes. Examine the project-specific DQOs. Contact the client as to additional measures to be taken.			
LODs	Annual	Per Laboratory SOP	Reanalyze LOD	TestAmerica Laboratory	Sensitivity	Low enough to support RLs

QAPP Worksheet #28 – QC Samples Table

Matrix	Air
Analytical Group	VOC
Analytical Method / SOP Reference	TO-15/005

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Blank Spike	Each batch or every 20 samples, whichever is sooner.	%R for all analytes within 70-130	Reanalyze Sample	TestAmerica Laboratory	Accuracy	Per Laboratory SOP
Method Blank	Each batch or every 20 samples.	< RL	Reanalyze Sample	TestAmerica Laboratory	Contamination	See worksheet 15 for lab LOQs
Internal Standard	All standards, field and QC samples	+/- 40% area response from last acceptable calibration. RT +/- 0.33 min (20 seconds) from last acceptable calibration.	Reanalyze Sample	TestAmerica Laboratory	Instrument Performance	Per Laboratory SOP
LODs	Annual	Per Laboratory SOP	Reanalyze LOD	TestAmerica Laboratory	Sensitivity	Low enough to support RLs

QAPP Worksheet #29 – Project Documents and Records Table

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Logbook	Sample Receipt, Custody, and Tracking Records	Sample Receipt, Custody, and Tracking Records	Field Sampling Checklists	
Boring Log	Equipment Calibration Logs	Equipment Calibration Logs	Field Analysis Audit Checklists	
COC Records	CA Forms	CA Forms	Data Validation Reports	
Air Bills	Reported Field Sample Results	Reported Field Sample Results	CA Forms	
Custody Seals	Sample Disposal Records	Reported Results for Standards, QC Checks, and QC Samples		
CA Forms	Health and Safety Inspection Forms	Data package Completeness Checklist		
Monitoring Well Construction Log		Sample Disposal Records		
		Extraction/Cleanup-up Records		
		Raw Data (stored on disk CD-R)		

QAPP Worksheet #30 – Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations/ Identification (ID) Number	Analytical SOPs	Data Package Turnaround Time	Primary Laboratory	QA Laboratory
Groundwater / Surface water	VOCs, SVOCs, metals, landfill leachate indicators, PCBs, and pesticides	Low to High	LTM sites at the Former Griffiss AFB	DV-MS-0010, DV-MS-0011, DV-MT-0019, DV-MT-0021, DV-IP-0010, DV-MT-0017, DV-WC-0006, DV-WC-0020, DV-WC-0040, DV-WC-0082, DV-WC-0060, DV-OP-0006, DV-OP-0007, DV-0P-0008, DV-GC-0020, DV-GC-0026, DV-GC-0021, DV-GC-0030	20 days for full data package	TA Denver	N/A
Soil	VOCs, SVOCs, metals, pesticides	Low-to-Medium	Building 301, 255, AOI 72, and Building 211	DV-MT-0019, DV-MT-0021, DV-IP-0015, DV-MT-0016, DV-MT-0023, DV-MS-0010, DV-MS-0011, DV-OP-0007, DV-OP-0010, DV-GC-0020, DV-GC-	20 days for full data package	TA Denver	N/A

QAPP Worksheet #30 – Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations/ Identification (ID) Number	Analytical SOPs	Data Package Turnaround Time	Primary Laboratory	QA Laboratory
				0026, DV-GC-0021, DV-GC-0030, SA-GE-001			
SVI Vapor	VOCs	Low-to-Medium	SD-52 SVI System Sites and ST006	005	20 days for full data package	TA Burlington	N/A

QAPP Worksheet #31 – Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Review Field Logbooks, Boring Logs, Monitoring Well Completion Logs, and COC forms	As work progresses	Internal	FPM and AECOM	Daniel Baldyga, FPM Technical Lead and Dan Servetas, AECOM Technical Lead	Daniel Baldyga, FPM Technical Lead and Dan Servetas, AECOM Technical Lead	Daniel Baldyga, FPM Technical Lead and Dan Servetas, AECOM Technical Lead	Daniel Baldyga, FPM Technical Lead and Dan Servetas, AECOM Technical Lead

QAPP Worksheet #32 – Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Review Field Logbooks, Boring Logs, and Chain of Custody forms	Marked up copy of document	Daniel Baldyga, FPM Technical Lead and Dan Servetas, AECOM Technical Lead	Within 24 hours of finding deficiency	Review of corrected documentation	Daniel Baldyga, FPM Technical Lead and Dan Servetas, AECOM Technical Lead	24 hours after notification

QAPP Worksheet #33 – QA Management Reports Table

Type of Report	Frequency[1] (daily, weekly, monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Field Activity Reports	Weekly	NA, will be included in the Annual Reports	Daniel Baldyga, FPM Technical Lead and Dan Servetas, AECOM Technical Lead	
Sampling Data and Site O&M Report for Landfill AOCs	Two Annual reports. Landfill 5 and 7 in one report following the Spring Sampling Round and one for Landfill 1, 2/3, and 6 following the Fall Sampling Round.	4th Quarter of each year (calendar)	Daniel Baldyga/ FPM Technical Lead	David Farnsworth, AFCEC Project Management, Robert Morse, USEPA Remedial Project Manager, and Heather Bishop, NYSDEC Environmental Engineer
Sampling Data and Site O&M Report for On-Base Groundwater AOCs	Annually (Following Spring Sampling Round).	3rd Quarter of each year (calendar)	Daniel Baldyga/ FPM Technical Lead	
Quarterly O&M Reports for SVE and SVI Mitigation Systems	Report will be submitted following data collection and analysis.	Quarterly	Daniel Baldyga/ FPM Technical Lead	
LUC/IC Site Closure Reports	Report will be submitted following data collection and analysis.	3rd Quarter of 2014	Daniel Baldyga/ FPM Technical Lead	

QAPP Worksheet #33 – QA Management Reports Table

Type of Report	Frequency[1] (daily, weekly, monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Sampling Data and Site O&M Report for Petroleum Spill Sites LTM and Remediation O&M	Semi-Annually (Following Spring and Fall Sampling Rounds)	Quarterly	Dan Servetas/ AECOM Technical Lead	David Farnsworth, AFCEC Project Management and Mark Tibbe, NYSDEC Petroleum Spills Program

QAPP Worksheet #34 – Verification (Step I) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
COC and shipping forms	COC forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the COC should be initialed by the reviewer, a copy of the COC retained in the project file, and the original and remaining copies taped inside the cooler for shipment. If the lab courier is used, the courier signs the COC upon receipt of sample coolers, the original is given to the lab courier and a copy is retained with the shipper.	I	Daniel Baldyga, FPM and Dan Servetas, AECOM
Daily QC Reports	Upon report completion, a copy of the report will be placed in the project file.	I	Daniel Baldyga, FPM and Dan Servetas, AECOM
Field Logbooks	Field logbooks will be reviewed internally and placed in the project file.	I	Daniel Baldyga, FPM and Dan Servetas, AECOM
Laboratory Data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal All received data packages will be verified externally according to the data validation procedures specified in Worksheet # 35	I E	TA

QAPP Worksheet #35 – Validation (Steps IIa and IIb) Process Table

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIb	Field Analytical Measurements	All field analytical parameters will be reviewed against the QAPP requirements for completeness and accuracy based on the field calibration records.	Daniel Baldyga, FPM and Dan Servetas, AECOM
IIa	SOPs	Ensure that all sampling and analytical SOPs were followed.	Connie van Hoesel, FPM
IIb	Documentation of QC Sample Results	Establish that all required QC samples were analyzed and met evaluation criteria.	Connie van Hoesel, FPM
IIb	Project Quantitation Limits	Verify that sample results met the quantitation limits specified in the QAPP.	Connie van Hoesel, FPM

QAPP Worksheet #36 – Validation (Steps IIa and IIb) Summary Table

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator
IIa	Groundwater / Surface water	VOCs, SVOCs, metals, landfill leachate indicators, PCBs, and pesticides.	Low-to-High	DOD QSM 4.2	Connie van Hoesel, FPM
IIa	Groundwater / Surface water	VOCs, SVOCs, metals, landfill leachate indicators, PCBs, and pesticides.	Low-to-High	QAPP Worksheets 12, 15 and 24. QAPP Tables 12-1 through 12-6	Connie van Hoesel, FPM
IIa	Soil / Sediment	VOCs, SVOCs, metals, PCBs, pesticides	Low-to-Medium	DOD QSM 4.2	Connie van Hoesel, FPM
IIa	Soil / Sediment	VOCs, SVOCs, metals, PCBs, pesticides	Low-to-Medium	QAPP Worksheets 12, 15 and 24. QAPP Tables 12-1 through 12-6	Connie van Hoesel, FPM
IIa	SVI Vapor	VOCs	Low-to-Medium	DOD QSM 4.2	Connie van Hoesel, FPM
IIa	SVI Vapor	VOCs	Low-to-Medium	QAPP Worksheets 12, 15 and 24. QAPP Tables 12-1, 12-2 and 12-7	Connie van Hoesel, FPM

QAPP Worksheet #37 – Usability Assessment

A complete (100%) data review will be performed on the samples collected during the sampling event. The review will consist of verification and validation based on completeness and compliance checks of sample receipt conditions and both sample-related and instrument-related QC results, as addressed in Worksheet 12. The Data Usability Assessment will be performed by FPM personnel. Connie van Hoesel, FPM Chemical QC Coordinator will be responsible for information in the Usability Assessment. Note that the Data Usability Assessment will be conducted on verified/validated data. After the Data Usability Assessment has been performed, data deemed appropriate for decision-making purposes will be used to assess contaminant extents at sites at the former Griffiss AFB. The results of the Data Usability Assessment will be presented in the Site Specific LTM Report. The following items will be assessed and conclusions drawn based on their results.

Precision: Results of field duplicates will be presented separately in tabular format for each sample pair. For each field duplicate pair, the results will be assessed as stated in Tables 12-3 through 12-7. MS/MSD RPDs are calculated by the laboratory and those with RPDs outside the criteria established in Table 12-1 will be listed in tabular form in the data verification report. A discussion will follow summarizing the results of the laboratory precision. Any conclusions about the precision of the analyses will be drawn and any limitations on the use of the data will be described.

Accuracy/Bias Contamination: Results for all laboratory method blanks will be evaluated and analytes detected in these blanks will be listed in tabular form in the data verification report. Laboratory data will be qualified based on the criteria listed in Tables 12-3 through 12-7. A discussion will follow summarizing the results of the laboratory accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Overall Accuracy/Bias: Results for all laboratory control sample (LCS), surrogate and MS/MSD recoveries that are outside evaluation criteria will be presented in tabular format in the data verification reports. The results will be checked versus those listed in Tables 12-1 and 12-2. A discussion will follow summarizing the overall accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Representativeness: A measure of representativeness will be provided by assessing if the proper analytical procedures, appropriate methods, laboratory SOPs, holding times and field duplicates are followed. Any conclusions about the representativeness of the analyses will be drawn and any limitations on the use of the data will be described.

QAPP Worksheet #37 – Usability Assessment

Comparability: In accordance with this UFP QAPP the data are comparable when collection techniques, measurement method and reporting procedures are the same for each data set.

Completeness: A completeness check will be performed on all data generated by the laboratory. Completeness criteria are presented on Worksheet #12. Completeness will be calculated as the number of data points for each analyte that is deemed useable (not rejected) divided by the total number of data points for each analyte. A discussion will follow summarizing the results of the calculation of data completeness. Any conclusions about the completeness of the data will be drawn and any limitations on the use of the data will be described. Data completeness addresses only those samples that are collected and only data that is analyzed by the laboratory.

Graphics: Figures and maps will be prepared showing the fuel oil impact levels at each sampling location.

Reconciliation: Each of the measurement performance criteria listed in Worksheet #12 will be examined to determine if the objective was met. Each analysis will be evaluated separately in terms of the major impacts observed from the data verification/validation, data quality indicators and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be determined. Usability of the data will be based on the quality assessment. After establishing the usability of the data, it will be determined if the DQO was met and if project action limits were met. The final report will include a summary of all points that comprised the reconciliation of each objective. Any conclusions or limitations on the usability of any of the data will be described.

Tables

Figures

Appendix A

Field Standard Operating Procedures

Appendix B

Laboratory Standard Operating Procedures

Appendix C

Project Schedule

Tables

TABLE 12-1
ACCURACY AND PRECISION CRITERIA FOR CHEMICAL ANALYSIS
FORMER GRIFFISS AIR FORCE BASE

Spiking Compound	Accuracy (%R)		Precision (RPD)	
	Aqueous	Soil	Aqueous	Soil
VOCs				
1,1,1,2-Tetrachloroethane	80 - 130	75 - 125	30	30
1,1,1-Trichloroethane	65 - 130	70 - 135	30	30
1,1,2,2-Tetrachloroethane	65 - 130	55 - 130	30	30
1,1,2-Trichloro-1,2,2-trifluoroethane	N/A	N/A	30	30
1,1,2-Trichloroethane	75 - 125	60 - 125	30	30
1,1-Dichloroethane	70 - 135	75 - 125	30	30
1,1-Dichloroethene	70 - 130	65 - 135	30	30
1,1-Dichloropropene	75 - 130	70 - 135	30	30
1,2,3-Trichlorobenzene	55 - 140	60 - 135	30	30
1,2,3-Trichloropropane	75 - 125	65 - 130	30	30
1,2,4-Trichlorobenzene	65 - 135	65 - 130	30	30
1,2,4-Trimethylbenzene	75 - 130	65 - 135	30	30
1,2-Dibromo-3-chloropropane	50 - 130	40 - 135	30	30
1,2-Dibromoethane (EDB)	80 - 120	70 - 125	30	30
1,2-Dichlorobenzene	70 - 120	75 - 120	30	30
1,2-Dichloroethane	70 - 130	70 - 135	30	30
1,2-Dichloropropane	75 - 125	70 - 120	30	30
1,3,5 -Trimethylbenzene	75 - 130	65 - 135	30	30
1,3-Dichlorobenzene	75 - 125	70 - 125	30	30
1,3-Dichloropropane	75 - 125	75 - 125	30	30
1,4-Dichlorobenzene	75 - 125	70 - 125	30	30
1,4-Dioxane	N/A	N/A	30	30
1-Chlorohexane	N/A	N/A	30	30
2,2-Dichloropropane	70 - 135	65 - 135	30	30
2-Butanone	30 - 150	30 - 160	30	30
2-Chlorotoluene	75 - 125	70 - 130	30	30
2-Hexanone	55 - 130	45 - 145	30	30
4-Chlorotoluene	75 - 130	75 - 125	30	30
4-Methyl-2-pentanone	60 - 135	45 - 145	30	30
Acetone	40 - 140	20 - 160	30	30
Benzene	80 - 120	75 - 125	30	30
Bromobenzene	75 - 125	65 - 120	30	30
Bromochloromethane	65 - 130	70 - 125	30	30
Bromodichloromethane	75 - 120	70 - 130	30	30
Bromoform	70 - 130	55 - 135	30	30
Bromomethane	30 - 145	30 - 160	30	30
Carbon disulfide	35 - 160	45 - 160	30	30
Carbon tetrachloride	65 - 140	65 - 135	30	30
Chlorobenzene	80 - 120	75 - 125	30	30
Chloroethane	60 - 135	40 - 155	30	30
Chloroform	65 - 135	70 - 125	30	30
Chloromethane	40 - 125	50 - 130	30	30
cis-1,2-Dichloroethene	70 - 125	65 - 125	30	30
cis-1,3-Dichloropropene	70 - 130	70 - 125	30	30
Cyclohexane	N/A	N/A	30	30
Dibromochloromethane	60 - 135	65 - 130	30	30
Dibromomethane	75 - 125	75 - 130	30	30
Dichlorodifluoromethane	30 - 155	35 - 135	30	30
Ethylbenzene	75 - 125	75 - 125	30	30

TABLE 12-1
ACCURACY AND PRECISION CRITERIA FOR CHEMICAL ANALYSIS
FORMER GRIFFISS AIR FORCE BASE

Spiking Compound	Accuracy (%R)		Precision (RPD)	
	Aqueous	Soil	Aqueous	Soil
Hexachlorobutadiene	50 - 140	55 - 140	30	30
Isopropylbenzene	75 - 125	75 - 130	30	30
Methyl acetate	N/A	N/A	30	30
Methylene Chloride	55 - 140	55 - 140	30	30
Methyl-tert-butyl Ether	65 - 125	67 - 119	30	30
Methylcyclohexane	N/A	N/A	30	30
m-p-Xylene	75 - 130	80 - 125	30	30
Naphthalene	55 - 140	40 - 125	30	30
n-Butylbenzene	70 - 135	65 - 140	30	30
n-Propylbenzene	70 - 130	65 - 135	30	30
o-Xylene	80 - 120	75 - 125	30	30
p-Isopropyltoluene	75 - 130	75 - 135	30	30
sec-Butylbenzene	70 - 125	65 - 130	30	30
Styrene	65 - 135	75 - 125	30	30
tert-Butylbenzene	70 - 130	65 - 130	30	30
Tetrachloroethene	45 - 150	65 - 140	30	30
Toluene	75 - 120	70 - 125	30	30
trans-1,2-Dichloroethene	60 - 140	65 - 135	30	30
trans-1,3-Dichloropropene	55 - 140	65 - 125	30	30
Trichloroethene	70 - 125	75 - 125	30	30
Trichlorofluoromethane	60 - 145	25 - 185	30	30
Vinyl chloride	50 - 145	60 - 125	30	30
Xylenes (total)	80 - 120	70 - 130	30	30
SVOCs/PAHs				
1,1'-Biphenyl	N/A	N/A	30	30
1,2,4,5-Tetrachlorobenzene	N/A	N/A	30	30
1,2,4-Trichlorobenzene	35 - 105	45 - 110	30	30
1,2-Dichlorobenzene	35 - 100	45 - 100	30	30
1,3-Dichlorobenzene	30 - 100	40 - 100	30	30
1,4-Dichlorobenzene	30 - 100	35 - 105	30	30
2,3,4,6-Tetrachlorophenol	N/A	N/A	30	30
2,4,5-Trichlorophenol	50 - 110	50 - 110	30	30
2,4,6-Trichlorophenol	50 - 115	45 - 110	30	30
2,4-Dichlorophenol	50 - 105	45 - 110	30	30
2,4-Dimethylphenol	30 - 110	30 - 105	30	30
2,4-Dinitrophenol	15 - 140	15 - 130	30	30
2,4-Dinitrotoluene	50 - 120	50 - 115	30	30
2,6-Dinitrotoluene	50 - 115	50 - 110	30	30
2-Chloronaphthalene	50 - 105	45 - 105	30	30
2-Chlorophenol	35 - 105	45 - 105	30	30
2-Methylnaphthalene	45 - 105	45 - 105	30	30
2-Methylphenol	40 - 110	40 - 105	30	30
2-Nitroaniline	50 - 115	45 - 120	30	30
2-Nitrophenol	40 - 115	40 - 110	30	30
3,3'-Dichlorobenzidine	20 - 110	10 - 130	30	30
3/4-Methylphenol	30 - 110	40 - 105	30	30

TABLE 12-1
ACCURACY AND PRECISION CRITERIA FOR CHEMICAL ANALYSIS
FORMER GRIFFISS AIR FORCE BASE

Spiking Compound	Accuracy (%R)		Precision (RPD)	
	Aqueous	Soil	Aqueous	Soil
3-Nitroaniline	20 - 125	25 - 110	30	30
4,6-Dinitro-2-methylphenol	40 - 130	30 - 135	30	30
4-Bromophenyl phenyl ether	50 - 115	45 - 115	30	30
4-Chloro-3-methylphenol	45 - 110	45 - 115	30	30
2/4-Chloroaniline	15 - 110	10 - 95	30	30
4-Chlorophenyl phenyl ether	50 - 110	45 - 110	30	30
4-Nitroaniline	35 - 120	35 - 115	30	30
4-Nitrophenol	10 - 125	15 - 140	30	30
Acenaphthylene	50 - 105	45 - 105	30	30
Acenapthene	45 - 110	45 - 110	30	30
Acetophenone	N/A	N/A	30	30
Anthracene	55 - 110	55 - 105	30	30
Atrazine	N/A	N/A	30	30
Benzaldehyde	N/A	N/A	30	30
Benzo(a)anthracene	55 - 110	50 - 110	30	30
Benzo(a)pyrene	55 - 110	50 - 110	30	30
Benzo(b)fluoranthene	45 - 120	45 - 115	30	30
Benzo(g,h,i)perylene	40 - 125	40 - 125	30	30
Benzo(k)fluoranthene	45 - 125	45 - 125	30	30
Benzoic acid	10 - 125	10 - 110	30	30
Benzyl alcohol	30 - 110	20 - 125	30	30
bis(2-Chloroethoxy) methane	45 - 105	45 - 110	30	30
bis(2-Chloroethyl) ether	35 - 110	40 - 105	30	30
bis(2-Chloroisopropyl) ether (2,2'-oxybi	25 - 130	20 - 115	30	30
bis(2-Ethylhexyl) phthalate	40 - 125	45 - 125	30	30
Butyl benzyl phthalate	45 - 115	50 - 125	30	30
Caprolactam	N/A	N/A	30	30
Carbazole	50 - 115	45 - 115	30	30
Chrysene	55 - 110	55 - 110	30	30
Dibenz(a,h)anthracene	40 - 125	40 - 125	30	30
Dibenzofuran	55 - 105	50 - 105	30	30
Diethyl phthalate	40 - 120	50 - 115	30	30
Dimethyl phthalate	25 - 125	50 - 110	30	30
Di-n-butyl phthalate	55 - 115	55 - 110	30	30
Di-n-octyl phthalate	35 - 135	40 - 130	30	30
Fluoranthene	55 - 115	55 - 115	30	30
Fluorene	50 - 110	50 - 110	30	30
Hexachlorobenzene	50 - 110	45 - 120	30	30
Hexachlorobutadiene	25 - 105	40 - 115	30	30
Hexachlorocyclopentadiene	10 - 82	35 - 123	30	30
Hexachloroethane	30 - 100	35 - 110	30	30
Indeno(1,2,3-cd)pyrene	45 - 125	40 - 120	30	30
Isophorone	50 - 110	45 - 110	30	30
Naphthalene	40 - 100	40 - 105	30	30
Nitrobenzene	45 - 110	40 - 115	30	30
N-Nitrosodi-n-propylamine	35 - 130	40 - 115	30	30
N-Nitrosodiphenylamine	50 - 110	50 - 115	30	30
Pentachlorophenol	40 - 115	25 - 120	30	30

TABLE 12-1
ACCURACY AND PRECISION CRITERIA FOR CHEMICAL ANALYSIS
FORMER GRIFFISS AIR FORCE BASE

Spiking Compound	Accuracy (%R)		Precision (RPD)	
	Aqueous	Soil	Aqueous	Soil
Phenanthrene	50 - 115	50 - 110	30	30
Phenol	10 - 115	40 - 100	30	30
Pyrene	50 - 130	45 - 125	30	30
Pesticides/PCBs				
Aldrin	25 - 140	45 - 140	30	30
Aroclor 1016	25 - 145	40 - 140	30	30
Aroclor 1221	70 - 130	70 - 130	30	30
Aroclor 1232	N/A	N/A	N/A	N/A
Aroclor 1242	N/A	N/A	N/A	N/A
Aroclor 1248	N/A	N/A	N/A	N/A
Aroclor 1254	70 - 130	70 - 130	30	30
Aroclor 1260	30 - 145	60 - 130	30	30
alpha-BHC	60 - 130	60 - 125	30	30
beta-BHC	65 - 125	60 - 125	30	30
delta-BHC	45 - 135	55 - 130	30	30
gamma-BHC	25 - 135	60 - 125	30	30
alpha-Chlordane	65 - 125	65 - 120	30	30
gamma-Chlordane	60 - 125	65 - 125	30	30
4,4'-DDD	25 - 150	30 - 135	30	30
4,4'-DDE	35 - 140	70 - 125	30	30
4,4'-DDT	45 - 140	45 - 140	30	30
Dieldrin	60 - 130	65 - 125	30	30
Endosulfan I	50 - 110	15 - 135	30	30
Endosulfan II	30 - 130	35 - 140	30	30
Endosulfan sulfate	55 - 135	60 - 135	30	30
Endrin	55 - 135	60 - 135	30	30
Endrin ketone	75 - 125	65 - 135	30	30
Endrin aldehyde	55 - 135	35 - 145	30	30
Heptachlor	40 - 130	50 - 140	30	30
Heptachlor epoxide	60 - 130	65 - 130	30	30
Methoxychlor	55 - 150	55 - 145	30	30
Toxaphene	41 - 126	25 - 138	N/A	N/A
Metals				
Aluminum	80-120	80-120	20	20
Antimony	80-120	80-120	20	20
Arsenic	80-120	80-120	20	20
Barium	80-120	80-120	20	20
Beryllium	80-120	80-120	20	20
Cadmium	80-120	80-120	20	20
Calcium	80-120	80-120	20	20
Chromium	80-120	80-120	20	20
Cobalt	80-120	80-120	20	20
Copper	80-120	80-120	20	20
Iron	80-120	80-120	20	20
Lead	80-120	80-120	20	20
Magnesium	80-120	80-120	20	20

TABLE 12-1
ACCURACY AND PRECISION CRITERIA FOR CHEMICAL ANALYSIS
FORMER GRIFFISS AIR FORCE BASE

Spiking Compound	Accuracy (%R)		Precision (RPD)	
	Aqueous	Soil	Aqueous	Soil
Manganese	80-120	80-120	20	20
Molybdenum	80-120	80-120	20	20
Mercury	80-120	80-120	20	20
Nickel	80-120	80-120	20	20
Potassium	80-120	80-120	20	20
Selenium	80-120	80-120	20	20
Silver	80-120	75-120	20	20
Sodium	80-120	80-120	20	20
Thallium	80-120	80-120	20	20
Vanadium	80-120	80-120	20	20
Zinc	80-120	80-120	20	20
Inorganics				
Bromide	86 - 110		10	
Chloride	89 - 110		10	
Nitrate	87 - 110		10	
Sulfate	86 - 110		10	
Cyanide	90 - 110		10	
TOC	86 - 114		12	
TKN	77 - 115		25	
BOD	85 - 115		20	
COD	90 - 110		11	
Alkalinity	90 - 110		10	
TDS	86 - 110		20	
Hardness	90 - 110		10	
Soil Gas VOCs	%R (gas)		RPD (gas)	
1,1,1-TCA	70-130		25	
1,2-DCA	70-130		25	
1,2-Dibromoethane	70-130		25	
Benzene	70-130		25	
Carbon tetrachloride	70-130		25	
Chloroform	70-130		25	
Styrene	70-130		25	
TCE	70-130		25	
m,p-Xylene	70-130		25	
o-Xylene	70-130		25	
Tetrachloroethylene	70-130		25	
Toluene	70-130		25	
Ethylbenzene	70-130		25	
cis-1,2-DCE	70-130		25	
Methylene chloride	70-130		25	
Chloromethane	70-130		25	
Chloroethane	70-130		25	
Vinyl Chloride	70-130		25	
1,1,2,2-Tetrachloroethane	70-130		25	
1,1-Dichloroethene	70-130		25	
1,1,2-Trichloroethane	70-130		25	

TABLE 12-1
ACCURACY AND PRECISION CRITERIA FOR CHEMICAL ANALYSIS
FORMER GRIFFISS AIR FORCE BASE

Spiking Compound	Accuracy (%R)		Precision (RPD)	
	Aqueous	Soil	Aqueous	Soil
1,1-DCA	70-130		25	
1,2-Dichloropropane	70-130		25	
trans-1,2-DCE	70-130		25	

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Control limits based on DOD QSM, when available; otherwise, based on the laboratory's in-house limits.

%R - Percent Recovery
N/A - Not Applicable
PAHs - Polynuclear Aromatic Hydrocarbons
PCBs - Polychlorinated Biphenyls
RPD - Relative Percent Difference
SVOCs - Semi-Volatile Organic Compounds
VOCs - Volatile Organic Compounds

TABLE 12-2
ORGANIC SURROGATE COMPOUND ACCURACY CRITERIA
FORMER GRIFFISS AIR FORCE BASE

Analysis	Spiking Compound	Accuracy (%R)	
		Aqueous	Soil
VOCs	1,2-Dichloroethane-d_4	70 - 120	80 - 120
	4-Bromofluorobenzene	75 - 120	85 - 120
	Toluene-d_8	85 - 120	85 - 115
	Dibromofluoromethane	85 - 115	80 - 120
SVOCs/PAHs	2,4,6-Tribromophenol	40 - 125	35 - 125
	2-Fluorobiphenyl	50 - 110	45 - 105
	2-Fluorophenol	20 - 110	35 - 105
	Nitrobenzene-d_5	40 - 110	35 - 100
	Phenol-d_5	10 - 115	40 - 100
	Terphenyl-d_{14}	50 - 135	30 - 125
Pesticides/PCBs	Decachlorobiphenyl (8081)	30 - 135	55 - 130
	Tetrachloro-m-xylene (8081)	25 - 140	55 - 125
	Decachlorobiphenyl (8082)	40 - 135	60 - 125
	Tetrachloro-m-xylene (8082)	25 - 120	53 - 128
Inorganics	NA	NA	NA

Control limits based on DOD QSM, when available; otherwise, based on the laboratory's in-house limits.

%R - Percent Recovery
N/A - Not Applicable
PAHs - Polynuclear Aromatic Hydrocarbons
PCBs - Polychlorinated Biphenyls
SVOCs - Semi-Volatile Organic Compounds
VOCs - Volatile Organic Compounds

TABLE 12-3
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8260B AND 8270C
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
LOD determination and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOD verification checks shall be performed.	See DOD QSM v 4.2. LOD verification checks must produce a signal at least 3 times the instrument's noise level.	Repeat detection limit determination and LOD verification check at higher level and set LOD.	LOD is 2-3x the detection limit (for a single-analyte standard) or greater than 1-4x the detection limit (for a multi-analyte standard).	Apply **R-**flag to data without a valid LOD verification
LOQ establishment and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOQ verification checks shall be performed.	See DOD QSM v 4.2. LOQ must be set within the calibration range prior to sample analysis.	N/A	N/A	N/A
Holding time	Every sample	Soil VOCs: 7 days unpreserved, 14 days preserved. Soil SVOCs: 14 days to extract, 40 days to analysis Water VOCs: 7 days unpreserved, 14 days preserved. Water SVOCs: 7 days to extract, 40 days to analysis.	Contact FPM as to additional measures to be taken.		Apply **J**-flag to detects and **UJ**-flag to nondetects to samples < 2X holding time criteria. Apply **J**-flag to detects and **R**-flag to nondetects to samples > 2X holding time criteria.
Sample temperature	Every cooler	4±2 °C	Contact FPM as to additional measures to be taken.	None	Samples arriving at temperature 6-10°C, apply **J**-flag to detects and **UJ**-flag to nondetects. Samples arriving at temperature > 10°C, apply **J**-flag to detects and **R**-flag to nondetects (SVOCs only). VOC samples received at temperature > 10°C, **R**-flag all results.
Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Problem must be corrected. No samples may be accepted without a valid tune.	Apply **R-**flag to data without a valid tune
Breakdown check (DDT Method 8270C only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check	No samples shall be run until degradation ≤ 20%.	Apply **R-**flag to data without a valid breakdown check

TABLE 12-3
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8260B AND 8270C
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Minimum five point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	1. Average response factor (RF) for SPCCs: VOCs - ≥ 0.30 for Chlorobenzene and 1,1,2,2- tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs - ≥ 0.050.	Correct problem then repeat initial calibration	Problem must be corrected. No samples may be run until ICAL has passed	Apply **R-**flag to data without a valid ICAL
		2. RSD for RFs for CCCs: VOCs and SVOCs - ≤ 30% and one option below; Option 1: RSD for each analyte ≤ 15% Option 2: linear least squares regression r ≥ 0.995 Option 3: non-linear regression - coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points shall be used for second order, 7 points shall be used for third order)			Apply **R-**flag to data without a valid ICAL
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 20% of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Problem must be corrected. No samples may be run until calibration has been verified.	Apply **R-**flag to data without second source verification.

TABLE 12-3
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8260B AND 8270C
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Evaluation of relative retention times	Each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct problem, then rerun ICAL	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance.	Apply **R**-flag to data outside retention time window
Manual Integration	All	Acceptance by FPM Chemist	Provide justification for each instance of manual integration	Laboratory will provide chromatograms before and after each manual integration	Apply **R**-flag to all compounds with improper integration
Calibration verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time.	Average RF for SPCCs: VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050.			Apply **J**-flag to detects and **UJ**-flag to nondetects if average RF not met
		% Difference/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20%D (Note: D ≤ difference when using RFs or drift when using least squares regression or non-linear calibration.)	Correct problem, then rerun CV. If that fails, repeat initial calibration. Reanalyze all samples since last acceptable CCV.	Problem must be corrected. No results may be reported without a valid CCV. Flagging criteria is only appropriate in cases where the samples cannot be reanalyzed.	High bias: Apply **J**-flag to detects Low bias: Apply **J**-flag to detects and **R**-flag to nondetects
Internal standards verification	In all field samples and standards	Retention time ± 30 seconds from retention time of the midpoint standard in the CV	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Sample results are not acceptable without a valid IS verification.	If corrective action fails in field samples, apply **J**-flag to detects and **UJ**-flag to nondetects to analytes with IS recoveries between 30%-50% or > 150%. Apply **R**-flag to samples with IS recoveries < 30%.
		EICP area within - 50% to + 100% of ICAL midpoint standard			

TABLE 12-3
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8260B AND 8270C
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Method blank	One per preparatory batch	No analytes detected > 1/2 RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **B**-flag to analytes detected in field samples < 5X blank contamination (<10X for common laboratory contaminants).
Laboratory control sample (LCS)	One per preparatory batch	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<30%): Apply **J**-flag to detects and **R**-flag to nondetects.
Matrix spike/Matrix spike duplicate (MS/MSD)	One per preparatory batch per matrix	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Examine the project-specific DQOs. Contact URS as to additional measures to be taken.	For matrix evaluation only. If MS results are outside QC limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met. MS/MSD data should not be used alone to qualify data.
Laboratory sample duplicate	One per preparatory batch per matrix (if MS/MSD is not performed)	RPD $\leq$ 30% (sample and sample duplicate)	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met.	Data shall be evaluated to determine the source of difference. Apply **J**-flag to detects if acceptance criteria are not met.

TABLE 12-3
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8260B AND 8270C
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Surrogate spike	All field and QC samples	QC acceptance criteria specified in UFP-QAPP Table 12-2.	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available.	Analytes identified in UFP-QAPP Table 12-2.	High bias: Apply **J**-flag to detects Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<10%): Apply **J**-flag to detects and **R**-flag to nondetects.
Results reported between DL and LOQ	N/A	N/A	N/A	N/A	Appy **J**-flag to all results between DL and LOQ.
Field Duplicate	One per 10 field samples	See UFP-QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2).	N/A	N/A	Appy **J**-flag to detects and **UJ**-flag to nondetects.

TABLE 12-4
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8081A, 8082
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
LOD determination and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOD verification checks shall be performed.	See DOD QSM v 4.2. LOD verification checks must produce a signal at least 3 times the instrument's noise level.	Repeat detection limit determination and LOD verification check at higher level and set LOD.	LOD is 2-3x the detection limit (for a single-analyte standard) or greater than 1-4x the detection limit (for a multi-analyte standard).	Apply **R-**flag to data without a valid LOD verification
LOQ establishment and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOQ verification checks shall be performed.	See DOD QSM v 4.2. LOQ must be set within the calibration range prior to sample analysis.	N/A	N/A	N/A
Holding time	Every sample	Soil samples: 14 days to extract, 40 days to analysis Aqueous samples: 7 days to extract, 40 days to analysis	Contact FPM as to additional measures to be taken.	None None	Apply **J**-flag to detects and **UJ**-flag to nondetects to samples < 2X holding time criteria. Apply **J**-flag to detects and **R**-flag to nondetects to samples > 2X holding time criteria.
Sample temperature	Every cooler	4±2 °C	Contact FPM as to additional measures to be taken.	None	Samples arriving at temperature 6-10°C, apply **J**-flag to detects and **UJ**-flag to nondetects. Samples arriving at temperature > 10°C, apply **J**-flag to detects and **R**-flag to nondetects.
Breakdown check (Endrin/DDT Method 8081A only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤ 15% for both endrin and DDT	Correct problem then repeat breakdown check	No samples shall be analyzed until degradation ≤ 15%	Apply **R-**flag to data without valid breakdown check
Minimum five point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	One of the options below: Option 1: RSD for each analyte ≤ 20% Option 2: linear least squares regression r ≥ 0.995 Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration	None	Apply **R-**flag to data without a valid ICAL

TABLE 12-4
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8081A, 8082
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 20% of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Problem must be corrected. No samples may be run until calibration has been verified.	Apply **R**-flags to data without second source verification.
Retention time window verification	Each calibration verification standard	Analyte within established window	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window	No samples shall be run without a verified retention time window at the initial calibration	Apply **R**-flag to data outside retention time window
Manual Integration	All	Acceptance by FPM Chemist	Provide justification for each instance of manual integration	Laboratory will provide chromatograms before and after each manual integration	Apply **R**-flag to all compounds with improper integration
Calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All analytes within $\pm$ 20% of expected value form ICAL	Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **R**-flag to nondetects.
Method blank	One per preparatory batch	No analytes detected > 1/2 RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **B**-flag to analytes detected in field samples < 5X blank contamination.

TABLE 12-4
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8081A, 8082
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Laboratory control sample (LCS)	One per preparatory batch	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<30%): Apply **J**-flag to detects and **R**-flag to nondetects.
Matrix spike/Matrix spike duplicate (MS/MSD)	One per preparatory batch per matrix	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	For matrix evaluation only. If MS results are outside QC limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met. MS/MSD data should not be used alone to qualify data.
Laboratory sample duplicate	One per preparatory batch per matrix (if MS/MSD is not performed)	RPD $\leq$ 30% (sample and sample duplicate)	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met.	Data shall be evaluated to determine the source of difference. Apply **J**-flag to detects if acceptance criteria are not met.
Surrogate spike	All field and QC samples	QC acceptance criteria specified in UFP-QAPP Table 12-2.	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available.	Analytes identified in UFP-QAPP Table 12-2	High bias: Apply **J**-flag to detects Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<10%): Apply **J**-flag to detects and **R**-flag to nondetects.
Confirmation of positive results (second column or detector)	All positive results must be confirmed	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD $\leq$ 40%	N/A	Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and document in the case narrative.	Apply **J**-flag if RPD >40%. Apply **U**-flag if primary result not confirmed.

TABLE 12-4
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 8081A, 8082
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Results reported between DL and LOQ	N/A	N/A	N/A	N/A	Appy **J**-flag to all results between DL and LOQ.
Field Duplicate	One per 10 field samples	See UFP-QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2)	N/A	N/A	Appy **J**-flag to detects and **UJ**-flag to nondetects.

TABLE 12-5
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 6010B, 7470A/7471A, 7196A
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
LOD determination and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOD verification checks shall be performed.	See DOD QSM v 4.2. LOD verification checks must produce a signal at least 3 times the instrument's noise level.	Repeat detection limit determination and LOD verification check at higher level and set LOD.	LOD is 2-3x the detection limit (for a single-analyte standard) or greater than 1-4x the detection limit (for a multi-analyte standard).	Apply **R-**flag to data without a valid LOD verification
LOQ establishment and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOQ verification checks shall be performed.	See DOD QSM v 4.2. LOQ must be set within the calibration range prior to sample analysis.	N/A	N/A	N/A
IDL study (ICP only)	At initial set-up and after significant change	Detection limits established shall be $\leq$ LOD	N/A	Samples cannot be analyzed without a IDL.	Apply **R-**flag to data without a valid IDL study
Linear dynamic range or high-level check standard (ICP only)	Every 6 months	Within ±10% of true value	N/A	N/A	N/A
Holding time	Every sample	Soil samples: 6 months (Hg 28 days) Aqueous samples (preserved with HNO_3, pH<2): 6 months (Hg 28 days)	Contact FPM as to additional measures to be taken.		Apply **J**-flag to detects and **UJ**-flag to nondetects to samples < 2X holding time criteria. Apply **J**-flag to detects and **R**-flag to nondetects to samples > 2X holding time criteria.
Sample temperature	Every cooler	4±2 °C	Contact FPM for additional measures to be taken.		Samples arriving at temperature 6 - 10 °C, apply **J**-flag to detects and **UJ**-flag to nondetects. Samples arriving at temperature > 10°C, apply **J**-flag to detects and **R**-flag to nondetects.
Initial calibration for all analytes (ICAL) ICP: minimum one high standard and a blank CVAA: minimum 5 standards and a calibration blank	Daily initial calibration prior to sample analysis	$r \geq 0.995$	Correct problem then repeat initial calibration	Problem must be corrected. No samples may be run until ICAL has passed.	Apply **R-**flag to data without a valid ICAL

TABLE 12-5
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 6010B, 7470A/7471A, 7196A
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Second source calibration verification (ICV)	Once after each initial calibration, prior to sample analysis	Value of second source for all analytes within ± 10% of expected value (initial source)	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat initial calibration.	Problem must be corrected. No samples may be run until calibration has been verified.	Apply **R-**flag to data without second source verification
Calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence.	ICP: All analytes within ± 10% of expected value from ICAL. CVAA: Mercury within ± 20% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **R-**flag to data with CCV outside criteria.
Method blank	One per preparatory batch	No analytes detected > 1/2 RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **B-**flag to analytes detected in field samples < 5X blank contamination (<10X for common laboratory contaminants).
Calibration blank	Before beginning a sample run, after every 10 samples, and at the end of the analysis sequence	No analytes detected > LOD	Correct problem. If required, reprep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **B-**flag to analytes detected in field samples < 5X blank contamination.
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run	ICS-A: Absolute value of concentration for all nonspiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes). ICS-AB: Within ± 20% of expected value.	Terminate analysis; locate and correct problem; reanalyze ICS.	No samples may be analyzed without a valid ICS	Apply **R-**flag to data with ICS outside criteria.

TABLE 12-5
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHODS 6010B, 7470A/7471A, 7196A
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Laboratory control sample (LCS)	One per preparatory batch	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (ICP Metals %R<60%, Hg %R < 50%): Apply **J**-flag to detects and **R**-flag to nondetects.
Dilution test (ICP only)	Each preparatory batch or when a new or unusual matrix is encountered	Five fold dilution must agree within ± 10% of the original determination.	ICP: Perform post-digestion spike (PDS) addition.	Only applicable for samples with concentrations > 50X LOQ (6010B)	No flagging is required.
Post-digestion spike (ICP only)	When dilution test fails or analyte concentration in all samples < 50X LOD	75-125%	See flagging criteria.	The spike addition should produce a level between 10-100X LOQ	Apply **J**-flag to analytes in parent sample outside criteria
Matrix spike/Matrix spike duplicate (MS/MSD)	One per preparatory batch per matrix	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	For matrix evaluation only. If MS results are outside QC limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. No data flagging if native concentrations are > 4X spiking amount	For the specific analyte(s) in the batch. High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<30%): Apply **J**-flag to detects and **R**-flag to nondetects.
Laboratory sample duplicate	One per preparatory batch per matrix (if MS/MSD is not performed)	RPD ≤ 20% (sample and sample duplicate)	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	Data shall be evaluated to determine the source of difference.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met.
Results reported between DL and LOQ	N/A	N/A	N/A	N/A	Appy **J**-flag to all results between DL and LOQ.
Field Duplicate	One per 10 field samples	See UFP-QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2).	N/A	N/A	Apply **J**-flag to detects and **UJ**-flag to nondetects.

TABLE 12-6
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD 9056 AND LANDFILL LEACHATE INDICATOR METHODS
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
LOD determination and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOD verification checks shall be performed.	See DOD QSM v 4.2. LOD verification checks must produce a signal at least 3 times the instrument's noise level.	Repeat detection limit determination and LOD verification check at higher level and set LOD.	LOD is 2-3x the detection limit (for a single-analyte standard) or greater than 1-4x the detection limit (for a multi-analyte standard).	Apply **R-**flag to data without a valid LOD verification
LOQ establishment and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOQ verification checks shall be performed.	See DOD QSM v 4.2. LOQ must be set within the calibration range prior to sample analysis.	N/A	N/A	N/A
Holding time	Every sample	48 hours (nitrate/nitrite)/28 days (chloride, bromide, sulfate); see Worksheet #19	Contact FPM as to additional measures to be taken.		Apply **J**-flag to detects and **UJ**-flag to nondetects to samples < 2X holding time criteria. Apply **J**-flag to detects and **R**-flag to nondetects to samples > 2X holding time criteria.
Initial calibration for all analytes (ICAL) Minimum three standards and one calibration blank	Daily initial calibration prior to sample analysis	$r \geq 0.995$	Correct problem then repeat initial calibration	Problem must be corrected. No samples may be run until ICAL has passed.	Apply **R-**flag to data without a valid ICAL
Second source calibration verification (ICV)	Once after each initial calibration, prior to sample analysis	Value of second source for all analytes within ± 10% of expected value (initial source) and retention times within appropriate windows	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat initial calibration.	Problem must be corrected. No samples may be run until calibration has been verified.	Apply **R-**flag to data without second source verification

TABLE 12-6
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD 9056 AND LANDFILL LEACHATE INDICATOR METHODS
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Midrange Continuing Calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence.	All analytes within ± 10% of expected value from ICAL. All project analytes within established retention time windows.	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **R**-flag to data with CCV outside criteria.
Method blank	One per preparatory batch	No analytes detected > 1/2 RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **B**-flag to analytes detected in field samples < 5X blank contamination.
Laboratory control sample (LCS)	One per preparatory batch	Laboratory in-house limits not to exceed ±20%. Control limits may be not greater than ±3 x the standard deviation of the mean LCS recovery.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<30%): Apply **J**-flag to detects and **R**-flag to nondetects.
Matrix spike/Matrix spike duplicate (MS/MSD)	One per preparatory batch per matrix	QC acceptance criteria specified in UFP-QAPP Table 12-1 (not to exceed ±20%).	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	For matrix evaluation only. If MS results are outside QC limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. No data flagging if native concentrations are > 4X spiking	For the specific analyte(s) in the batch. High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<30%): Apply **J**-flag to detects and **R**-flag to nondetects.
Laboratory sample duplicate	One per preparatory batch per matrix (if MS/MSD is not performed)	RPD ≤ 15% (sample and sample duplicate)	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	Data shall be evaluated to determine the source of difference.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met.

TABLE 12-6
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD 9056 AND LANDFILL LEACHATE INDICATOR METHODS
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Results reported between DL and LOQ	N/A	N/A	N/A	N/A	Appy **J**-flag to all results between DL and LOQ.
Sample Duplicate	One per 10 field samples	RPD $\leq$ 10% (sample and sample duplicate)	Correct problem and reanalyze sample and duplicate.	N/A	Apply **J**-flag to detects and **UJ**-flag to nondetects if sample cannot be rerun or reanalysis does not correct problem.

TABLE 12-7
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD TO-15
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
LOD determination and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOD verification checks shall be performed.	See DOD QSM v 4.2. LOD verification checks must produce a signal at least 3 times the instrument's noise level.	Repeat detection limit determination and LOD verification check at higher level and set LOD.	LOD is 2-3x the detection limit (for a single-analyte standard) or greater than 1-4x the detection limit (for a multi-analyte standard).	Apply **R-**flag to data without a valid LOD verification
LOQ establishment and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOQ verification checks shall be performed.	See DOD QSM v 4.2. LOQ must be set within the calibration range prior to sample analysis.	N/A	N/A	N/A
Holding time	Every sample	30 days	Contact FPM as to additional measures to be taken.	None None	Apply **J**-flag to detects and **UJ**-flag to nondetects to samples < 2X holding time criteria. Apply **J**-flag to detects and **R**-flag to nondetects to samples > 2X holding time criteria.
MS tuning check (Use BFB)	Prior to initial calibration and calibration verification	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Problem must be corrected. No samples may be accepted without a valid tune.	Apply **R-**flag to data without a valid tune
Minimum five point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	RSD for each analyte $\leq$ 30% with at most 2 exceptions up to 40%	Correct problem then repeat initial calibration	None	Apply **R-**flag to data without a valid ICAL
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 30% of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Problem must be corrected. No samples may be run until calibration has been verified.	Apply **R-**flags to data without second source verification.

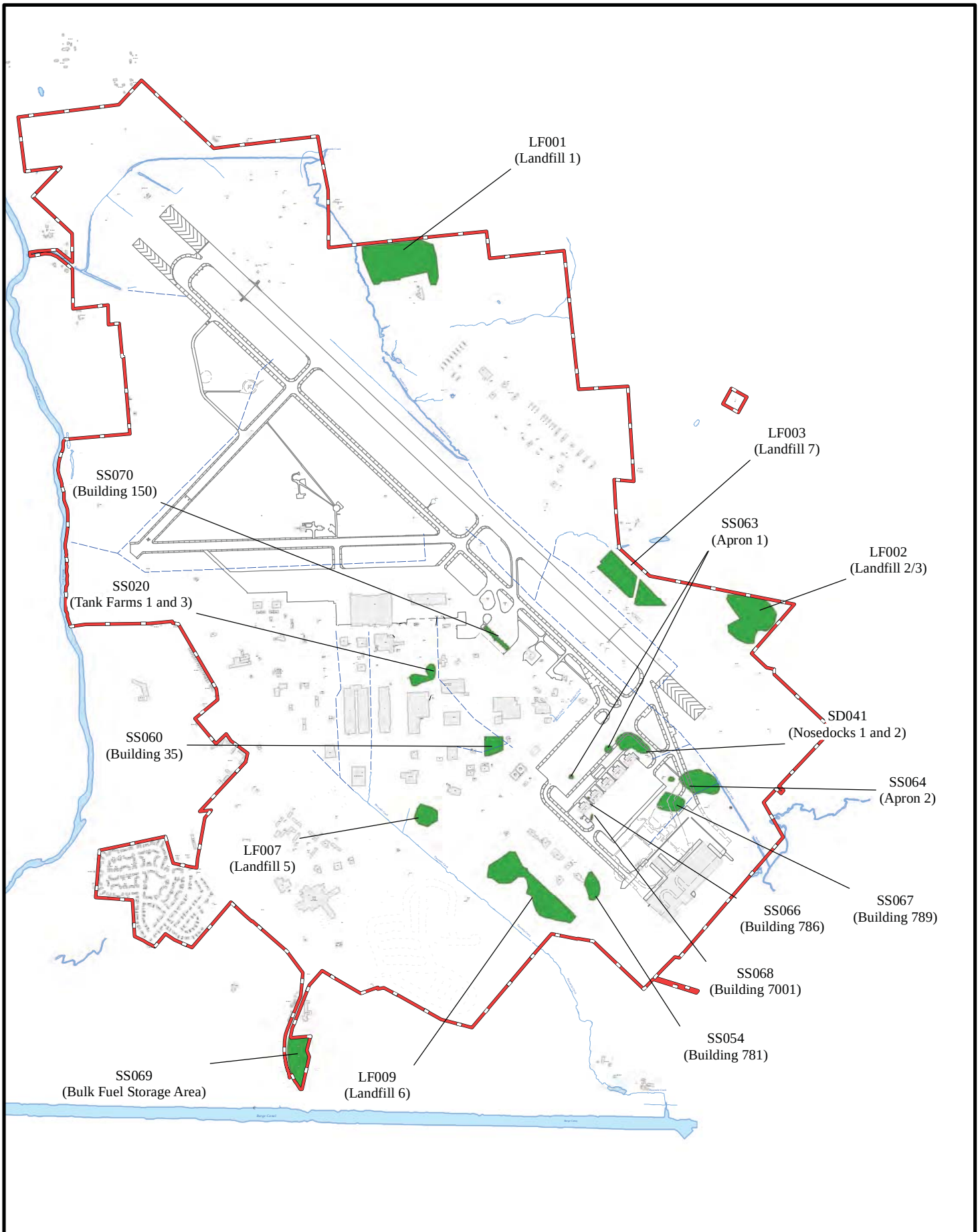
TABLE 12-7
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD TO-15
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Manual Integration	All	Acceptance by FPM Chemist	Provide justification for each instance of manual integration	Laboratory will provide chromatograms before and after each manual integration	Apply **R**-flag to all compounds with improper integration
Calibration verification (CCV)	Prior to sample analysis (unless ICAL performed on same day), and every 24 hours of analysis time	All analytes within ± 30% of expected value form ICAL	Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **R**-flag to nondetects.
Method blank (humid zero air)	Immediately after ICAL or daily CCV	No analytes detected > 1/2 RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Apply **B**-flag to analytes detected in field samples < 5X blank contamination.
Laboratory control sample (LCS)	One per preparatory batch	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	LCS not required per method. The laboratory performs an LCS as an evaluation of percent recovery in a blank matrix in approximately 1 of every 20 samples.	High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to nondetects. Very low bias (%R<30%): Apply **J**-flag to detects and **R**-flag to nondetects.
Matrix spike/Matrix spike duplicate (MS/MSD)	One per preparatory batch per matrix	QC acceptance criteria specified in UFP-QAPP Table 12-1.	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	For matrix evaluation only. If MS results are outside QC limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met. MS/MSD data should not be used alone to qualify data.

TABLE 12-7
DATA REVIEW/VALIDATION CRITERIA FOR USEPA METHOD TO-15
FORMER GRIFFISS AIR FORCE BASE

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Laboratory sample duplicate	One per preparatory batch per matrix (if MS/MSD is not performed)	RPD $\leq$ 25% (sample and sample duplicate)	Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met.	Data shall be evaluated to determine the source of difference. Apply **J**-flag to detects if acceptance criteria are not met.
Internal standards (ISs)	Each sample	Retention time ±0.33 minutes from retention time of the IS in the most recent valid calibration. (ICAL mid-point standard or CCV) EICP area within ±40% of area of the IS in most recent valid calibration	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Sample results are not acceptable without a valid IS verification.	If corrective action fails in field samples, apply J-flag to detects and UJ-flag to nondetects to analytes with IS recoveries between 30%-60% or > 140%. Apply R-flag to samples with IS recoveries < 30%.
Results reported between DL and LOQ	N/A	N/A	N/A	N/A	Appy **J**-flag to all results between DL and LOQ.
Field Duplicate	One per 10 field samples	See UFP-QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2)	N/A	N/A	Appy **J**-flag to detects and **UJ**-flag to nondetects.

Figures


LF001
(Landfill 1)
LF003
(Landfill 7)
SS070
(Building 150)
SS063
(Apron 1)
LF002
(Landfill 2/3)
SS020
(Tank Farms 1 and 3)
SD041
(Nosedocks 1 and 2)
SS060
(Building 35)
SS064
(Apron 2)
LF007
(Landfill 5)
SS067
(Building 789)
SS066
(Building 786)
SS068
(Building 7001)
SS054
(Building 781)
SS069
(Bulk Fuel Storage Area)
LF009
(Landfill 6)
Base Location
N
Map is projected in accordance with the State Plane New York Central - 3102 (NAD 83, Feet)
Key to Features
Culvert / Ditch
Surface Water
Base Boundary
Existing Facilities
Griffiss Sites
0 500 1,000 2,000 3,000
Feet
UNITED STATES AIR FORCE
FORMER GRIFFISS AIR FORCE BASE
ROME, NEW YORK
Figure 17-1
Site Location Map
March 2013
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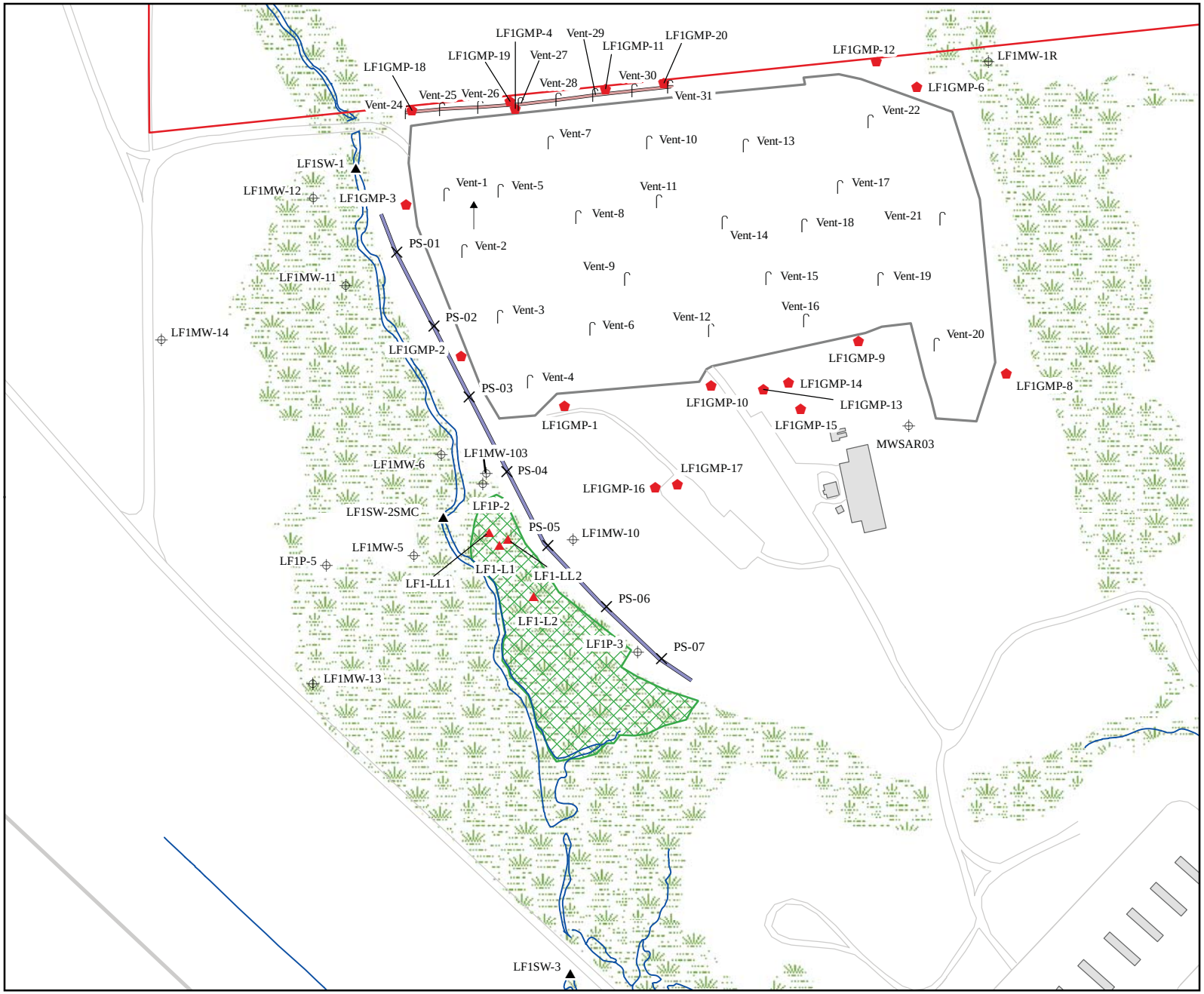

N

Site Location

Legend

- Pump Station and Sampling Location
- Leachate Sampling Location
- Groundwater Monitoring Well with ID
- Surface Water Sampling Location with ID
- Gas Monitoring Probe with ID
- Gas Vent with ID
- Leachate Collection Trench
- Passive Gas Collection Trench
- AFB Boundary
- Surface Water
- Road
- Airfield
- Landfill Boundary
- Building
- Rich Sloping Fen
- Wetland Area

250 0 250

Feet

United States Air Force
Former Griffiss Air Force Base
Rome, New York

Figure 17-2
LF001
(Landfill 1)
Sampling Locations

FPM Remediation, Inc. CAPE

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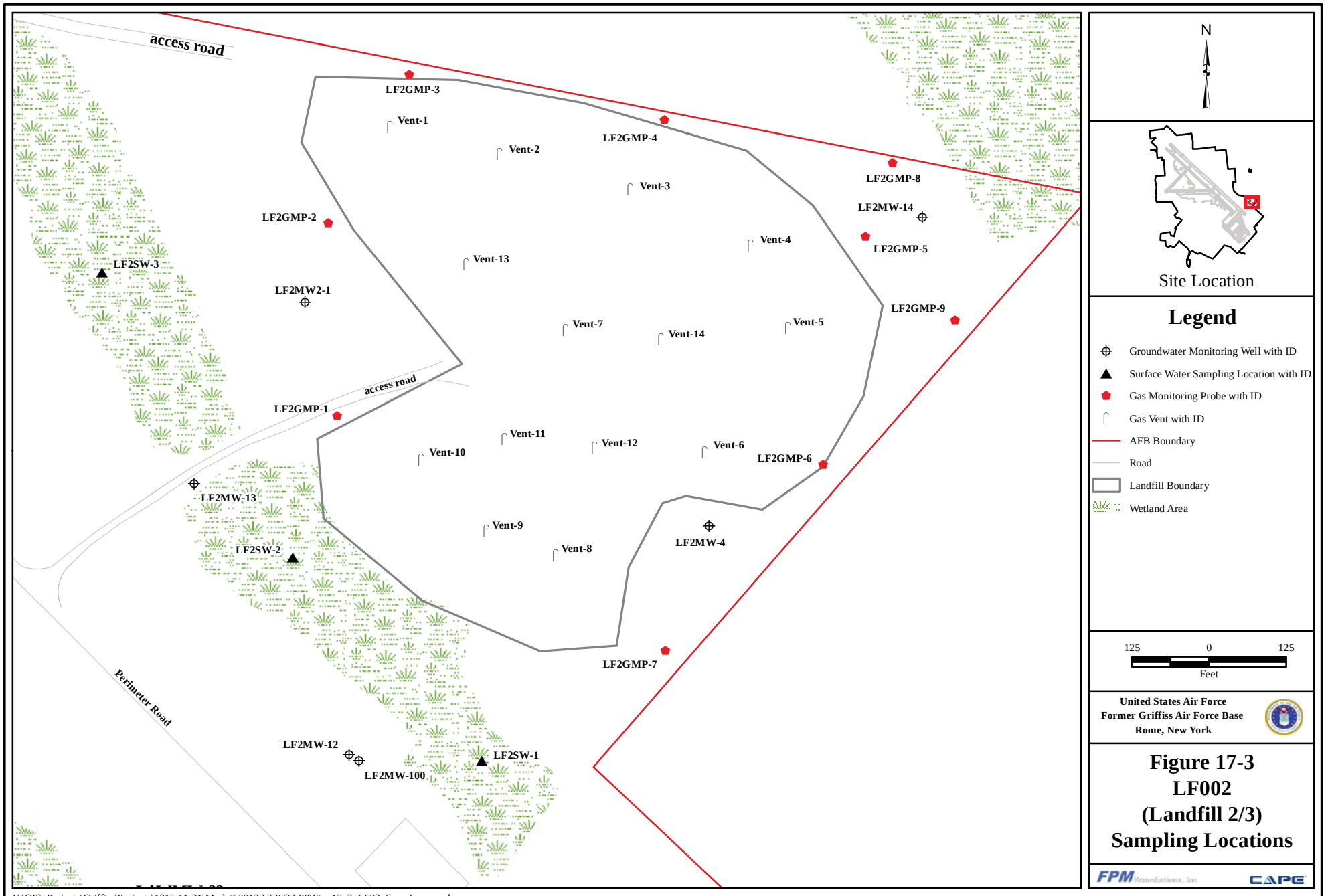

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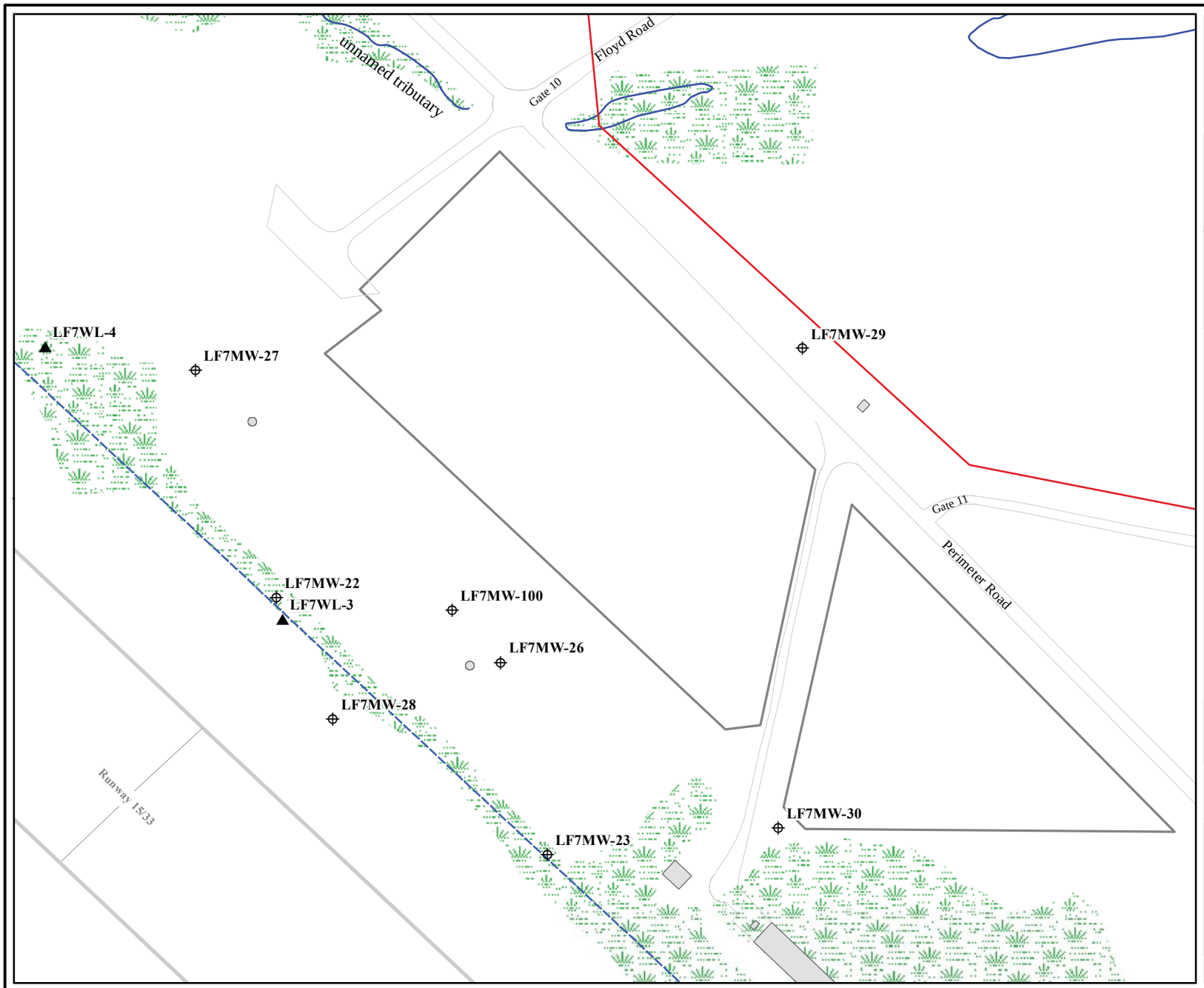

N

Site Location

Legend

- Groundwater Monitoring Well with ID
- Surface Water Sampling Location with ID
- AFB Boundary
- Surface Water
- Storm Drain
- Road
- Airfield
- Landfill Boundary
- Building
- Wetland

150 0 150

Feet

United States Air Force
Former Griffiss Air Force Base
Rome, New York

Figure 17-4
LF003
(Landfill 7)
Sampling Locations

FPM CAPE

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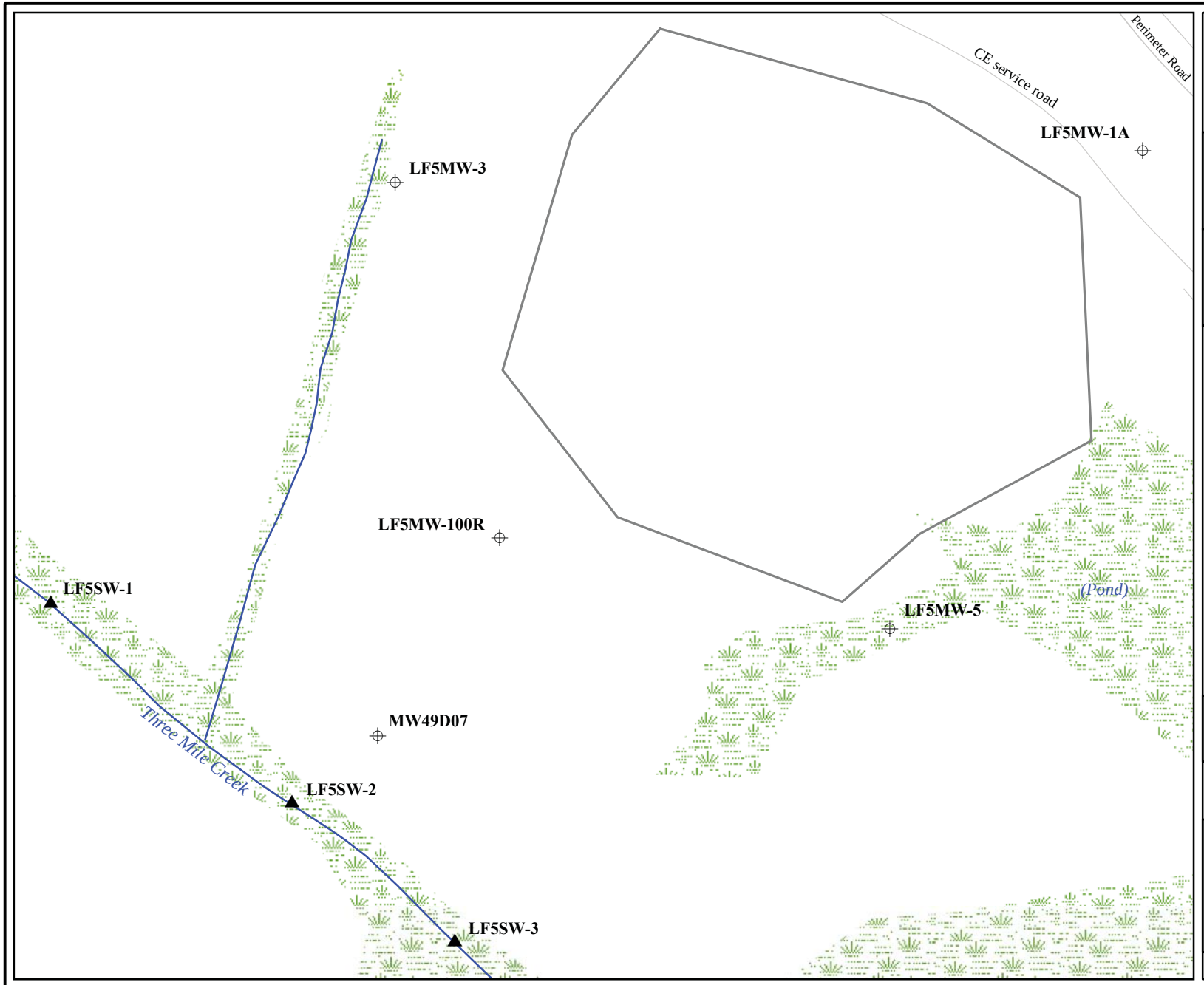

N

Legend

- Groundwater Monitoring Well with ID
- Surface Water Sampling Location with ID
- Road
- Surface Water
- Landfill Boundary
- Wetlands

75 0 75
Feet

United States Air Force
Former Griffiss Air Force Base
Rome, New York

Figure 17-5
LF007
(Landfill 5)
Sampling locations

FPM CAPE

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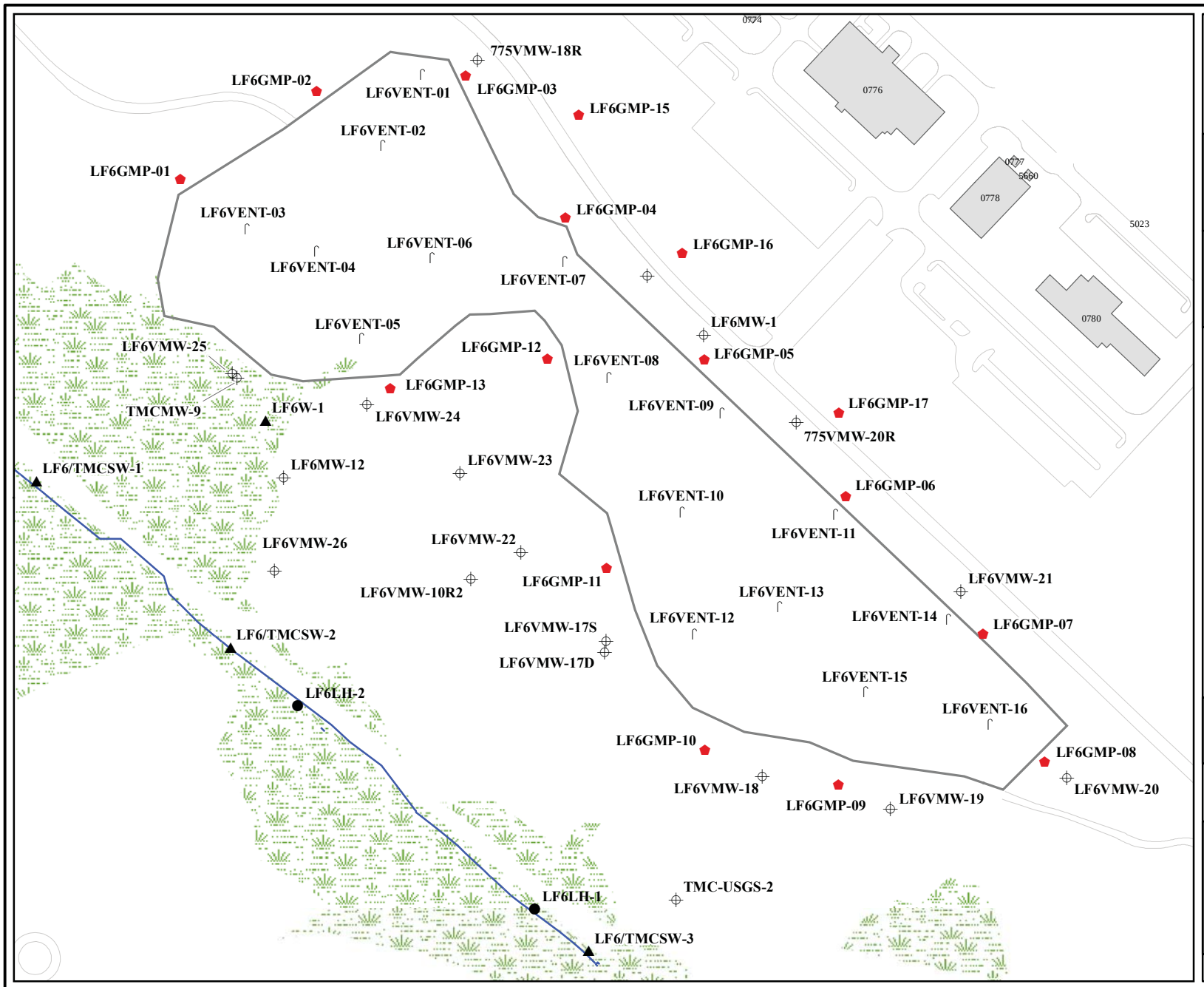

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Legend

- Groundwater Monitoring Well with ID
- Surface Water Sampling Location with ID
- Leachate Sampling Location with ID
- Gas Monitoring Probe with ID
- Gas Vent with ID
- Surface Water
- Road
- Landfill Boundary
- Building
- Wetland Area

200 0 200
Feet

United States Air Force
Former Griffiss Air Force Base
Rome, New York

Figure 17-6
LF009
(Landfill 6)
Sampling Locations

FPM CAPE

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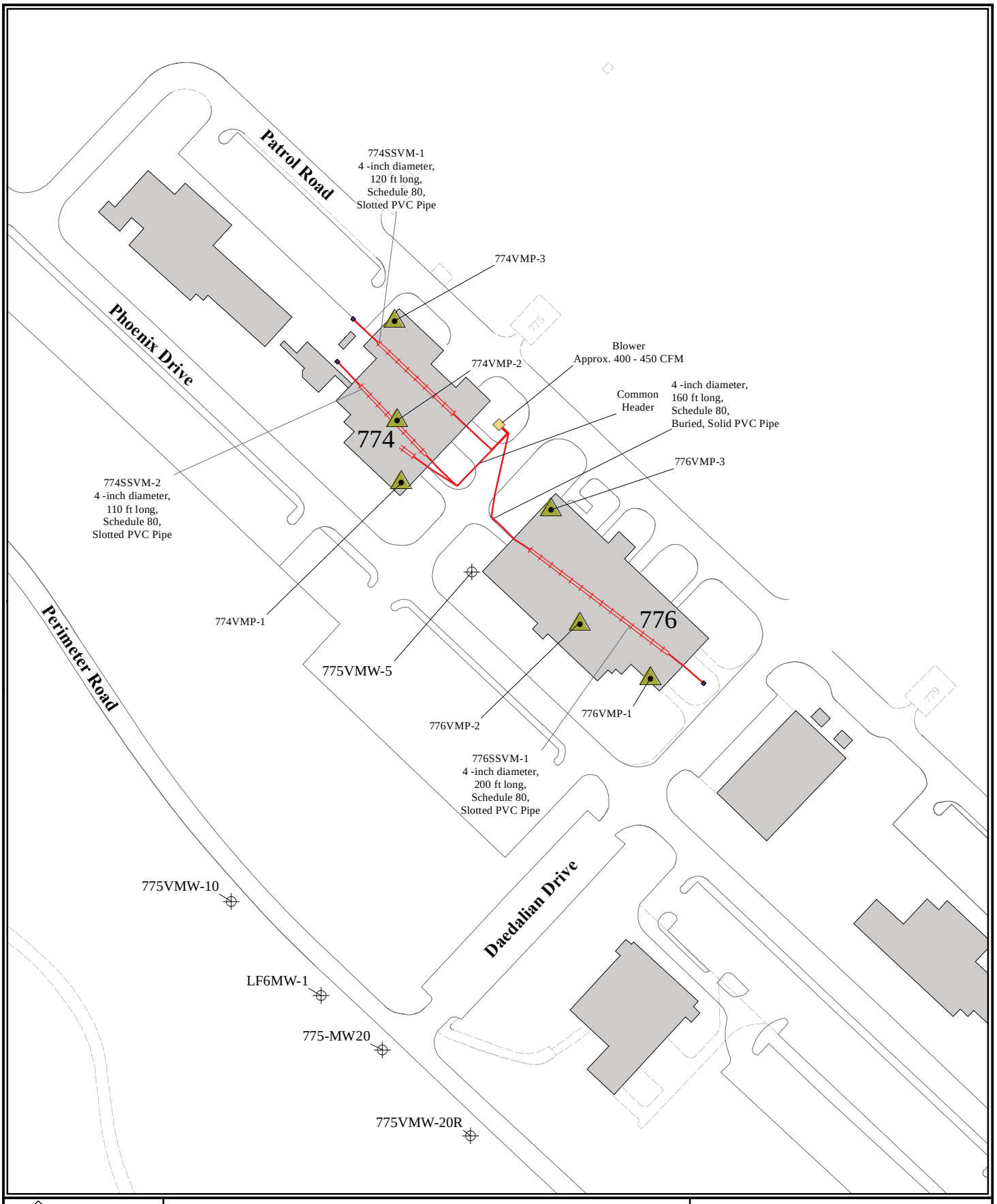

Site Location

Key to Features

- 2011 Sample Location
- Monitoring Well
- Demolished Road
- Existing Road
- Horizontal Well
- Screen
- Riser
- SVE System
- Entrance/Exit Pit
- Demolished Facility
- Existing Facility

N

100 50 0 100 Feet

UNITED STATES AIR FORCE
GRIFFISS AIR FORCE BASE
ROME, NEW YORK

Figure 17-7
SD052
(SVI Systems)
Building 774 and 776
Sampling Locations

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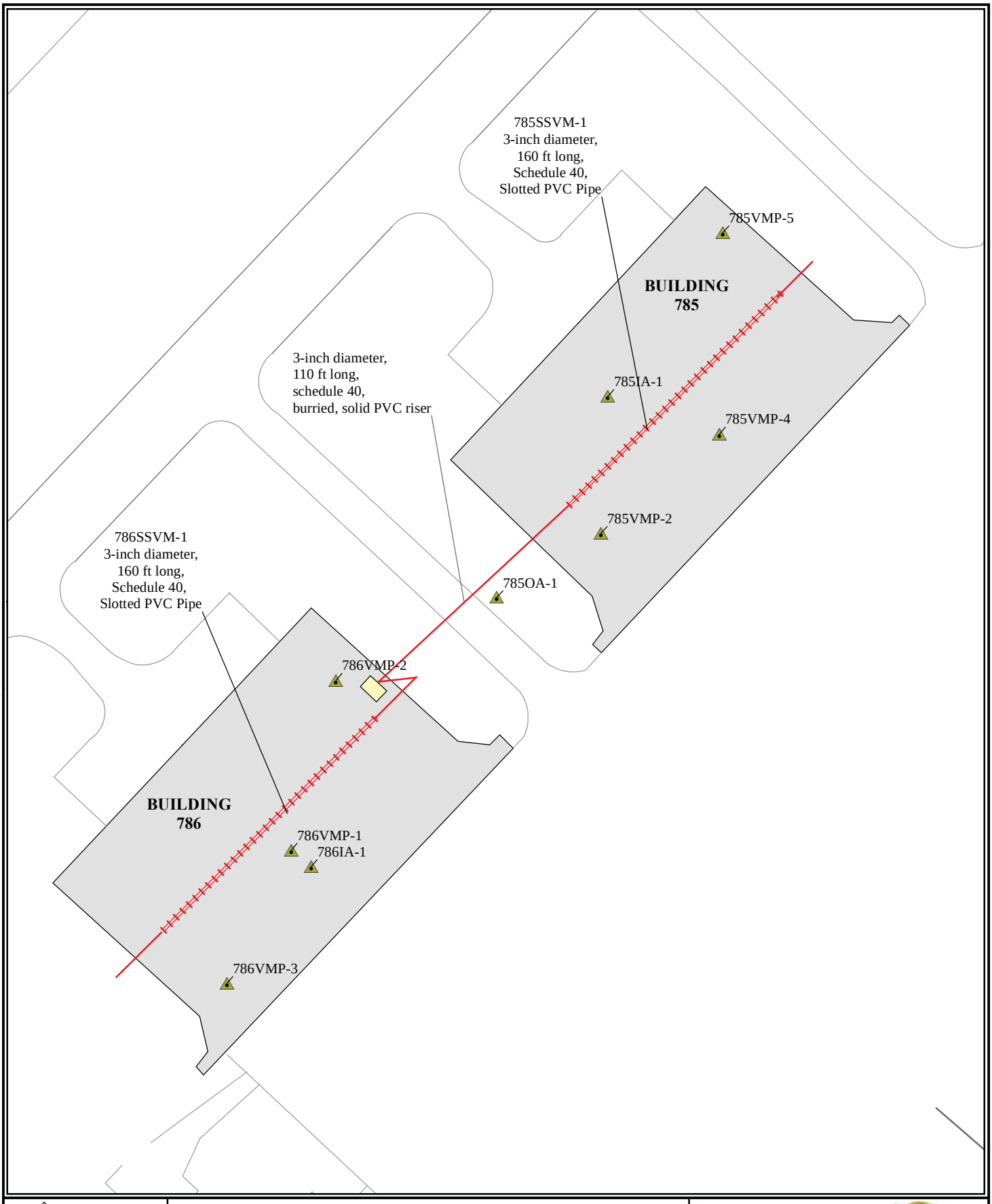

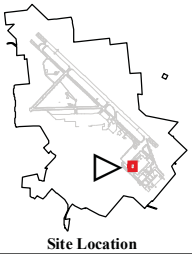

Legend

- Sample Location
- Road
- Horizontal Well
- Screen
- Riser
- SVE System
- Existing Facility

N

50 25 0 50

Feet

UNITED STATES AIR FORCE
GRIFFISS AIR FORCE BASE
ROME, NEW YORK

Figure 17-8
SD052
(SVI Systems)
Building 785 and 786
Sampling locations

FPM CAPE

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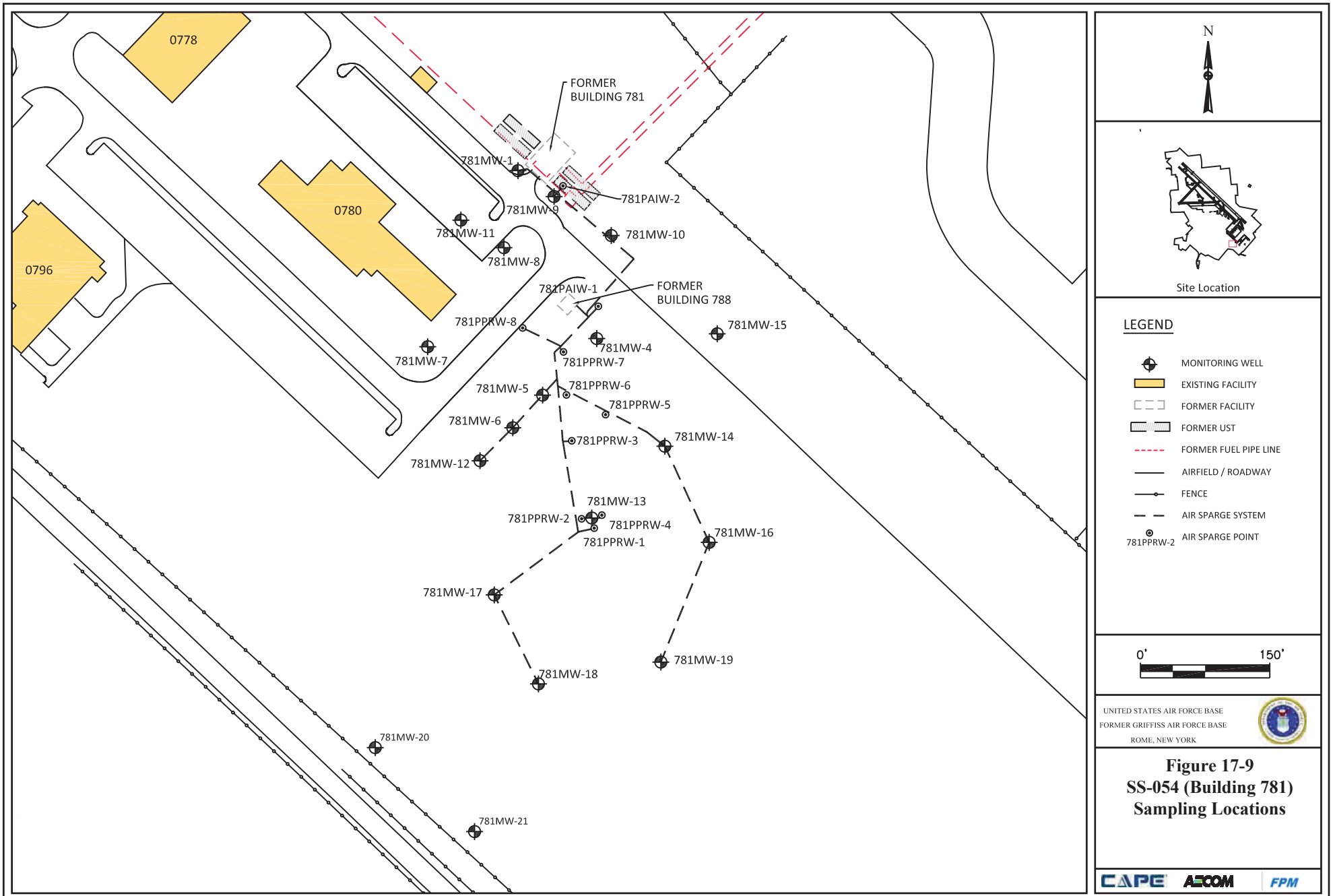

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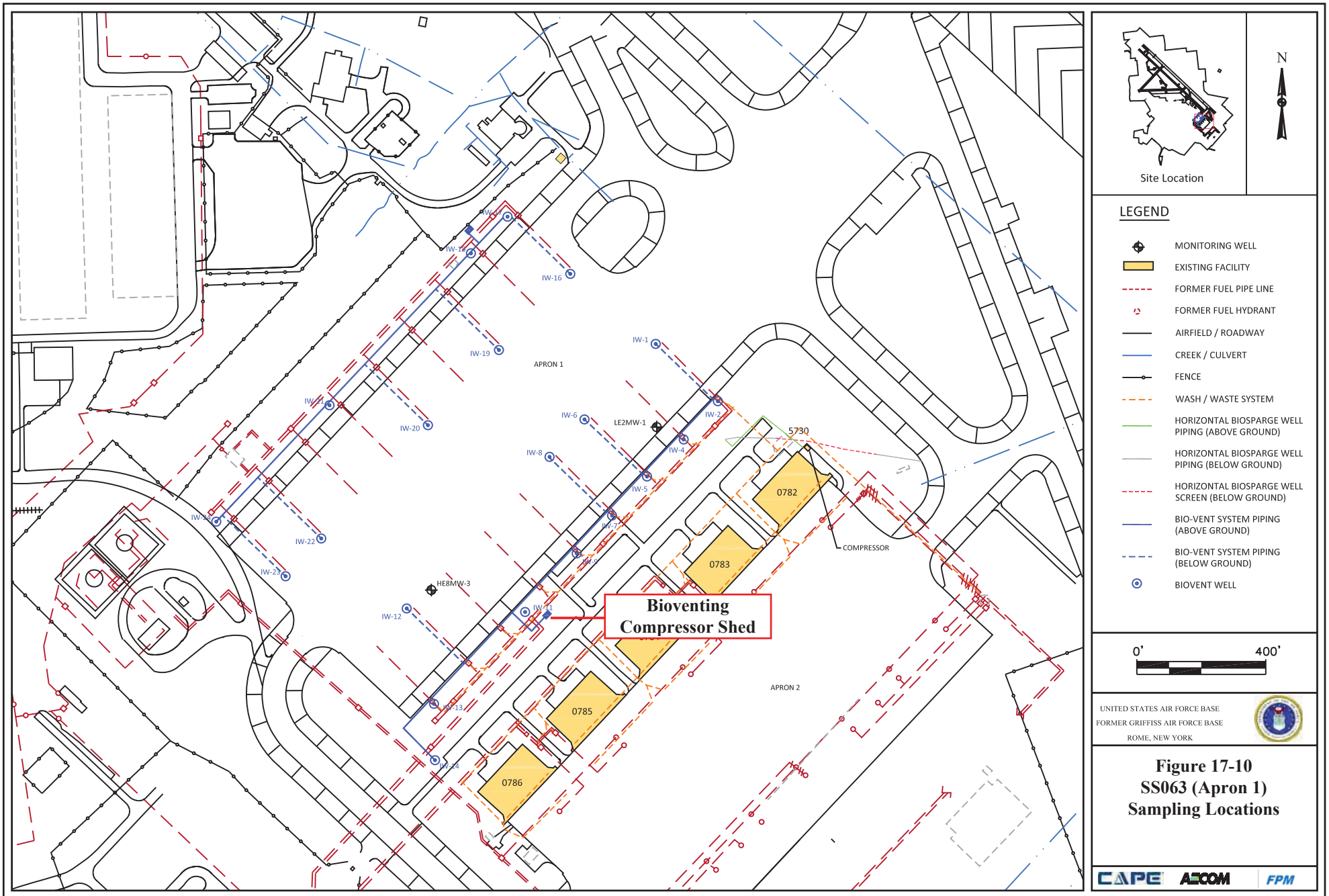

UNITED STATES AIR FORCE BASE
FORMER GRIFFISS AIR FORCE BASE
ROME, NEW YORK

Figure 17-10
SS063 (Apron 1)
Sampling Locations

CAPE AECOM FPM

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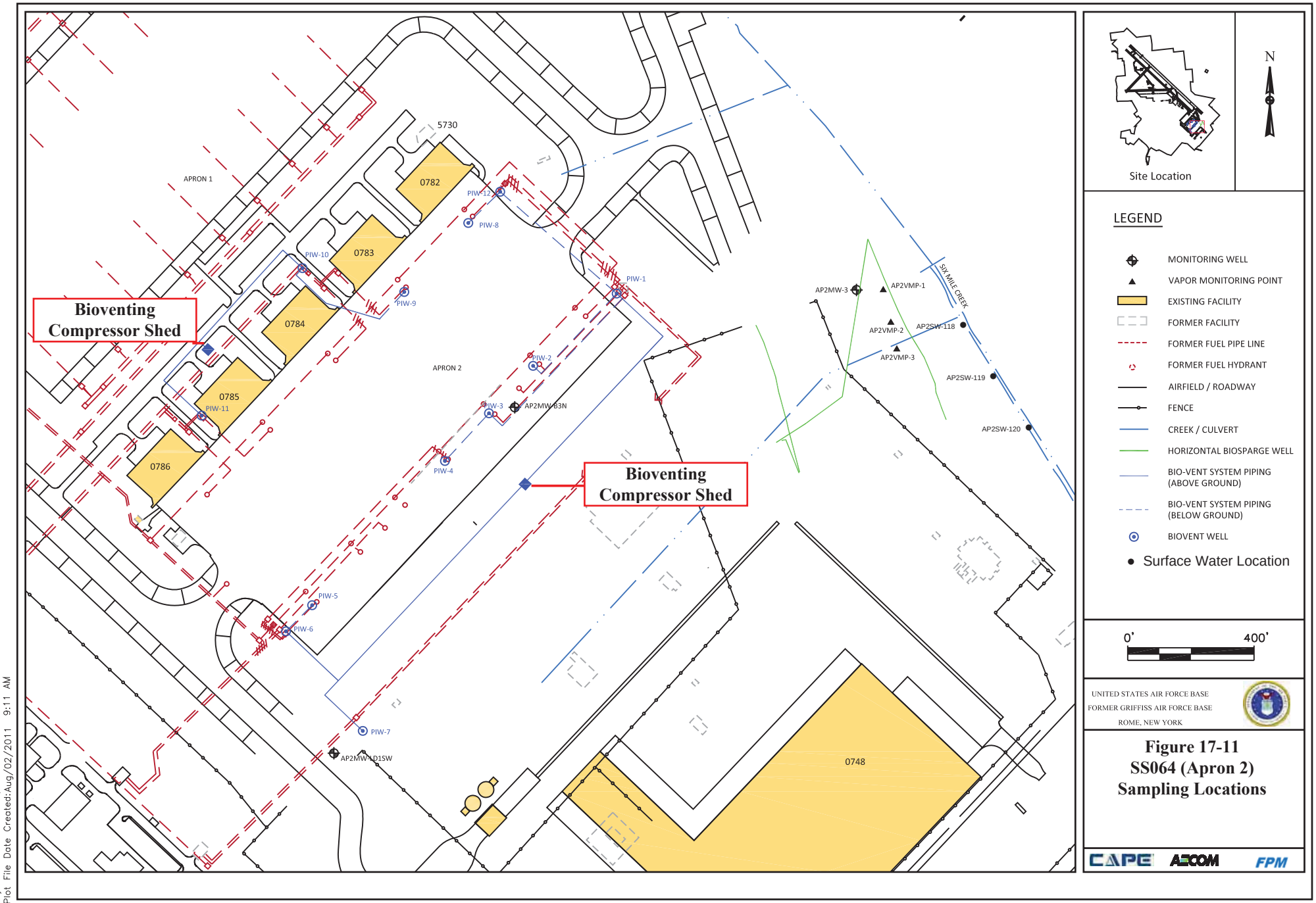

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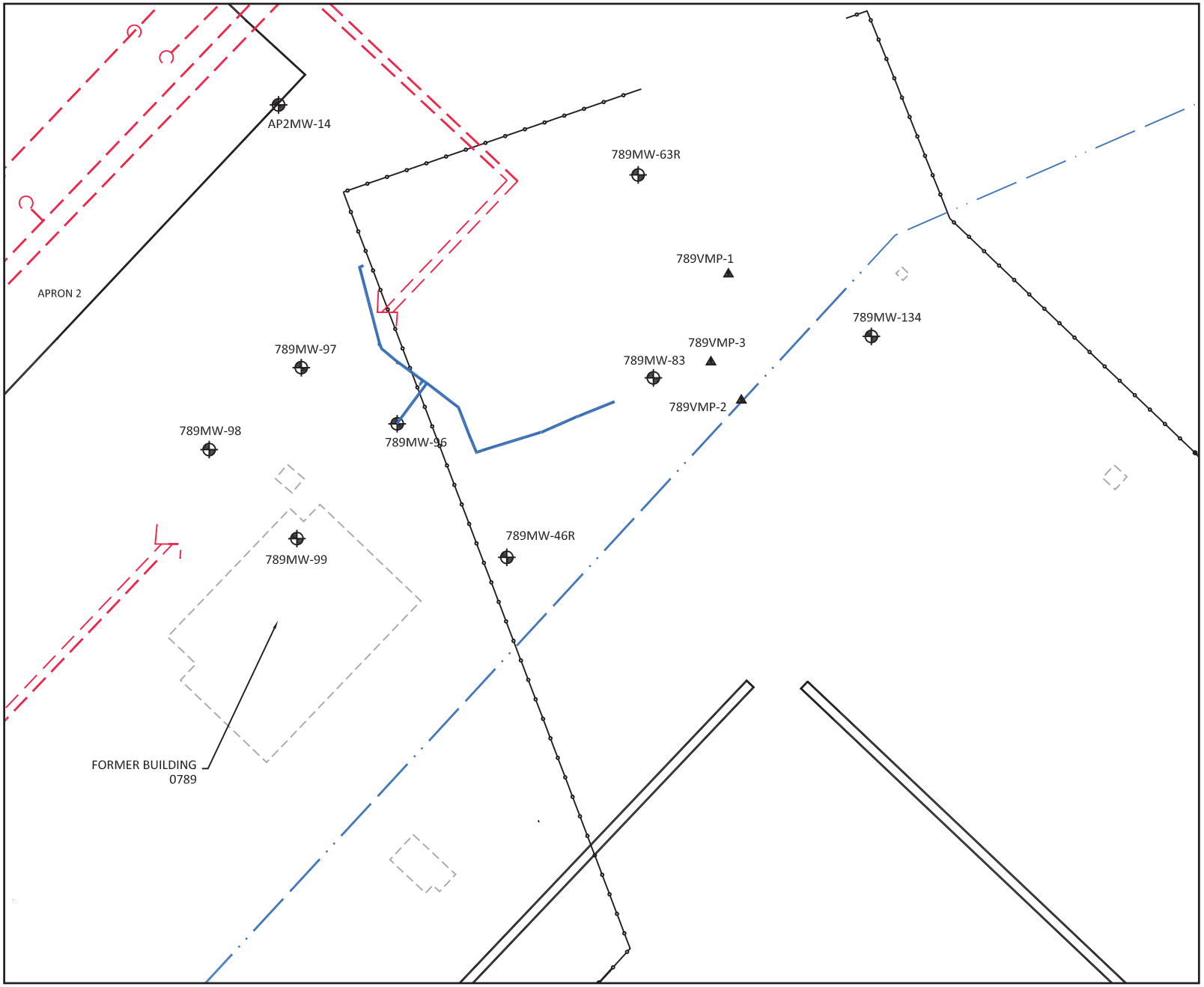

N

Site Location

LEGEND

789MW-63R — MONITORING WELL

789VMP-2 — VAPOR MONITORING POINT

EXISTING FACILITY

FORMER FACILITY

FORMER FUEL PIPE LINE

FORMER FUEL HYDRANT

AIRFIELD / ROADWAY

CREEK / CULVERT

FENCE

VES SYSYTEM

0' 150'

UNITED STATES AIR FORCE BASE
FORMER GRIFFISS AIR FORCE BASE
ROME, NEW YORK

Figure 17-12
SS067 (Building 789)
Sampling Locations

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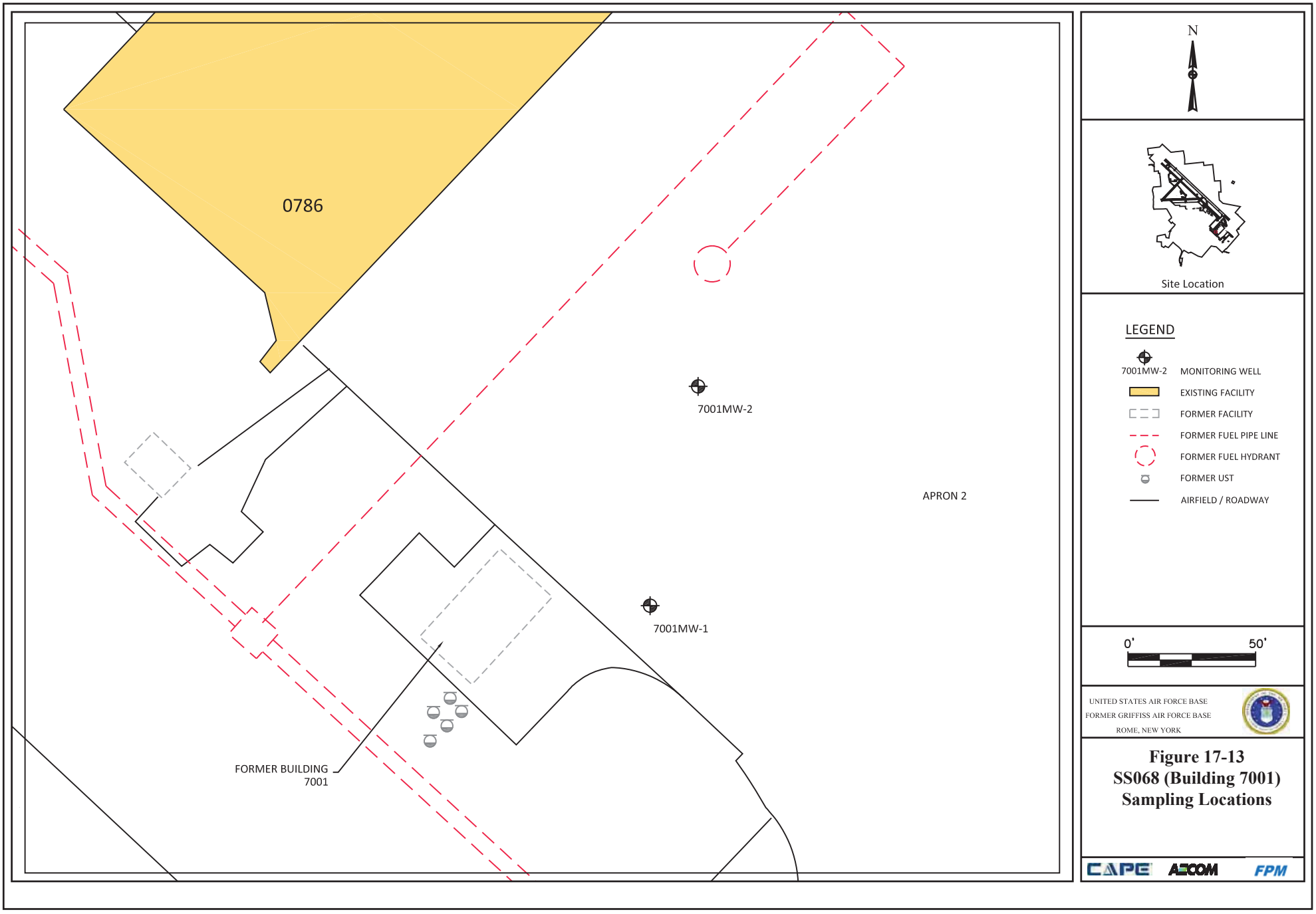

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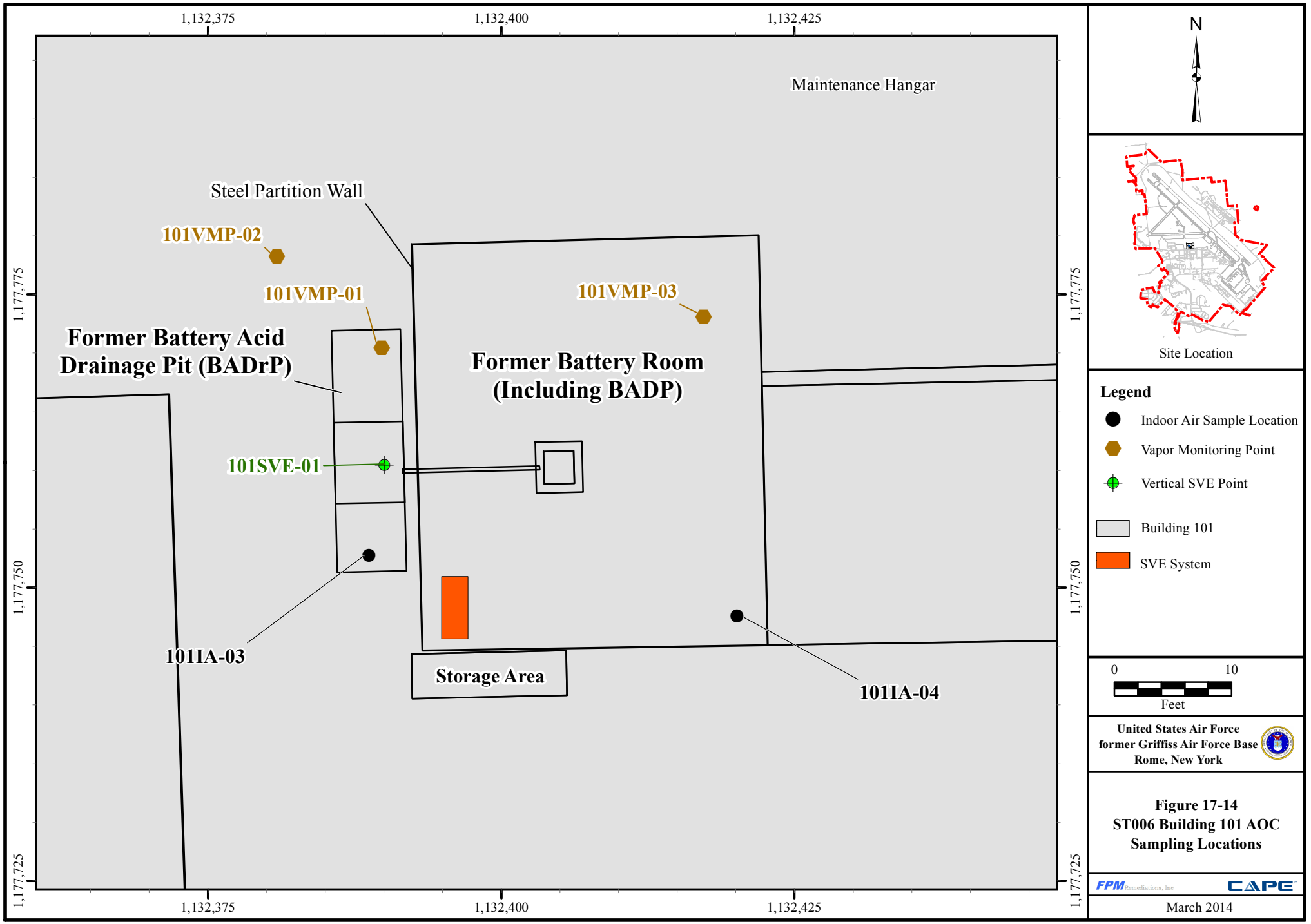

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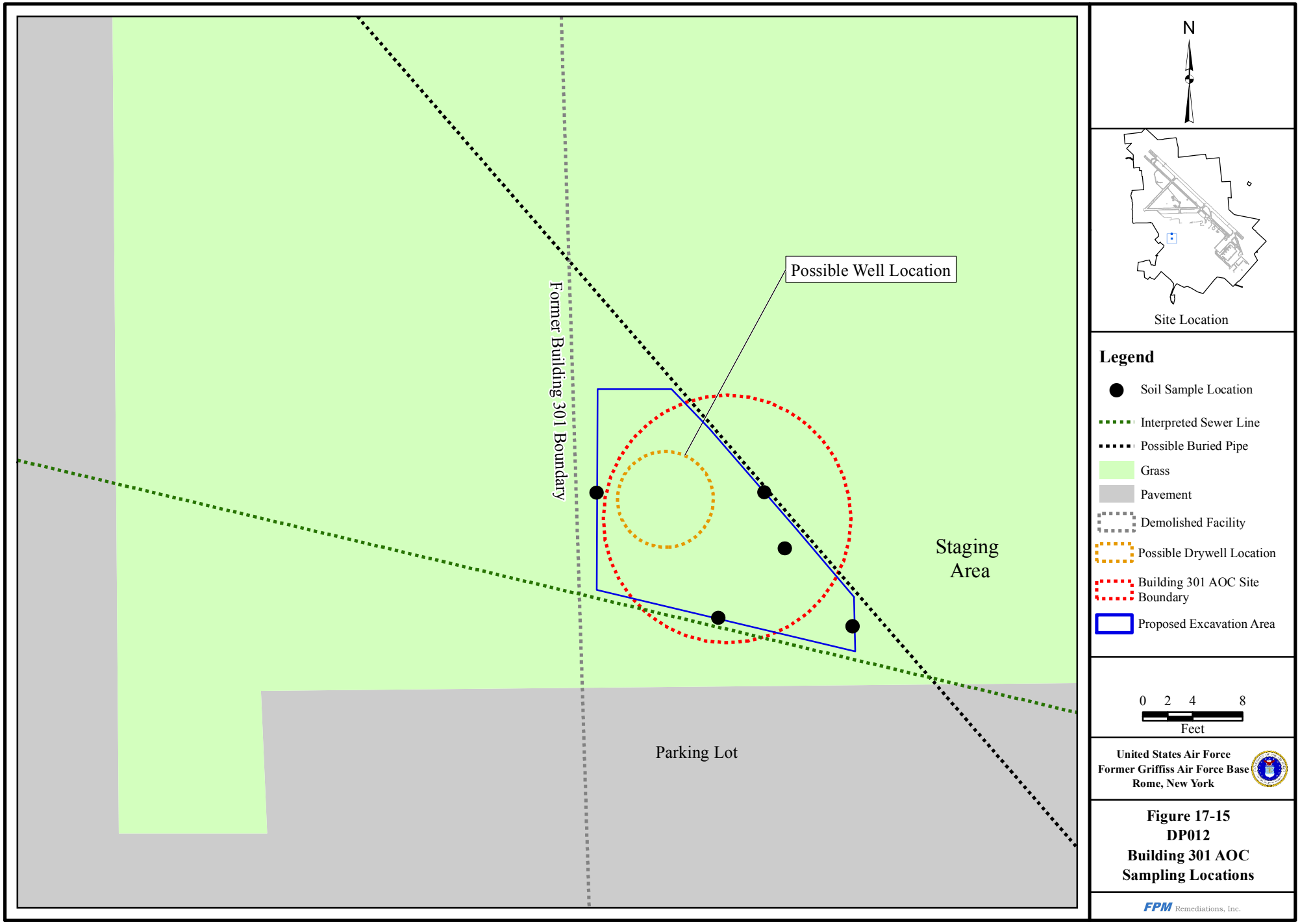

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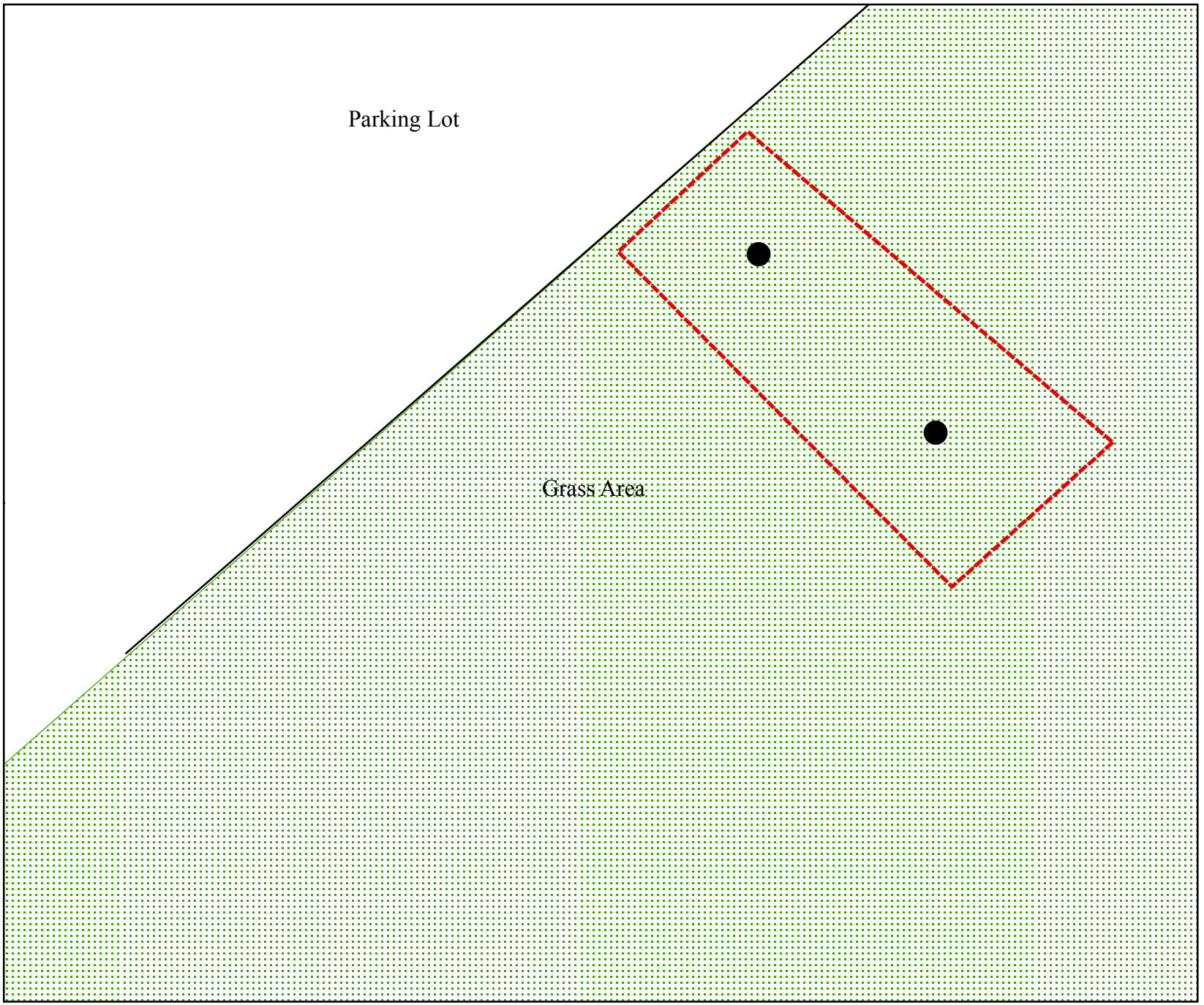

N

Site Location

Legend

- Soil Sample Location
- Parking Lot Boundary
- Proposed Excavation Boundary
- Grassed Area

Feet
0 5 10

United States Air Force
Former Griffiss Air Force Base
Rome, New York

Figure 17-16
AOI 72
Sampling Locations

FPM Remediations, Inc.

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N

Site Location

Legend

- 2013 Soil Boring Sample Location
- Proposed 2014 Soil Boring Sample Location
- Demolished Facility
- Building 255 AOC Site Boundary
- Sampling Area

50 25 0 50

Feet

United States Air Force
Former Griffiss Air Force Base
Rome, New York

Figure 17-17
DP013 Building 255 AOC Sampling Locations

FPM Remediations, Inc.

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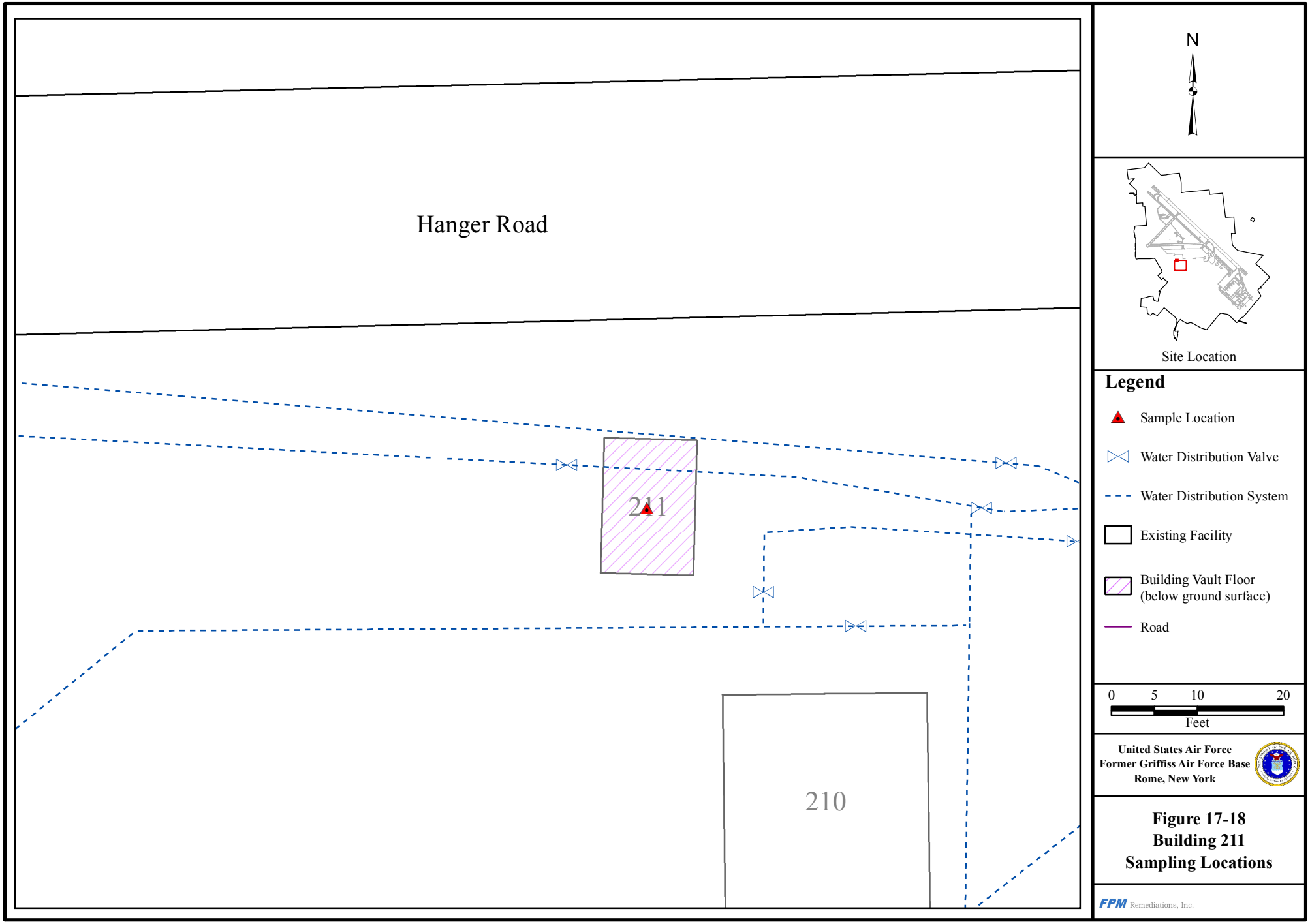

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1 Sub-Surface Soil Sampling

1.1 Purpose and Scope

This Standard Operating Procedure (SOP) describes the equipment, materials, field procedures, and documentation procedures for collecting sub-surface soil samples using direct push or auger methods for soil characterization and chemical analysis.

Health and safety procedures and equipment to be used during soil sampling are described in a separate site-specific Site Safety and Health Plan (SSHP). These SOPs are intended to be used with the former Griffiss AFB Uniform Federal Policy Quality Assurance Project Plan (UFP QAPP), the existing former Griffiss AFB Field Sampling Plan (FSP) and with other SOPs listed below:

SOP No. 6, Sample Handling, Documentation, and Tracking

SOP No. 7, Decontamination

1.2 Equipment and Materials List

One of the following drilling equipment:

Direct push rig (e.g., Geoprobe® rig or similar) with appropriate drilling and sampling tools (sub-surface soil)

Hollow Stem Auger Kit and electric drill

Hand Auger

The following equipment and materials should be on site for sub-surface soil sampling regardless of the drilling equipment used:

Photoionization Detector (PID) (with 10.2 eV lamp)

Weighted tape measure and ruler with 0.01-foot increments

Surveyor's stakes and flags

Field logbook

Drilling Log form

Sample Collection Field Form

Stainless-steel bowl and spoon

Sample containers

Sample container labels

Label tape (clear)

Ziploc® bags

Paper towels

Digital Camera

Waterproof and permanent marking pens

Plastic sheeting

Trash bags

Ice chest with ice

Appropriate health and safety equipment, as specified in the SSHP

Appropriate decontamination supplies, as specified in SOP No. 8

Granular bentonite and potable water

1.3 Locating the Sampling Points

The facilities designated for sampling are shown on figures provided in the UFP-QAPP (Worksheet #17). The approximate soil sampling locations will be identified on site figures before field work commences. The exact soil sampling locations will be determined in the field. Sampling coordinates will be mapped on the front of the Drilling Log in the Location Sketch/Comments Area. The sampling locations will be defined in the investigation specific work plan similar to previous investigation and long term monitoring locations.

When each soil sampling location is identified in the field, the sampling point identification will be entered in the field logbook and on the Drilling Log. Include any information concerning nearby landmarks, or other information that will help to re-locate the point in the future. Mark the sample locations using surveyor's stakes and flags, and label the flag using indelible ink with the sample point identification. A field map will be prepared as the sampling points are laid out to identify locations and tie the locations to site landmarks (such as foundations) if available. If the surveyor's stake is offset from the sample location, the offset will be noted on the field map and the field logbook.

1.4 Soil Sampling Procedures

At several sampling sites, the sampling locations may be in concrete or asphalt covered areas. Therefore, at these locations, cores will be drilled through the concrete or asphalt at areas most likely to contain contamination (significant cracks or low points). Direct push technology will be utilized after the concrete has been cored. Direct push samples will be collected using a dual tube sampling system or a discrete interval, piston-type sampler (Geoprobe®, MacroCore®, or equivalent). With a dual tube system, the outer rods remain in the ground while the inner rod and sample liner are extracted to retrieve a soil sample from the desired interval. Soil samples may be collected continuously throughout the depth of the direct push boring or from discrete intervals. The direct push rods will be decontaminated between boring locations, but not between samples at the same boring since a new acetate liner is used for each sample.

With a piston-type sampler, a four-foot or five-foot-long stainless steel sampler with an acetate liner is advanced to the top of the desired sampling interval. The sampler is closed to soil during advancement of the sampler to the desired sampling interval. When the top of the desired sampling interval is reached, a piston rod inside the sampler is unlocked through the drill rods,

and the sampler is advanced to the bottom of the sampling interval. The sampler and all drill rods are then removed from the ground, and the acetate liner is removed from the piston sampler. Aside from the cutting shoe, the soil sampler never comes in contact with the soil sample. The cutting shoe is decontaminated after each sample collected, and a new acetate liner is used for every sample interval. The outer sampling barrel is decontaminated after each boring is completed. The sampling will be documented in the field logbook and drill log.

With a hand auger or hollow stem auger kit, the auger head will be advanced manually to the depth. Auger extensions will be used when sampling at depths exceeding 4 feet. Once the desired depth is achieved, the auger is removed for sample collection as described below. Following collection, the hand auger or hollow stem auger kit will be decontaminated. When using manual samplers, the sampling will be documented in the field logbook and Soil/Sediment sampling form.

At each sampling location, the sampler will be advanced by a combination of hydraulic vertical pressure and percussion hammering. Once the target depth is achieved, the sample will be withdrawn and the liner filled with the soil sample is retrieved.

The following procedures will be followed once the soil sample has been retrieved:

Don a clean pair of nitrile gloves.

Cut acetate sleeve to provide access to the soil sample (direct push sampling only).

Measure the recovery. Record the sampling interval and recovery on the drilling log.

Remove soil smear from the outside of the acetate sleeve and examine the sample, with particular attention for visible evidence of staining, odors, or other evidence of contamination. Record the soil description on the Drilling Log or Soil/Sediment Sampling Form.

Conduct PID screening of the soil. The soil with the highest PID levels will be collected for a sample.

The soil from the sampling interval will be removed from the liner and homogenized in a stainless-steel bowl. Once the soil has been homogenized, fill the appropriate sample containers as specified in the UFP - QAPP (Worksheet #19). Record the sample interval and analysis requested on the Drilling Log or Soil/Sediment Sampling Form and the chain of custody (COC).

Label, store, transport, and document the samples (depending on the use of the sample) according to SOP No. 7. The parameters for analysis and preservation are specified in UFP QAPP Worksheet #19.

If no other samples will be collected from the boring, abandon the boring by backfilling the hole with hydrated granular bentonite. Pour the granular bentonite down the hole in approximate 1-foot to 2-foot lifts, and then pour approximately 0.5 gallon of potable water down the hole to hydrate the bentonite. Continue this from the bottom of the hole to the surface.

1.5 Field Quality Assurance/Quality Control Samples

Field quality assurance/quality control (QA/QC) samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample

collection and handling. All QA/QC samples will be labeled with QA/QC identification numbers and sent to the laboratory with the other samples for analyses.

1.5.1 Field Blanks

Field blanks are QC samples collected to evaluate potential external contamination of samples and will consist of trip, ambient, and equipment blanks. The sample collection coordinator or the project QA/QC coordinator will designate these blanks. The blanks will be assigned a QA/QC identification number, stored in an iced cooler, and shipped to the laboratory with the other samples.

A trip blank serves as a check on sample contamination originating from the container or sample transport. A trip blank consists of a VOA vial which was filled with VOA-free water at the lab, transported to the site, kept in the same cooler as the normal samples throughout the entire sampling day, and shipped back to the laboratory with the normal samples. One trip blank will be sent with each cooler containing water samples for volatile organic analyses.

The ambient blank serves as a check on sample contamination originating from ambient air during volatile organic compounds (VOCs) sample collection. An ambient blank consists of an empty VOA vial which is filled in the field with VOA free water. While pouring the sample, the water is given ample contact with ambient air conditions. The ambient blank is typically collected at the sampling location that potentially exhibits the largest ambient influence (near a busy road, airfield, etc.).

The equipment blank serves as a check on sample contamination originating from sampling equipment reuse during sample collection. The equipment blank consists of a set of sample bottles identical to the normal sample, which is filled with lab-grade water that is flushed over a decontamined, reusable piece of equipment.

1.5.2 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. All sampling containers will be filled in the following order: volatile or gaseous analyses first, then semi-volatile organic compounds (SVOCs), including polynuclear aromatic hydrocarbons (PAHs); metals; mercury; cyanide; total organic carbon; anions; other remaining analytes (no specific order). The initial sample containers will be filled first, and then the duplicate sample containers for the same parameter(s) and so on until all sample containers for both the initial sample and the duplicate sample have been filled. The duplicate samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

1.5.3 Matrix Spikes and Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) analyses are used to assess the potential for matrix effects. Samples will be designated for MS/MSD analysis on the COC form and on the

containers. It may be necessary to increase the sample volume for MS/MSD samples. If additional volume is necessary, the additional sample containers will be filled in the identical fashion as described above in the duplicate sample section. MS/MSD samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

1.6 Field Documentation

Field documentation for sub-surface soil sampling includes field logbooks and field forms. The most important aspect of field documentation is thorough, organized, and accurate record keeping. Two forms are used in the field during sub-surface soil sampling. These forms include the Drill Log and the Soil/Sediment Sampling Form. Each form is described in Section 1.6.2. An important factor of record keeping is the proper preservation and storage of all field documentation. To preserve the field documentation, the field notes and field forms are scanned and the electronic record of the field notes is stored in the project folder and backed up on additional hard drives to prevent data loss.

Additional forms including Health and Safety Meeting forms, Health and Safety Inspection forms, and COCs used during the sampling event are detailed in SOP No. 7.

1.6.1 Field Logbook

All information pertinent to soil sampling and not documented on the field forms will be recorded in a bound field logbook with consecutively numbered pages. The field logbook notes will be recorded in indelible ink. The field logbooks notes are entered to create an accurate record of the work performed so that the sampling activity can be reconstructed without relying on the memory of field personnel. Information documented in the field logbook may include information on date of notes, weather conditions, field personnel, site, mobilization, work performed including location and time, etc. After each day, field notes are reviewed by the field team leader or site responsible person for accuracy. Refer to SOP No. 7 for detailed procedures regarding documentation in the field logbook.

1.6.2 Field Forms

<u>Drill Log</u>

The Drilling Log contains the following minimum information:

Project name and number

Contractor company, field personnel

Boring Identifier

Drilling subcontractor company and name of drilling personnel

Site Identifier

Brand and model of drill rig

Sizes and types of drilling and sampling equipment

Surface elevation (if available, this may be entered later after the survey)

Date drilling started and finished

Overburden thickness, depth drilled into rock, and total depth of hole

Depth to water during drilling and depth to water after drilling with elapsed time

Number of geotechnical samples, type of samples, and core boxes (if cores are saved)

Number of chemical samples and requested analyses

Signature of field geologist who completed the Drilling Log field form

Field sketch showing the boring location

Sampling interval and measured sample recovery.

A description of the recovered soil sample in accordance with the Unified Soil Classification method for unconsolidated geologic materials. The descriptions should include origin, grain size, sorting, texture, structure, bedding, color, moisture content, and consistency.

Sample Identifier

Sample Collection Time

As applicable, field screening results, geotechnical samples, chemical samples, and blow counts (split-spoon sampling only).

As applicable, record pertinent observations (such as odors, staining, colors, changes in drill rod advancement, chatter, water, etc.) in the "Remarks" column.

If portions of the Drilling Log are not applicable (e.g., if samples are not collected for chemical analysis or if cores are not collected, etc.) record an "NA" in the appropriate location on the form.

Bore hole abandonment (method of abandonment)

Soil/Sediment Sampling Form

The Soil/Sediment Sampling Form contains the following minimum information:

Field personnel

Project name and number

Site Identifier

Sample Location Identifier

Sizes and types of sampling equipment

Date of sample

Sampling depth.

A description of the recovered soil sample. The descriptions should include origin, grain size, texture, structure, color, and odor.

Comments or Observations
Sample Identifier
Sample Collection Time

2 Groundwater Sampling

2.1 Purpose and Scope

This section defines the SOP for the collection of groundwater samples at the former Griffiss AFB. This procedure describes equipment, field procedures, and QA/QC procedures necessary to collect groundwater samples. The sample locations and frequency of collection are specified in the project UFP QAPP.

This SOP is intended to be used together with the UFP QAPP and other appropriate SOPs. Health and safety procedures and equipment that will be required during the investigation are detailed in the SSHP. Applicable SOPs are listed below:

SOP No. 6, Sample Handling, Documentation, and Tracking

SOP No. 7, Decontamination

2.2 Equipment and Materials List

The following equipment will be used during well purging and sampling:

<u>Bailer Sampling:</u>
Well lock keys (if required)

Water level probe with 0.01-foot intervals

Assorted tools (knife, screwdriver, etc.)

Disposable bailers

Nylon rope

Multi-parameter water quality meter (Horiba U-52, YSI 556, or similar)

Calibration fluids

Plastic squeeze or spray bottle filled with de-ionized water

Plastic or glass container (for field parameter measurements)

Paper towels

Calculator

Field logbook

Waterproof and permanent marker

Appropriate containers for holding purged water

Appropriate health and safety equipment, as specified in the SSHP

Well purging and sampling form for bailer sampling

Appropriate decontamination supplies, as specified in SOP No. 8.

Cooler with ice

Garbage bag

Sample labels

Sample bottles with preservatives added will be obtained from the analytical laboratory

Several extra sample bottles in case of breakage or other problems

Low Flow Sampling:

Well lock keys (if required)

Water level probe with 0.01-foot intervals

Assorted tools (knife, screwdriver, etc.)

QED MP10 micropurge digital controller (or similar)/ Well wizard® 3020 oil-less battery powered electric compressor (or similar)

Marine battery

Multi-parameter water quality meter (Horiba U-52, YSI 556, or similar)

Polyethylene tubing (assorted diameters)

Flow-through cell

Plastic, see-through measuring cup (2 cups size)

Calibration fluids

Plastic squeeze or spray bottle filled with de-ionized water

Polyethylene (PE) or glass container (for field parameter measurements)

Paper towels

Garbage bags

Calculator

Field logbook

Waterproof and permanent marker

Appropriate containers for holding purge water

Appropriate health and safety equipment, as specified in the SSHP

Well purging and sampling form for low-flow sampling

Appropriate decontamination equipment, as specified in the SOP No. 8

Cooler with ice

Sample labels

Sample bottles with preservatives added will be obtained from the analytical laboratory

Several extra sample bottles in case of breakage or other problems

2.3 Identifying the Groundwater Sampling Locations

The groundwater sampling locations will be identified in the site-specific work plan (WP). All existing monitoring wells have been surveyed by a certified surveyor and included on maps and figures. All additional monitoring wells will be surveyed after well completion and development.

2.4 Groundwater Sampling Procedures

This section summarizes the step-by-step procedures for collecting groundwater samples in the field. Observations made during sample collection will be recorded in the field notebook and on the well purging and sampling form.

The purpose of well purging is to remove stagnant water from the well and obtain representative water from the geologic formation while minimizing disturbance to the collected samples. Before a sample is collected, the well will be purged until field parameters have stabilized or until the well is pumped or bailed dry. Evacuated groundwater shall be contained for proper disposal if the groundwater is significantly impacted (i.e., heavy sheen or free product), and necessary precautions shall be taken to prevent spilling of water. The following Sections 2.4.1 and 2.4.2 detail sample collection using the bailer collection method and low flow collection methods.

2.4.1 Bailer

Before well purging begins, the following procedures will be performed at each well:

The condition of the outer well casing, concrete well pad, and any unusual conditions of the area around the well will be noted in the field logbook.

The well will be opened.

The condition of the inner well cap and casing will be noted.

The depth of static water level and total well depth will be measured (to nearest 0.01 foot) and recorded from a measuring point on the well casing. The measuring point should be identified, and time indicated in the field logbook.

The volume of water in the well casing will be calculated in gallons based on water column height and casing diameter. Three casing volumes will be calculated.

An initial sample will be obtained for field measurements of temperature, pH, conductivity, turbidity, dissolved oxygen (DO), and oxygen reduction potential (ORP) and for observation of water quality. These measurements will also be used during the evaluation of chemical analytical data.

Three water volumes will be purged. Temperature, pH, conductivity, turbidity, DO, and ORP measurements will be recorded at a minimum of one set of readings per well casing volume purged to determine whether the water chemistry has stabilized. Generally, pH values within $\pm$0.1 pH unit, temperature within $\pm 1^{o}$C, and conductivity within $\pm$ 5% milli-siemens per centimeter (ms/cm) between three consecutive readings indicate adequate stability of the water

chemistry. If the parameters are not stable, purging will continue, measuring pH, temperature, and conductivity after each one-half well volume.

If the well is bailed dry during evacuation, it will be assumed that the purpose of removing 3 well volumes of water has been accomplished, that is, removing all stagnant water which had prolonged contact with the well casing or air. If recovery is very slow, samples may be obtained as soon as sufficient water is available.

The following sampling procedure is followed when using disposable bailers:

Typically, new disposable equipment (PE bailer and nylon rope) are used for each sampling location. Decontaminated sampling equipment will be assembled if necessary.

All sample bottles for all analyses will be gathered and identification labels for each sample bottle will be completed for each sample and affixed to the bottles.

The bailer will be lowered **slowly** and **gently** into contact with the water in the well. The well will be checked for light and dense NAPL. After checking for the presence of NAPL, the bailer will be lowered to the same depth in the well each time.

The bailer will be retrieved **smoothly** and the water will be **slowly** drained into the sample containers through the bailer's bottom discharge control device.

The individual sample bottles should be filled in the order given below:

i. VOCs
ii. Alkalinity
iii. SVOCs
iv. Metals
v. Mercury
vi. Cyanide
vii. Total Organic Carbon
viii. Anions
ix. Other remaining analytes (no specific order)

VOC sample vials should be completely filled so the water forms a convex meniscus at the top, then capped so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, discard the sample vial and repeat the procedure until no air bubbles appear.

Alkalinity sample bottles are also collected so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, add additional water until no air bubbles appear.

Fill bottles for SVOCs, metals and other analytes until almost full.

Time of sampling will be recorded.

The bailer and string will be removed from the well and placed in garbage bags for proper disposal as household waste.

The well cap will be replaced and locked.

Field documentation will be completed, including the COC.

2.4.2 Low Flow

Before well purging begins, the following procedures will be performed at each well:

The condition of the outer well casing, concrete well pad, and any unusual conditions of the area around the well will be noted in the field logbook.

The well will be opened.

The condition of the inner well cap, casing and associated tubing will be noted.

The depth of static water level will be measured (to nearest 0.01 foot) and recorded from a measuring point on the well casing, the measuring point should be identified, and time indicated in the field logbook.

The low flow equipment is set-up at the well. The set-up includes:

Connect the Well Wizard® compressor to the marine battery.

Connect air hose from MP10 controller and the Well Wizard® compressor and air hose from the MP10 controller to the air intake of the dedicated bladder pump.

Connect multi-parameter water quality meter with flow-through cell will be connected with new disposable PE tubing to the dedicated bladder pump and associated tubing.

The water purge rate will be set within the range of 100 to 500 ml per minute.

During water purging, water will flow through the multi-parameter water quality equipment flow-through cell. Temperature, pH, conductivity, DO, ORP, and turbidity measurements will be recorded until the parameters have stabilized. Measurements will be collected for each flow-through cell volume. Stabilization parameters are: pH values is $\pm$0.1 pH unit, conductivity values is $\pm$ 3% mS/cm, turbidity is $\pm$ 10% nephelometric turbidity units (NTU) or general turbidity readings below 50 NTU, dissolved oxygen is $\pm$ 10% milligrams per liter (mg/L), and ORP is $\pm$ 10 milliVolts (mV) for three consecutive measurement events. If the parameters are not stable, purging will continue for a maximum of one hour.

The following sampling procedure is to be used when using the low-flow method:

All sample bottles for all analyses for the sampling locations are organized and identification labels for all sample bottles are completed.

The discharge PE tubing will be unhooked from the flow-through cell.

Groundwater samples are collected with water purge rates at or below 250 ml/min.

The individual sample bottles should be filled in the order given below:

i. VOCs
ii. Alkalinity
iii. SVOCs
iv. Metals
v. Mercury
vi. Cyanide
vii. Total Organic Carbon
viii. Anions
ix. Other remaining analytes (no specific order)

VOC sample vials should be completely filled so the water forms a convex meniscus at the top, then capped so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, discard the sample vial and repeat the procedure until no air bubbles appear.

Alkalinity sample bottles are also collected so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, add additional water until no air bubbles appear.

Fill bottles for SVOCs, metals and other analytes until almost full.

Time of sampling will be recorded.

The sampling equipment is turned off and disconnected from the well.

The well cap will be replaced and locked.

Field documentation will be completed, including the COC.

2.5 Field Quality Assurance/Quality Control Samples

The well sampling order will be dependent on expected levels of contamination in each well, if known, and will be determined prior to sampling. Typically, the sampling order of the monitoring wells is from the least contaminated well to the most contaminated well. QA/QC samples will be collected during groundwater sampling.

Field QA/QC samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample collection and handling. All QA/QC samples are labeled with QA/QC identification numbers and sent to the laboratory in the same batch as the normal samples for analyses.

2.5.1 Field Blanks

Field blanks are QC samples that check for potential external contamination of samples and will consist of trip, ambient, and equipment blanks. The sample collection coordinator or the project QA/QC coordinator will designate these blanks. The blanks will be assigned a QA/QC identification number, stored in an iced cooler, and shipped to the laboratory with the other samples.

A trip blank serves as a check on sample contamination originating from the container or sample transport. A trip blank consists of a VOA vial which was filled with VOA-free water at the lab, transported to the site, kept in the same cooler as the normal samples throughout the entire sampling day, and shipped back to the laboratory with the normal samples. One trip blank will be sent with each cooler containing water samples for volatile organic analyses.

The ambient blank serves as a check on sample contamination originating from ambient air during VOCs sample collection. An ambient blank consists of an empty VOA vial which is filled in the field with VOA free water. While pouring the sample, the water is given ample contact with ambient air conditions. The ambient blank is typically collected at the sampling location that potentially exhibits the largest ambient influence (near a busy road, airfield, etc.).

The equipment blank serves as a check on sample contamination originating from sampling equipment reuse during sample collection. The equipment blank consists of a set of sample bottles identical to the normal sample, which is filled with lab-grade water that is flushed over a decontamined, reusable piece of equipment.

2.5.2 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. All sampling containers will be filled in the following order: volatile or gaseous analyses first, then SVOCs, including PAHs; metals; mercury; cyanide; total organic carbon; anions; other remaining analytes (no specific order). The initial sample containers will be filled first, and then the duplicate sample containers for the same parameter(s) and so on until all necessary sample containers for both the initial sample and the duplicate sample have been filled. The duplicate samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

2.5.3 Matrix Spikes and Matrix Spike Duplicates

MS and MSD analyses are used to assess the potential for matrix effects. Samples will be designated for MS/MSD analysis on the COC form and on the containers. It may be necessary to increase the sample volume for MS/MSD samples. If additional volume is necessary, the additional sample container will be filled in the identical fashion as described above in the duplicate sample section. MS/MSD samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

2.6 Field Documentation

Field documentation for groundwater sampling includes field logbooks and field forms. The most important aspect of field documentation is thorough, organized, and accurate record keeping. Two forms are used in the field during groundwater sampling. These forms include the Bailer Sampling Form and the Low-Flow Sampling Form. Each form is described in Section

2.6.2. An important factor of record keeping is the proper preservation and storage of all field documentation. To preserve the field documentation, the field notes and field forms are scanned and the electronic record of the field notes is stored in the project folder and backed up on additional hard drives to prevent data loss. The field forms will also be provided in the Daily Chemical Quality Control Reports (CQCR).

Additional forms including Health and Safety Meeting forms, Health and Safety Inspection forms, COCs, used during the sampling event are detailed in SOP No. 7.

2.6.1 Field Logbook

All information pertinent to soil sampling and not documented on the field forms will be recorded in a bound field logbook with consecutively numbered pages. The field logbook notes will be recorded in indelible ink. The field logbooks notes are entered to create an accurate record of the work performed so that the sampling activity can be reconstructed without relying on the memory of field personnel. Information documented in the field logbook may include information on date of notes, weather conditions, field personnel, site, mobilization, work performed including location and time, etc. After each day, field notes are reviewed by the field team leader or site responsible person for accuracy. Refer to SOP No. 7 for detailed procedures regarding documentation in the field logbook.

2.6.2 Field Forms

<u>Bailer Sampling Form</u>

The Bailer Sampling Form contains the following minimum information:

Project name and number

Sampling personnel

Site Identifier

Date of sample

Well number

Well Diameter

Weather conditions

Depth to water and total depth of well

Purge volume calculations

Purge date

Purge method

Water characteristics and appearance

Water parameter measurement results (pH, conductivity, temperature, turbidity, DO, and ORP)

Sample ID

Sample time
Any QA/QC Samples

Low-Flow Sampling Form

The Low-Flow Sampling Form contains the following minimum information:

Project name and number
Sampling personnel
Site Identifier
Date of sample
Well number
Well Diameter
Weather conditions
Depth to water and total depth of well
Pump intake depth
Depth of water during purging
Purge date
Purge method
Water characteristics and appearance
Water parameter measurement results (pH, conductivity, temperature, turbidity, DO, and ORP)
Purge rate
Sample ID
Sample time
Any QA/QC Samples

3 Surface Water Sampling

3.1 Purpose and Scope

The purpose of this section is to define the SOP for collecting surface water samples at the former Griffiss AFB. This SOP describes the equipment, field procedures, and QA/QC procedures implemented for the using the hand tools for collecting grab surface water samples.

This SOP is intended to be used together with the FSP and other appropriate SOPs. Health and safety procedures and equipment for the investigation are detailed in the project SSHP.

Applicable SOPs are listed below:

SOP No. 6, Sample Handling, Documentation, and Tracking

SOP No. 7, Decontamination

3.2 Equipment and Materials List

The following equipment and materials should be on site for surface water sampling:

Long-handled stainless steel sample cup

Multi-parameter water quality meter (Horiba U-52 or YSI 556 or similar)

Surveyor's stakes and flags

Field logbook

Field Sampling Forms

Container to hold water for water chemistry parameter measurements

Stainless steel surface water grab sampler (long handled stainless steel cup)

Sample containers

Sample container labels

Label tape (clear)

Ziploc® bags

Paper towels

Digital camera

Waterproof and permanent marking pens

Trash bags

Ice chest with ice

Appropriate health and safety equipment, as specified in the SSHP

Appropriate decontamination supplies, as specified in SOP No. 8

3.3 Locating the Sampling Points

The surface water sampling locations will be identified in the site specific WP and will be identical to current LTM sample locations. These locations have been plotted on sampling location maps for each site.

3.4 Surface water Sampling Procedures

The following procedures will be followed to collect surface water samples:

Decontaminate sampling equipment according to SOP No. 8.

Don a clean pair of nitrile gloves.

Collect surface water using grab sampler and place water into container for water chemistry parameters. The sample for these parameters will be collected while minimizing disturbance to the surface water or sediment in the creek.

Water quality measurements will be collected with a multi-parameter water quality system before sampling and will include pH, temperature, specific conductance, dissolved oxygen, ORP, and turbidity at each surface water sampling location. Measurements will be recorded on the field sampling form immediately.

After water quality parameter collection, the surface water sample will be collected using a grab sampler. Previously preserved sample bottles will be filled by pouring sample water from this cup.

The individual sample bottles should be filled in the order given below:

i. VOCs
ii. Alkalinity
iii. SVOCs
iv. Metals
v. Mercury
vi. Cyanide
vii. Total Organic Carbon
viii. Anions
ix. Other remaining analytes (no specific order)

VOC sample vials should be completely filled so the water forms a convex meniscus at the top, then capped so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, discard the sample vial and repeat the procedure until no air bubbles appear.

Alkalinity sample bottles are also collected so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, add additional water until no air bubbles appear.

Fill bottles for SVOCs, metals and other analytes until almost full.

Time of sampling will be recorded.

3.5 Field Quality Assurance/Quality Control Samples

Field QA/QC samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample collection and handling. All QA/QC samples will be labeled with QA/QC identification numbers and sent to the laboratory with the other samples for analyses.

3.5.1 Field Blanks

Field blanks are QC samples that check for potential external contamination of samples and will consist of trip, ambient, and equipment blanks. The sample collection coordinator or the project QA/QC coordinator will designate these blanks. The blanks will be assigned a QA/QC identification number, stored in an iced cooler, and shipped to the laboratory with the other samples.

A trip blank serves as a check on sample contamination originating from the container or sample transport. A trip blank consists of a VOA vial which was filled with VOA-free water at the lab, transported to the site, kept in the same cooler as the normal samples throughout the entire sampling day, and shipped back to the laboratory with the normal samples. One trip blank will be sent with each cooler containing water samples for volatile organic analyses.

The ambient blank serves as a check on sample contamination originating from ambient air during VOCs sample collection. An ambient blank consists of an empty VOA vial which is filled in the field with VOA free water. While pouring the sample, the water is given ample contact with ambient air conditions. The ambient blank is typically collected at the sampling location that potentially exhibits the largest ambient influence (near a busy road, airfield, etc.)

The equipment blank serves as a check on sample contamination originating from sampling equipment reuse during sample collection. The equipment blank consists of a set of sample bottles identical to the normal sample, which is filled with lab-grade water that is flushed over a decontamined, reusable piece of equipment.

3.5.2 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. All sampling containers will be filled in the following order: volatile or gaseous analyses first, then SVOCs, including PAHs; metals; mercury; cyanide; total organic carbon; anions; other remaining analytes (no specific order). The initial sample containers will be filled first, and then the duplicate sample containers for the same parameter(s) and so on until all necessary sample containers for both the initial sample and the duplicate sample have been filled. The duplicate samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

3.5.3 Matrix Spikes and Matrix Spike Duplicates

MS and MSD analyses are used to assess the potential for matrix effects. Samples will be designated for MS/MSD analysis on the COC form and on the containers. It may be necessary to increase the sample volume for MS/MSD samples. If additional volume is necessary, the additional sample container will be filled in the identical fashion as described above in the duplicate sample section. MS/MSD samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

3.6 Field Documentation

Field documentation for surface water sampling includes field logbooks and field forms. The most important aspect of field documentation is thorough, organized, and accurate record keeping. Field forms used during the surface water sampling include the Bailer Sampling Form. This form is described in Section 3.6.2 and was used for surface water sampling in previous LTM sampling rounds. An important factor of record keeping is the proper preservation and storage of all field documentation. To preserve the field documentation, the field notes and field forms are scanned and the electronic record of the field notes is stored in the project folder and backed up on additional hard drives to prevent data loss. The field forms will also be provided in the Daily Chemical Quality Control Reports (CQCR).

Additional forms including Health and Safety Meeting forms, Health and Safety Inspection forms, and COCs used during the sampling event are detailed in SOP No. 7.

3.6.1 Field Logbook

All information pertinent to soil sampling and not documented on the field forms will be recorded in a bound field logbook with consecutively numbered pages. The field logbook notes will be recorded in indelible ink. The field logbooks notes are entered to create an accurate record of the work performed so that the sampling activity can be reconstructed without relying on the memory of field personnel. Information documented in the field logbook may include information on date of notes, weather conditions, field personnel, site, mobilization, work performed including location and time, etc. After each day, field notes are reviewed by the field team leader or site responsible person for accuracy. Refer to SOP No. 7 for detailed procedures regarding documentation in the field logbook.

3.6.2 Field Forms

<u>Bailer Sampling Form</u>

The Bailer Sampling Form used during the surface water sampling contains the following minimum information:

Project name and number

Sampling personnel
Site Identifier
Date of sample
Sample location number
Weather conditions
Collection method
Water characteristics and appearance
Water parameter measurement results (pH, conductivity, temperature, turbidity, DO, and ORP)
Sample ID
Sample time
Any QA/QC Samples

4 Surface Soil and Sediment Sampling

4.1 Purpose and Scope

The purpose of this section is to define the SOP for collecting surface soil and sediment samples at the former Griffiss AFB using hand tools. This SOP describes the equipment, field procedures, and QA/QC procedures implemented for the using the Dutch auger, hollow stem auger (HSA), hand auger or shovel for surface soil and sediment sampling.

This SOP is intended to be used together with the FSP and other appropriate SOPs. Health and safety procedures and equipment for the investigation are detailed in the project SSHP.

Applicable SOPs are listed below:

SOP No. 6, Sample Handling, Documentation, and Tracking

SOP No. 7, Decontamination

4.2 Equipment and Materials List

One of the following hand-drilling equipment:

Stainless steel hand auger or hand trowel

Hollow stem auger

Dutch auger

Shovel

The following equipment and materials should be on site for surface soil or sediment sampling, regardless of the equipment used:

Surveyor's stakes and flags

Field logbook

Field Sampling Forms

Stainless-steel bowl and spoon

Sample containers

Sample container labels

Label tape (clear)

Ziploc® bags

Paper towels

Digital camera

Waterproof and permanent marking pens

Trash bags

Ice chest with ice

Appropriate health and safety equipment, as specified in the SSHP

Appropriate decontamination supplies, as specified in SOP No. 8

4.3 Locating the Sampling Points

Surface soil and sediment sampling locations will be identified in the site specific WP and will be identical to current LTM sample locations. The sampling locations designated for sampling are shown on figures in the UFP-QAPP (Worksheet #17). At the time of locating each sampling point, enter the sampling point identification in the field logbook and LTM sample location maps.

Sediment sampling locations will be detailed in the site specific WP and will be identical to the current LTM sample locations. These locations have been plotted on sampling location maps for each site. The sample locations will be identified in the field by fiberglass stakes with ID tags.

4.4 Surface Soil and Sediment Sampling Procedures

The following procedures will be followed to collect surface soil and sediment samples:

Decontaminate sampling equipment according to SOP No. 8.

Don a clean pair of nitrile gloves.

Clear and remove vegetation and surface debris as necessary.

Collect a sample using hand drilling equipment and deposit it in a stainless steel bowl or Ziploc® bags.

Homogenize the sample with a stainless steel spoon or by manipulating the Ziploc® bag. Remove any rocks and gravel or foreign material that might interfere with the sample collection.

Deposit an aliquot of the homogenized soil into the sampling container.

Label, store, transport, and document the samples (depending on the use of the sample) according to SOP No. 7. The parameters for analysis and preservation are specified in Worksheet #19 of the project-specific UFP QAPP.

4.5 Field Quality Assurance/Quality Control Samples

Field QA/QC samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample collection and handling. All QA/QC samples will be labeled with QA/QC identification numbers and sent to the laboratory with the other samples for analyses.

4.5.1 Field Blanks

Field blanks are QC samples that check for potential external contamination of samples and will consist of trip, ambient, and equipment blanks. The sample collection coordinator or the project QA/QC coordinator will designate these blanks. The blanks will be assigned a QA/QC identification number, stored in an iced cooler, and shipped to the laboratory with the other samples.

A trip blank serves as a check on sample contamination originating from the container or sample transport. A trip blank consists of a VOA vial which was filled with VOA-free water at the lab, transported to the site, kept in the same cooler as the normal samples throughout the entire sampling day, and shipped back to the laboratory with the normal samples. One trip blank will be sent with each cooler containing water samples for volatile organic analyses.

The ambient blank serves as a check on sample contamination originating from ambient air during VOCs sample collection. An ambient blank consists of an empty VOA vial which is filled in the field with VOA free water. While pouring the sample, the water is given ample contact with ambient air conditions. The ambient blank is typically collected at the sampling location that potentially exhibits the largest ambient influence (near a busy road, airfield, etc.)

The equipment blank serves as a check on sample contamination originating from sampling equipment reuse during sample collection. The equipment blank consists of a set of sample bottles identical to the normal sample, which is filled with lab-grade water that is flushed over a decontamined, reusable piece of equipment.

4.5.2 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. All sampling containers will be filled in the following order: volatile or gaseous analyses first, then SVOCs, including PAHs; metals; mercury; cyanide; total organic carbon; anions; other remaining analytes (no specific order). The initial sample containers will be filled first, and then the duplicate sample containers for the same parameter(s) and so on until all necessary sample containers for both the initial sample and the duplicate sample have been filled. The duplicate samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

4.5.3 Matrix Spikes and Matrix Spike Duplicates

MS and (MSD analyses are used to assess the potential for matrix effects. Samples will be designated for MS/MSD analysis on the COC form and on the containers. It may be necessary to increase the sample volume for MS/MSD samples. If additional volume is necessary, the additional sample container will be filled in the identical fashion as described above in the duplicate sample section. MS/MSD samples will be handled, preserved, stored, and shipped in

the same manner as the primary samples. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

4.6 Field Documentation

Field documentation for surface soil/sediment sampling includes field logbooks and field forms. The most important aspect of field documentation is thorough, organized, and accurate record keeping. The field form includes the soil/sediment sampling form and is described in section 4.6.2. An important factor of record keeping is the proper preservation and storage of all field documentation. To preserve the field documentation, the field notes and field forms are scanned and the electronic record of the field notes is stored in the project folder and backed up on additional hard drives to prevent data loss. The field forms will also be provided in the Daily CQCRs.

Additional forms including Health and Safety Meeting forms, Health and Safety Inspection forms, and COCs used during the sampling event are detailed in SOP No. 7.

4.6.1 Field Logbook

All information pertinent to soil sampling and not documented on the field forms will be recorded in a bound field logbook with consecutively numbered pages. The field logbook notes will be recorded in indelible ink. The field logbooks notes are entered to create an accurate record of the work performed so that the sampling activity can be reconstructed without relying on the memory of field personnel. Information documented in the field logbook may include information on date of notes, weather conditions, field personnel, site, mobilization, work performed including location and time, etc. After each day, field notes are reviewed by the field team leader or site responsible person for accuracy. Refer to SOP No. 7 for detailed procedures regarding documentation in the field logbook.

4.6.2 Field Forms

<u>Soil/Sediment Sampling Form</u>

The Soil/Sediment Sampling Form contains the following minimum information:

Field personnel

Project name and number

Site Identifier

Sample Location Identifier

Sizes and types of sampling equipment

Date of sample

Sampling depth.

A description of the recovered soil sample. The descriptions should include origin, grain size, texture, structure, color, and odor.

Comments or Observations
Sample Identifier
Sample Collection Time

5 Soil Vapor Sampling (indoor, outdoor, and sub-slab vapor)

The purpose of this section is to define the SOP for collecting soil vapor samples at the former Griffiss AFB using electrical drills and soil vapor probes. This SOP describes the equipment, field procedures, and QA/QC procedures implemented for soil vapor sampling.

The sampling methodologies provided below were adapted from the NYSDOH SVI guidance document (NYSDOH, October 2006). Site-specific details and modifications have been implemented through the Sub-Slab Vapor Mitigation Design Work Plan.

Applicable SOPs are listed below:

SOP No. 6, Sample Handling, Documentation, and Tracking

SOP No. 7, Decontamination

5.1 Equipment and Materials List

The following equipment and materials should be on site for soil sampling:

Summa® canisters, minicans, or similar

PID (ppbRAE or similar)

Regulator for vapor sample canister preset to the appropriate sample duration

Vacuum pump (manual or electric)

Stainless steel or PE vapor implants with 'speedfit' push fitting

PE tubing

Box cutter

Tee's for duplicate sample collection

Field logbook

Field Sampling Forms

Digital camera

Waterproof and permanent marking pens

Appropriate health and safety equipment, as specified in the SSHP

Appropriate decontamination supplies, as specified in SOP No. 8

5.2 Locating the Sampling Points

The indoor, outdoor, and sub-slab vapor sample locations will be predetermined in accordance with the site-specific sampling WP.

5.3 Soil Vapor Sampling Procedures

5.3.1 Soil Vapor Sampling

5.3.1.1 Temporary Soil Vapor Probe Installation and Abandonment

The installation and abandonment procedure is as follows:

- A Geoprobe® shall be employed to attain a depth of at least 5 ft below ground surface (bgs) for each soil vapor probe. A 2.5-inch coring machine shall be used to core through the concrete prior to engaging the Geoprobe. If necessary; a hollow-stem auger can be used to attain the desired depth;
- Once the target depth is reached, the rods will be pulled up one foot, exposing the void space, and the sampling apparatus will be set up in the borehole;
- New ¼-inch laboratory grade polyethylene tubing equipped with a threaded stainless steel fitting will be attached to a disposable soil vapor drive point to prevent infiltration of the atmospheric air present at land surface directly above the soil boring (ambient air);
- A clay seal will then be placed at land surface in the annular space between the Geoprobe® rods and the concrete surface, as well as between the tip of the rods and the sample tubing;
- The sampling tubing will be connected to a 'T' connector three-way valve assembly, with one end of the 'T' connector leading to a vacuum pump and the other end leading to a pre-evacuated summa canister with a calibrated regulator;
- The soil vapor sample tubing will then be purged of approximately two volumes of the sample tubing using a vacuum pump set at a rate of approximately 0.2 liters per minute;
- After sampling is completed, the borehole shall be abandoned by being tremie grouted to land surface using a bentonite grout.

5.3.1.2 Soil Vapor Sample Collection

The sampling procedure described below shall be followed at each location to minimize discrepancies between sampling points:

- Prior to formal sample collection, a tracer gas (i.e., helium) shall be used to verify the integrity of the soil vapor probe seal. To do so:
  - ✓ The immediate vicinity of the area where the probe intersects the ground surface shall be exposed to tracer gas using a garbage bag, cardboard box, or plastic pail;
  - ✓ At least one implant volume (i.e., the volume of the sample probe and tube) shall be purged using a flow rate of not more than 0.2 L/min;
  - ✓ Using the same flow rate as the purge (i.e., less than 0.2 L/min), a vapor sample shall be collected from the probe using a Tedlar bag;
  - ✓ The Tedlar bag shall be fitted with a portable monitoring device (i.e., a Gas Check 3000 meter, which measures the rate of the helium leakage at the land surface) and screened for helium. The enriched area (i.e., within the garbage bag/cardboard box/plastic pail) will also be screened for helium.

- ✓ If the concentration of helium is greater than 20% of the helium detected in the enriched area, the seal is not adequate and should be reset. The sample rods will be purged again until the helium is no longer detected at levels greater than 20% of the enriched area located directly above the borehole.

- Once the integrity of the seal has been verified, to ensure samples collected are representative, three implant volumes (i.e., the volume of the sample probe and tube) must be purged prior to collecting the sample;
- Flow rates for both purging and collecting shall not exceed 0.2 L/min to minimize outdoor air filtration during sampling;
- Following the purging, the valve leading to the pump will be closed, the pump will be turned off, and the soil vapor will be directed to a 100% certified 1-L Summa® canister provided by the laboratory. The sample shall be collected using the canister's regulator to restrict the sample collection rate.
- After sample collection, the soil vapor will be screened using a photoionization detector (PID), calibrated daily with a 100 parts per million (ppm) isobutylene standard.

The field sampling team must maintain a sample log sheet summarizing the pertinent sample information, and any relevant observations such as odors and readings from field instrumentation.

5.3.2 Sub-slab Vapor Sampling

5.3.2.1 Temporary Sub-slab Vapor Probe Installation and Construction

As noted in the NYSDOH guidance document, during colder months, heating systems should be operating at least 24 hours prior to and during the scheduled sampling time to maintain normal indoor air temperatures. Prior to installation of the sub-slab vapor probes, the building floor should be inspected and any penetrations (i.e., cracks, floor drains, utility perforations, sumps, etc.) should be noted and recorded. Probes should be installed at locations where the potential for ambient air infiltration via floor penetrations is minimal.

The installation procedure is as follows:

- A rotary hammer drill will be used to create 1-inch diameter holes through concrete and into sub-slab material (e.g., sand or sand and gravel). Drilling into sub-slab material will create an open cavity to prevent obstruction of probes by small pieces of gravel;
- Probes will be constructed from dedicated ¼ inch-diameter laboratory grade polyethylene tubing;
- Tubing shall not extend further than 2 inches into the sub-slab material;
- The implant shall be sealed to the surface with permagum grout, melted beeswax, putty, or other non-VOC-containing and non-shrinking product;
- After sampling is completed, the borehole shall be abandoned in accordance with the procedures described in Section 5.5.3, in the UFP QAPP for Performance Based-Remediation at the Former Griffiss AFB (CAPE/FPM, November 2011).

5.3.2.2 Sub-slab Vapor Sample Collection

The sampling procedure described below shall be followed at each location to minimize discrepancies between sampling points:

- To ensure samples collected are representative, three implant volumes (i.e., the volume of the sample probe and tube) must be purged prior to collecting the sample;
- Flow rates for purging shall not exceed 0.2 L/min to minimize outdoor air filtration during sampling. Purge air shall be collected in a Tedlar bag so it is not released into the building;
- Samples shall be collected over an 24-hour time period, consistent with concurrent indoor and outdoor air samples, if possible;
- Samples shall be collected in 100% certified 6-L Summa® canisters provided by the laboratory.

The field sampling team must maintain a sample log sheet summarizing the pertinent sample information, the uses of VOCs in commercial or industrial processes and/or during building maintenance, weather conditions and ventilation conditions, and any relevant observations such as spills, floor stains, odors and readings from field instrumentation.

In addition, floor plan sketches should be drawn that include the floor layout with sample locations, chemical storage areas, garages, doorways, stairways, location of basement sumps or subsurface drains and utility perforations through building foundations, HVAC system air supply and return registers, compass orientation (north) and any other pertinent information. If possible, photographs should accompany floor plan sketches.

5.3.3 Indoor/Outdoor Air Sampling

5.3.3.1 Pre-sampling Inspection and Documentation

As noted in the NYSDOH guidance document, during colder months, heating systems should be operating at least 24 hours prior to and during the scheduled sampling time to maintain normal indoor air temperatures. Prior to collecting indoor air samples, a pre-sampling inspection should be performed prior to each sampling event to identify conditions that may affect or interfere with the proposed testing. The inspection should evaluate the type of structure, floor layout, physical conditions, and airflows of the building(s) being studied. The inspection information should be identified on the attached Indoor Air Quality Questionnaire and Building Inventory form. In addition, potential sources of chemicals of concern should be evaluated within the building by conducting a product inventory.

In addition, floor plan sketches should be drawn that include the floor layout with sample locations, chemical storage areas, garages, doorways, stairways, location of basement sumps or subsurface drains and utility perforations through building foundations, HVAC system air supply and return registers, compass orientation (north) and any other pertinent information should be documented. If possible, photographs should accompany floor plan sketches.

Finally, outdoor plot sketches should be drawn that include the building site, area streets, outdoor air sample locations, the location of potential interferences (e.g., gasoline stations, factories, other facilities, lawn mowers, etc.), compass orientation (north), footings that create separate foundation sections, and paved areas. Significant activities in the vicinity of the sample locations (e.g., operation of heavy equipment) should be recorded.

5.3.3.2 Indoor/Outdoor Air Sample Collection

Indoor air samples shall be collected in the vicinity of the sub-slab samples from a height above the ground to represent the breathing zone when occupants normally are seated (i.e., 5 ft.). The locations of the outdoor samples shall be chosen from areas away from wind obstructions, and at a height above the ground to represent the breathing zone (i.e., 3 to 5 ft.).

For either indoor or outdoor air samples, the sampling procedure described below shall be followed at each location to minimize discrepancies between sampling points:

- Samples should be collected during normally occupied periods to be representative of typical exposure;
- Sample collection intakes should be located to approximate the breathing zone for building occupants (i.e., 5 feet above the floor level where occupants are normally seated);
- To ensure that an air sample is representative of the conditions being tested and to avoid undue influence from sampling personnel, samples should be collected for a period of twenty-four (24) hours, and personnel should avoid lingering in the immediate area of the sampling device while samples are being collected;
- The sampling team members should avoid actions (e.g., fueling vehicles, using permanent marking pens) that can cause sample interference in the field;
- Flow rates for collecting samples shall not exceed 0.2 L/min to be consistent with concurrent sub-slab sampling;
- Samples shall be collected in 100% certified 6-L Summa® canisters provided by the laboratory; and
- Indoor and outdoor samples should be collected simultaneously;
- Ideally, samples shall be collected over the same period of time as concurrent sub-slab samples.

The field sampling team must maintain a sample log sheet summarizing the pertinent sample information, the uses of VOCs in commercial or industrial processes and/or during building maintenance, weather conditions and ventilation conditions, and any relevant observations such as spills, floor stains, odors and readings from field instrumentation.

5.4 Field Quality Assurance/Quality Control Samples

Field QA/QC samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample collection and handling. All QA/QC samples will be labeled with QA/QC identification numbers and sent to the laboratory with the other samples for analyses.

5.4.1 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. A nylon T-barb will be installed in the PE tubing to allow for sampling of one airstream from one sampling point with two vapor sample canisters simultaneously. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

5.4.2 Matrix Spikes and Matrix Spike Duplicates

MS and MSD analysis are used to assess the potential for matrix effects. The MS/MSD sample will be collected from a randomly selected normal sample by the lab. Following the normal analysis, the lab spikes the normal sample canister with the matrix spike and analyses the air in the canister. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

5.5 Field Documentation

The most important aspect of field documentation is thorough, organized, and accurate record keeping. This includes proper preservation and storage of all field documentation. Field documentation for sub-slab vapor sampling includes field logbooks and field forms. The field forms, described in section 6.5.2, include the sub-slab vapor probe monitoring form, indoor/outdoor air monitoring form, weather observation form, and the NYSDOH Indoor Air Quality Questionnaire and Building Inventory Center for Environmental Health form.

5.5.1 Field Logbook

All information pertinent to sub-slab sampling will be recorded in a bound field logbook with consecutively numbered pages. The field sampling team must maintain a sample log sheet summarizing the pertinent sample information, the uses of VOCs in commercial or industrial processes and/or during building maintenance, weather conditions and ventilation conditions, and any relevant observations such as spills, floor stains, odors and readings from field instrumentation. Refer to SOP No. 7 for detailed procedures regarding documentation in the field logbook.

5.5.2 Field Forms

<u>Sub-slab Probe Monitoring Form</u>

The Sub-slab Probe Monitoring Form contains the following minimum information:

Date

Time

Sample identification

Sample depth

Field personnel

Instruments

Tracer gas identified and concentration

Sample purge volume

Volume of soil vapor extracted

Summa canister: vacuum before sampling and vacuum after sampling

Apparent moisture content

Comments and observations during sampling

Weather conditions, including the outdoor temperature, barometric pressure, precipitation, ventilation conditions, heating system active?, and windows closed

Indoor/Outdoor Air Monitoring Form

The Indoor/Outdoor Air Monitoring Form contains the following minimum information:

Date

Time

Sample identification

Sample height

Field personnel

Instruments

Type of sample

Duration of air sampled

Volume of sample

Summa canister: vacuum before sampling and vacuum after sampling

Comments and observations during sampling

VOCs used during normal operations of facility

Weather conditions, including the outdoor temperature, barometric pressure, precipitation, ventilation conditions, heating system active?, and windows closed

Weather Observation Form

The Weather Observation Form contains the following minimum information:

Location

Date

Field Personnel

Instruments

Time

Conditions collected prior to sampling, mid day, and end of sampling include:

Precipitation

Atmospheric pressure

Temperature

Wind speed

NYSDOH Indoor Air Quality Questionnaire and Building Inventory Center for Environmental Health Form

The NYSDOH Indoor Air Quality Questionnaire and Building Inventory Center for Environmental Health Form contains the following minimum information:

Preparer's name

Date/Time

Preparer's affiliation

Phone number

Field Personnel

Occupant

Name

Address

Phone Number

Number of occupants in building and age

Owner or landlord

Name

Address

Phone Number

Building Characteristics

Type of Building

Property type

Multiple units

Air flow

Basement and Construction Characteristics

Heating, Venting, and Air Conditioning information

Occupancy

Factors that may influence indoor air quality
Water and sewer information
Relocation information
Floor Plans
Outdoor plot
Product inventory form

6 Sample Handling, Documentation, and Tracking

6.1 Purpose and Scope

This SOP describes the procedures for sample handling, documentation, and tracking. This SOP is intended to be used with the UFP-QAPP, FSP and with other SOPs listed below:

SOP No. 1, Soil Sampling

SOP No. 2, Groundwater Sampling

SOP No. 3, Surface water Sampling

SOP No. 4, Surface Soil and Sediment Sampling

SOP No. 5, Soil Vapor Sampling

SOP No. 8, Monitoring Well Installation and Development

6.2 Sample Identification

The sampling locations, sample types, and naming conventions will be established prior to field activities. On-site personnel will obtain assistance in defining any special sampling requirements from the FPM Project Manager or designated Task Manager. Each sample will have a discrete, alpha-numeric sample identification (ID). A unique sample ID is needed to track each sample during the life of this project. In addition, the sample IDs will be used in the database to identify and retrieve the analytical results received from the laboratory. Each sample ID will be assigned at the time of sampling.

Sample ID

The sample ID will be designated as follows: Site Code, Sample Type and Sampling Location Indicator, Sample Depth Identifier, and Sample Type Qualifier.

Site Code

The first segment consists of two to five alphanumeric characters that designate the site code. Site codes for monitoring wells named in previous Griffiss AFB sampling efforts (Law, 1996; FPM, 2001) are listed below:

- LF1 Landfill 1

For the sample designated “LF1M0213AA”, the “LF1” indicates that the site from which the sample was obtained, is the Landfill 1 AOC Site.

Sample Type and Sampling Location Indicator

The second segment consists of one or two alphanumeric characters that indicate the sample type and sampling location indicator. Sample types are as shown below:

- M Groundwater from monitoring well sampling locations
- T Groundwater from direct-push groundwater samples that were not completed as permanent monitoring wells (i.e., temporary well point)
- SW Surface water sample
- SD Sediment sample
- SS Soil Sample
- FS Fish Tissue Sample
- IA Indoor Air
- OA Outdoor Air
- SSV Sub-slab Vapor

The two-digit number following the sample indicator completes the identification of the sampling location at a specific site. For example, for the sample "LF1M0213AA", the "M" indicates that the sample was groundwater taken from a monitoring well, and the "02" indicates that this sample was taken from monitoring well LF1MW-02.

Sample Depth Identifier

The third segment consists of two numerical characters that will be used to identify the depth in feet below TOIC the sample was taken. For the sample designated "LF1M0213AA", the "13" indicates that the sample was obtained at a depth of 13 feet below TOIC.

Sample Type Qualifier

The fourth segment is two alphabetic characters used to designate the type of sample. The first letter denotes the round of sampling completed (e.g., "A" for first quarterly sampling round, "B" for second quarterly sampling round, etc.). The sample types will be identified by the second character as listed below:

- A = Primary sample
- B = Primary sample
- C = Field duplicate groundwater sample
- D = Matrix Spike Duplicate (MSD)
- E = Equipment blank
- F = Ambient blank
- R = Trip blank
- S = Matrix Spike (MS)

The letter A or B appearing at the end of a sample number indicates that the sample is a primary sample. These letters will be selected randomly to mask the predominance of primary samples over QA/QC samples. This system was devised to minimize the likelihood that the laboratory personnel can distinguish the primary samples from the QA/QC samples using the sample identification.

To complete the example, the sample number “LF1M0213AA”, would therefore indicate a primary first-round groundwater sample taken from monitoring well LF1MW-02 at 13 feet below TOIC at the Landfill 1 AOC Site.

6.3 Sample Labels

Sample labels will be completed as much as possible by a designated member of the sampling team prior to beginning field-sampling activities each day. All sample labels will be filled out using waterproof ink. For the pre-designated sampling events (LTM), labels are preprinted by the lab using the COCs developed during sample planning. At a minimum, each label will contain the following information:

Sampler's company affiliation

Site location

Sample ID

Date and time of sample collection

Analyses required

Method of preservation (if any) used

Sample matrix (i.e., soil, groundwater, surface water)

Sampler's signature or initials

6.4 Sample Handling Procedures

This section discusses proper sample containers, preservatives, and handling and shipping procedures. The UFP-QAPP summarizes the information contained in this section and also includes the sample holding times for each analyte.

6.4.1 Sample Containers

Certified, commercially clean sample containers will be obtained from the contract analytical lab. Required preservatives will be prepared and placed in the containers at the laboratory prior to shipment to the site. Appropriate sample containers for the specific analyses required will be listed in the UFP-QAPP (Worksheet #19).

6.4.2 Sample Preservation

Sample preservation efforts will commence at the time of sample collection and will continue until analyses are performed. Samples will be stored on ice at 4°C in coolers immediately following collection. Chemical preservatives, if necessary, will be added to the sample containers by the laboratory prior to shipment to the field, unless otherwise specified in the UFP-QAPP.

6.4.3 Sample Handling and Shipping

The sample containers will be wiped clean of all sample residue and then wrapped in protective packing material (bubble wrap) and taped. Samples will be single-bagged with plastic bags and then placed upright in an iced cooler. A COC form will accompany each cooler.

Coolers will be picked up at the FPM Rome office by the lab courier or shipped by overnight express carrier to the analytical laboratory. All samples must be shipped for laboratory receipt and analyses within specific holding times (UFP QAPP, Worksheet #19). This may require daily shipment of samples with short holding times. The condition of all samples as received and temperature of all coolers will be reported by the laboratory.

6.4.4 Holding Times and Analyses

The holding time is specified as the maximum allowable time between sample collection and analysis and/or extraction, based on the analyte of interest and stability factors, and preservative (if any) used. Allowable holding times are listed in the UFP-QAPP (Worksheet #19).

6.5 Sample Documentation and Tracking

This section describes documentation required in the field notes, on the field sampling forms, on the Daily CQCRs, and on the COCs.

6.5.1 Field Logbook

The purpose of the field log book is to provide a chronological account of all field activities for future reference. Activities logging will be performed to include sufficient information so that the sampling activity can be reconstructed without relying on the memory of field personnel. The logbooks will be kept in the field team member's possession or in a secure place during the investigation. Following the investigation, the logbooks will become a part of the final project file.

All entries in logbooks will be made in waterproof ink and corrections will consist of line-out deletions that are initialed and dated. The following information (as applicable) shall be recorded in the header of the field log book:

Sampler’s printed name and signature

Names of other field personnel (CAPE Team and any CAPE Team subcontractors) and site visitors

Date (month, day, year)

General weather conditions

The following information (as applicable) shall be recorded in the field log book:

Results of equipment calibration

Time and location of sampling (including approximate distance to adjacent landmarks if possible)

Documentation of field measurement results such as total depths and depth to groundwater in monitoring wells.

Sample Identification and time of collection

Any QA/QC sample collected

Decontamination information

Brief discussion of any field decisions, unusual conditions, problems encountered and corrective action taken, and/or changes required by field conditions

Signature and date by person responsible for writing the field notes

In addition to field books, sample forms will also be prepared in the field. The sampling forms will contain the results of any field measurements, sample identification and sampling time. The field measurements included in the sampling form include water chemistry readings. A description of the sampling field forms are included in the sampling matrix specific sections.

6.5.2 Daily Chemical Quality Control Report

Daily CQCRs will be prepared to supplement the information recorded in the field logbook. Daily CQCRs will be prepared by members of the field sampling team and cross-checked for completeness at the end of each day by the sampling team leader and/or Field Manager. They will be signed and dated by individuals making entries. Daily CQCRs will be forwarded to the Quality Assurance Officer for review and approval. The Daily CQCRs will include the following information:

Project name

Project number

Personnel on site

Visitor on site

Subcontractors on site

Weather conditions

Field work performed

Quality control and health and safety activities

Name and title of person completing the Daily CQCR

6.5.3 Chain of Custody

During field sampling activities, traceability of the sample must be maintained from the time that the samples are collected until laboratory data are issued. Information concerning samples collection will be recorded in the field logbook as described above. Information on the custody, transfer, handling, and shipping of samples will be recorded on a COC form.

The sampler will be responsible for initialing and completing the COC. The sampler will sign the COC when the sampler relinquishes the samples to the lab courier. One COC will be completed daily for the site's samples. The COC will contain the following information:

Sampler's signature and affiliation

Project name

Date and time of collection

Sample ID

Sample type

Analyses requested

Number of containers per sample per analysis

Signature of persons relinquishing custody, dates, and times

Signature of persons accepting custody, dates, and times

Method of shipment

Shipping air bill number (if applicable)

The person responsible for sample shipment to the laboratory will sign the COC form, and retain a copy of the form, document the method of shipment, and send the original copy of the COC form with the samples. Copies of the COC forms documenting custody changes and all custody documentation will be received in the lab packages and kept in the central files. The original COCs will remain with the samples until final disposition of the samples by the laboratory. The analytical laboratory will dispose of the samples in an appropriate manner 60 to 90 days after data reporting.

7 Decontamination

7.1 Purpose and Scope

This SOP describes the equipment, materials, field procedures, and documentation procedures for decontaminating sampling equipment and personnel. The procedures presented below are intended to be used with other SOPs listed below:

SOP No. 1, Soil Sampling

SOP No. 2, Groundwater Sampling

SOP No. 3, Surface water Sampling

SOP No. 4, Surface Soil and Sediment Sampling

SOP No. 5, Soil Vapor Sampling

SOP No. 8, Monitoring Well Installation and Development

The overall objective of an environmental sampling program is to obtain samples that accurately represent the chemical, physical, and/or biological conditions at the sampling site. Extraneous contaminants can be brought onto the sampling location and/or introduced into the medium of interest during the sampling program (e.g. using sampling equipment that is not properly or fully decontaminated). Trace quantities of contaminants can consequently be captured in a sample and lead to false positive analytical results and, ultimately, to an incorrect assessment of the contaminant conditions associated with the site. Decontamination of sampling equipment (e.g., all non-disposable equipment that will come in direct contact with samples) and field support equipment (e.g., drill rigs, vehicles) is, therefore, required prior to, between, and after uses to ensure that sampling cross-contamination is prevented, and that on-site contaminants are not carried off-site.

7.2 Equipment and Materials List

The following is a list of equipment that may be needed to perform decontamination:

Brushes

Wash tubs

Buckets

Scrapers, flat bladed

Hot water – high-pressure sprayer

Sponges or paper towels

Liquinox® detergent (or equivalent)

Potable tap water

Laboratory-grade de-ionized water

Garden-type water sprayers

Appropriate Health and Safety equipment (i.e., nitrile gloves, safety glasses, etc.)

Appropriate containers for Investigation Derived Waste (IDW).

7.3 Decontamination Procedures

Site activities should be conducted with the general goal of preventing the contamination of personnel and equipment. CAPE Team sampling personnel will bag monitoring instruments, avoid contact with obvious contamination, and employ dust suppression methods as necessary to reduce the probability of becoming contaminated and, therefore, reduce the need and extent of decontamination. However, some type of decontamination will always be required on site.

7.3.1 Decontamination Solutions

A decontamination solution should be capable of removing, or converting to a harmless substance, the chemical of concern without harming the object being decontaminated. The preferred solution is a mixture of detergent and water, which is a relatively safe option compared to chemical decontaminants. A solution recommended for decontaminating consists of 1 to 1.5 tablespoons of Liquinox® per gallon of warm water. Skin should be decontaminated by washing with hand soap and water. The decontamination solution must be changed when it no longer foams or when it becomes dirty. Rinse water must be changed when it becomes discolored, begins to foam, or when the decontamination solution cannot be removed.

7.3.2 Personnel Decontamination

A sample personnel decontamination set-up guideline and equipment and supplies list are included in the SSHP.

7.3.3 Sampling Equipment Decontamination

The following steps will be used to decontaminate sampling equipment:

Personnel will dress in suitable safety equipment to reduce personal exposure as required by the SSHP. Typically for LTM programs, this includes personnel in level D PPE (long pants, long sleeve shirts, steel toe boots, and nitrile gloves).

Gross contamination on equipment will be scraped off at the sampling or construction site with a flat bladed scrape.

Equipment that cannot be damaged by water will be placed in a 5-gallon bucket containing a Liquinox® solution or low-sudsing non-phosphate detergent along with potable water and scrubbed with a bristle brush or similar utensil. Equipment will be rinsed with tap water in a second wash tub followed by a de-ionized water rinse.

Equipment that may be damaged by immersion in water will be carefully wiped clean using a sponge and detergent water and rinsed with de-ionized water. Care will be taken to prevent equipment damage.

Following decontamination, equipment will be placed in a clean area or on clean plastic sheeting to prevent contact with contaminated soil. If the equipment is not used immediately after decontamination, the equipment will be covered or wrapped in plastic sheeting, foil, or heavy-duty trash bags to minimize potential contact with contaminants.

7.3.4 Direct Push Equipment Decontamination

Direct push rigs will be decontaminated at a decontamination station located near the staging area. Direct push rods will be decontaminated at the various drilling locations. The following steps will be used to decontaminate direct push equipment:

The direct push rig will be decontaminated upon mobilization to the site and demobilization from the site. The direct push rods will be decontaminated between each boring location.

Personnel will dress in suitable PPE to reduce personal exposure as required by the SSHP.

Equipment showing gross contamination or having caked-on soil cuttings will be scraped with a flat-bladed scraper at the sampling or construction site.

The direct push rods will be washed with a hot water, high-pressure sprayer then rinsed with potable water. OSHA requires that proper PPE must be worn when operating pressure-washing equipment. A rain suit, boots, hard hat, and a face shield are recommended to be worn. All personnel must be kept out of the path of steam or water spray.

Following decontamination, direct push rods will be placed on a clean area. If the direct push rods are not used immediately, they must be stored in a designated clean area.

7.3.5 Equipment Leaving the Site

Vehicles used for activities in non-contaminated areas shall be cleaned on an as-needed basis, as determined by the Site Safety and Health Officer (SSHO), using soap and water on the outside and vacuuming the inside. On-site cleaning will be required for very dirty vehicles leaving the area or equipment that has been operated in contaminated areas. Drilling and trailers used in contaminated areas will be pressure washed before the equipment is removed from the site to limit exposure of off-site personnel to potential contaminants.

7.3.6 Responsible Authority

Decontamination operations at each hazardous waste site shall be supervised by the SSHO. The SSHO is responsible for ensuring that all personnel follow decontamination procedures and that all contaminated equipment is adequately decontaminated. The SSHO is also responsible for maintaining the decontamination zone and managing the wastes generated from the decontamination process.

7.3.7 Investigation Derived Waste

Liquid wastewater from decontamination will be drummed and properly disposed of. Solid waste, including sample liners and PPE, will be bagged and removed from the site as household waste.

7.4 Emergency Decontamination

Emergency decontamination procedures should be followed if necessary to prevent the loss of life or severe injury. In the case of threat to life, decontamination should be delayed until the victim is stabilized; however, decontamination should always be performed first, when practical, if it can be done without interfering with essential lifesaving techniques or first aid, or if a worker has been contaminated with an extremely toxic or corrosive material that could cause severe injury or loss of life. During an emergency, provisions must also be made for protecting medical personnel and disposing of contaminated clothing or equipment.

7.5 Documentation

Sampling personnel will be responsible for documenting the decontamination of sampling and drilling equipment. The documentation will be recorded with waterproof ink in the sampler's field notebook with consecutively numbered pages. The information entered in the field book concerning decontamination will include the following:

Decontamination personnel

Date and start and end times

Decontamination observations

Weather conditions

IDW handling

8 Monitoring Well Installation and Development

8.1 Purpose and Scope

This section described the SOP for drilling, installation, and development of monitoring wells at the former Griffiss AFB. The step-by-step procedures described herein are sufficiently detailed to allow field personnel to properly install and develop wells. All construction materials methods and details will be consistent with the requirements of the New York State Department of Environmental Conservation (NYSDEC) for well installation.

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This SOP is intended to be used with other SOPs listed below:

SOP No. 1, Soil Sampling

SOP No. 2, Groundwater Sampling

SOP No. 5, Soil Vapor Sampling

SOP No. 6, Sample Handling, Documentation, and Tracking

Health and safety procedures and equipment for the investigation are detailed in the SSHP.

8.2 Drilling and Well Installation Procedures

8.2.1 Equipment and Materials List

This section details the required equipment, drilling and installation procedures, and documentation procedures for installation of groundwater monitoring wells and vertical biosparging points at the former Griffiss AFB.

The following is an equipment list for monitoring well installation:

Hollow-stem auger rig capable of installing wells to the desired depth in the expected formation materials and conditions

Weighted tape measure

Water level probe

PID (with 10.2 eV lamp)

Well casing and well screen

Bentonite pellets

Filter pack sand (16-40 silica sand)

Portland Type I or II Cement and powdered bentonite or high solids bentonite for grouting

High pressure grout pump

Protective well casing with locking cap or flush mount manhole assembly with padlock

Steel guard posts (bollards) for stick-up wells

Inertial Pump (Waterra® pump or similar)

High-pressure steamer/cleanser

Long-handled bristle brushes

Wash/rinse tubs or pails

Liquinox® detergent

Plastic bags (Ziploc®)

Self-adhesive labels

Deionized water

Appropriate health and safety equipment

Log book

Boring log sheets

Well construction logs

Appropriate sample containers

Sample cooler and ice

8.2.2 Drilling Method

An auger section is a section of pipe with flanges welded onto it. Each auger section is referred to as a flight. Flights are typically five feet in length. The flights are linked together as each flight is advanced to the ground surface. Sampling tools and the center bit are advanced through the open pipe. The cutting bit has a finger plug which prevents loose soil from entering the open pipe. A split-spoon sampling device may be lowered inside the drill string and driven through the finger plug and ahead of the cutting bit for an in-situ sample as required. The drill string, therefore, serves as a form of casing because it does not have to be withdrawn each time a sample is collected. For the 2-inch-diameter wells that are planned, a 4-1/4 inch inside diameter HSA will be used.

There are several advantages of HSA boreholes. First, the method is rapid in most unconsolidated, fine- to medium-grained geologic materials. Second, drilling fluids are not used to remove cuttings and, therefore, the in-situ chemical conditions of the borehole are not further degraded by either diluting contaminants with added water or contributing suspended solids from drilling mud used to stabilize the borehole walls in soft materials. Third, HSA flights are easily cleaned and decontaminated. Fourth, the auger flights serve as a form of casing, which allows monitoring wells to be constructed by raising the flights as needed.

If flowing sands are encountered, potable water may be added to the augers to equalize the hydrostatic pressure in the boring. If water is added to the augers or borehole, it must be potable and the quantity used recorded in the field logbook.

8.2.3 Stratigraphic Logging

Borehole stratigraphy will be logged by examining continuous core soil samples or soil cuttings. The data will be recorded on the boring log and will include the following information:

Project name and number

Drilling company name

Date drilling started and finished

Type of bit and size

Casing sizes and depths

Well completion details

Driller's name

Geologist's name

Type of drill rig

Boring number

Surface elevation (if available)

Sample depths and times

Sample characteristics with depth, such as lithology, grain size, sorting, texture, structure, bedding, color, moisture content, and the Unified Soil Classification (if in unconsolidated geologic materials)

Water levels

Geophysical or video log run (if any)

Drilling observations

Other pertinent information

8.2.4 Well Material Specifications

This section describes the well materials to be used for groundwater monitoring well installations.

8.2.4.1 Well Casings

Well casing will consist of new, threaded, flush-joint, 2-inch ID, schedule 40 PVC. O-rings will be used at all joints. Heat-welded joints and/or gaskets will not be used. The tops of all well casings will be fitted with caps (J-plug) which can be easily removed by hand. The well casing will be brought to the site in its factory post-cleaning plastic wrapping and steam cleaned before installation will not be required.

8.2.4.2 Well Screens

Well screens will consist of new, 2-inch ID threaded PVC with factory machined slots. The screen slot sizes are 0.010-inches and 0.020-inches. An end-plug will be placed at the bottom of the screen. The screen depth will intersect the uppermost portion or the saturated zone. All well screens will have an inside diameter equal to or greater than that of the well casing. Well screen length will be 10 feet.

8.2.4.3 Filter Pack

The filter pack material for the monitoring wells will consist of a #16-40 pre-washed environmental grade silica sand or equivalent. For shallow wells, less than 30 ft bgs, the filter pack will be poured into the open boring. For deeper wells, the filter pack will be placed by tremie pipe from the bottom of the borehole to two feet above the top of the screen interval. Surging of the well may be necessary during filter pack placement to obtain an adequate pack placement around the well screen. The depth of the filter pack will be continuously probed with a weighted tape during placement to monitor pack placement and avoid bridging.

8.2.4.4 Bentonite Seal and Annular Seal

A bentonite seal will be installed above the filter pack in the monitoring wells. The seal will consist of a two-foot interval of bentonite chips or pellets placed by gravity feed from the ground surface and will be hydrated prior to placement of the annular seal. The annular seal will be placed by gravity feed from just above the bentonite seal to within three feet of the ground surface and shall consist of cement grout, neat cement, concrete, or bentonite grout.

8.2.4.5 Well Completion

Two well completion types will be used. These include the flush mount and stick-up well completions.

For high traffic areas, flush mount completions will be installed. The flush mount includes a 8-inch OD traffic rated manhole and concrete pad. Following manhole installation, a locking water-tight security plug will be installed on top of the PVC riser. At a minimum the monitoring well identification number and installation date will be stamped or engraved on to the tag.

For areas with no traffic, stick-up completions will be installed. The stick-up completions include a steel 8-inch OD stick-up pipe, traffic bollards, and concrete pad. Following stick-up completion installation, a locking water-tight security plug will be installed on top of the PVC riser. At a minimum the monitoring well identification number and installation date will be stamped or engraved on to the tag.

8.2.5 Well Installation Procedure

The procedure for monitoring well installation using HSA methods is as follows:

1. Decontaminate all well materials (if necessary) according to SOP No. 8. Following decontamination, all personnel that handle the casing will don a clean pair of rubber or nitrile gloves.
2. Measure each joint of casing and screen to nearest 0.10 foot.
3. Assemble screen and casing as it is lowered into the boring or inside the HAS pipe. Attach stainless steel centralizers if required.
4. Lower screen and casing to the bottom of the boring.
5. Record level of top of casing and calculate screened interval. Adjust screen interval by raising or lowering assembly to desired interval if necessary and add sand to raise the bottom of the boring to the base of the screen. A 1.5-inch diameter, 10-foot long pipe may be lowered into the well to check for straightness. If the pipe will not pass the entire length of the well casing, the well will have to be removed and reset or, if this is not possible, a new well will be installed.
6. Begin adding filter pack sand around the annulus of the casing by slowly gravity feeding the sand (through the tremie pipe if required). Repeated depth soundings should be taken to monitor the level of the sand.
7. Allow sufficient time for the filter sand to settle through the water column outside the screen and casing before measuring the sand level.
8. Extend the filter pack sand to two feet above the top of the well screen. Surging of the well may be required to obtain a good pack around the well screen.
9. Following sand filter pack placement, install a minimum 2-foot thick bentonite seal by slowly adding the pellets to avoid bridging. The bentonite will be hydrated with potable water if the seal is above the water table.
10. Grout the remaining annulus from the top of the bentonite seal to the ground surface using bentonite grout or similar. The grout will be placed into the borehole until the annulus is filled to within three feet of the ground surface.
11. Record the volume of the filter pack, bentonite seal, and grout used.
12. After the grout sets for 24 hours the well completion (flush mount or stick-up) enclosure will be installed. The enclosure will consist of a traffic-rated manhole. Completions will be flush with the surrounding surface. Completions will have a concrete pad sloped slightly away from the manhole. Manholes will have covers secured by bolts.

8.2.6 Surveys

The locations and elevations of any new monitoring wells will be surveyed by a surveyor licensed in the State of New York. At a minimum, the horizontal location of the well will be surveyed to the nearest one foot, the elevation of the ground surface next to the protective casing will be surveyed to the nearest 0.10-foot, and the elevation of the measuring point on the well riser will be surveyed to the nearest 0.01-foot.

8.2.7 Documentation

Observations and data acquired in the field during drilling and installation of monitoring wells will be recorded to provide a permanent record. These observations will be recorded with waterproof ink in a bound weatherproof field logbook and drilling log with consecutively numbered pages. Notes will be recorded daily when in the field. The drilling log is described in detail in SOP No. 1. The information in the field book will include the following as a minimum:

Field Logbook

Project name and number

Observer's name

Visitors and contractors on site

Drilling and well installation observations as described in Section 9.2

Decontamination observations as described in SOP No. 8

Weather conditions

The well installation details will be shown in a diagram which will be drawn in the field book.

Each well diagram will consist of the following (denoted in order of decreasing depth from ground surface):

Bottom of the boring

Casing depth (if intermediate casing is left in the hole)

Screen location(s)

Filter pack intervals

Bentonite seal intervals

Cave-in locations

Height of riser without cap (above ground surface)

Protective casing detail

Additional documentation for well construction in the field book will include the following:

Grout, sand, and bentonite volume calculations prior to well installation

The quantity and composition of the grout, seals, and filter pack actually used during construction

Screen slot size (in inches), slot configuration, outside diameter, nominal inside diameter, schedule/thickness, composition, and manufacturer

Coupling/joint design and composition

Protective casing composition and nominal inside diameter

Start and completion dates

Discussion of all procedures and any problems encountered during drilling and well construction.

8.3 Well Development

The purpose of well development is to remove well drilling fluids, solids, or other particulates which may have been introduced or deposited on the boring wall in a recently installed well during drilling and construction activities. This restores the hydraulic conductivity of the aquifer material surrounding the well to near pre-well installation conditions. Properly developed monitoring wells allow for the collection of groundwater samples that are chemically and physically representative of the aquifer. The procedure is also applicable to older or improperly developed wells which are suspected of not providing representative groundwater samples. This section describes the equipment, methods, and documentation that will be used for developing groundwater monitoring wells.

8.3.1 Equipment and Materials List

The following items are required to properly develop groundwater monitoring wells:

Well keys

Electronic water level indicator (oil/water interface probe for fuel sites)

Calculator

Field notebook

Waterproof pen

Electric inertial pump

Electric submersible pump and controller of appropriate size for the well diameter

Portable electric generator for submersible pumps

Disposable PE bailer (sized appropriately for well)

Nylon or polypropylene rope or wireline (for deep wells) for bailing

Multi-parameter water quality system with a flow-through cell for real-time groundwater parameter monitoring (temperature, pH, specific conductance, DO and ORP), with appropriate calibration solutions

Nephelometric turbidity meter

Polyethylene or glass container (for field parameter measurements)

Plastic spray bottle filled with deionized water

Drums or other large container for development water

Appropriate health and safety equipment

Liquinox® solution

Potable water for decontamination

Distilled or deionized water

Decontamination buckets/pails

Plastic brushes

Well completion information

Well development log

8.3.2 Procedure

The development of a newly installed monitoring well will proceed only after the cement/bentonite grout has been allowed to set for a minimum of 24 hours if such grout was used for constructing a well, and after the completed well has been allowed to equilibrate for at least 48 hours. Monitoring well development activities will be completed prior to purging and groundwater sampling for analytical testing. Before development begins, the development equipment will be decontaminated according to the procedures described in SOP No. 8. Equipment coming in contact with the well will also be decontaminated between wells.

Before development begins, the field personnel will verify that the multi-parameter water quality system, and water level probe are operating properly. The electronic water quality instruments require daily calibration or calibration checks prior to use. Calibration times and readings will be recorded in the field notebook and on calibration forms (SOP No. 11). Specific instructions for calibrating the various water quality instruments are provided in instrument-specific instruction manuals and in SOP No. 11.

Monitoring well development at the former Griffiss AFB will be accomplished by using a bailer, a submersible pump, or an inertial pump to flush the screen, sand pack material, and borehole wall of fine sediment resulting from well drilling and installation activities. This procedure also allows for the removal of fine sediment which may have accumulated within the inner well casing.

Development consists of removing a minimum of five well casing volumes of water during repeated surging and well evacuation episodes. Well surging is the process of causing water to move through the screen and into and out of the sand pack and aquifer formation. This will be accomplished by surging the entire length of well screen using bailer or pump. Surging may also be used during well construction to compact the sand filter pack around the well screen.

Well evacuation is the process of removing water from throughout the entire water column by periodically lowering and raising the pump intake or the point to which the bailer is lowered. Development water will be collected in drums or holding tanks for characterization. The volume of water required for removal during development is calculated using the following method:

1. Measure the depth to water in the well from the measuring point. This is usually the top of the well riser cap which has previously been surveyed.
2. Measure the total depth of the well from the same measuring point used for measuring the depth to water.
3. Calculate the height of water in the well casing by subtracting the depth of water from the total well depth.

4. Calculate the number of gallons of water corresponding to one well volume. This is done by multiplying the height of water column in the well casing by the conversion factor corresponding to the inside diameter of the well casing. The following equation shall be used to calculate the volume of water to be removed during well evacuation:

   For 2-inch well: Well Volume = (Total Well Depth – Water Level Depth X 0.17 gal/ft = gallons/1 well casing volume

   Multiply the volume of one well casing volume by five to obtain the minimum volume of water to be evacuated.

During the well development activities field measurements of temperature, pH, nephelometric turbidity, specific conductance, and dissolved oxygen are made, and the clarity, color, any presence of odors, and other comments regarding water quality are noted in the field notebook and on the well development log. The date, time, and volume of water removed are also recorded at this time. All measurements will be recorded for each well volume of water removed. A sample of water will be collected for measurement of the field water quality parameters at the beginning of well development in order to establish a baseline for comparison with the water quality as well development proceeds.

Initial monitoring well development activities with the bailer or pump will continue until at least five well casing volumes have been removed and measurements of the field parameters have stabilized within 10 percent or 0.1 units and the water removed from the well is as clear of sediment as is practical. Regardless of the clarity of the water removed, a minimum of five well volumes of water will be removed during the bailing/surging phase of well development. If the well is bailed dry, it will be allowed to recover. After initial development activities with the bailer are completed, the well will be further developed by purging after installing the submersible pump and lift pipe. Purging will continue with the submersible pump until the field water quality parameters are within 10 percent or 0.1 units for three consecutive readings.

8.3.3 Documentation

Documentation of observations and data acquired in the field will provide information on well development and also provide a permanent record. These observations and data will be recorded with waterproof ink in a bound weatherproof field book with consecutively numbered pages and on the well development form.

As part of the development process, the following information will be recorded in the field book:

Well designation

Well location

Field personnel

Date(s) and time of well development

Static water level from top of well casing before and after development

Volume of water in well prior to development

Volume of water removed and time of removal

Depth from top of well casing to bottom of well

Screen length

Depth from top of well casing to top of sediment inside well, if present, before and after development

Field measurements of pH, conductivity, turbidity, dissolved oxygen, and temperature taken during and after development

Physical character of removed water throughout development (color, odor, and turbidity)

Type and size/capacity of pump and/or bailer

Description of development technique

Decontamination observations

Instrument calibration record

9 Boring and Monitoring Well Abandonment

9.1 Purpose and Scope

This document defines the SOP for abandoning borings and gives descriptions of equipment and field procedures necessary to abandon borings and monitoring wells at the former Griffiss AFB. This SOP is intended to be used with the UFP QAPP and with other SOPs listed below:

- SOP No. 1: Soil Sampling
- SOP No. 2: Groundwater Sampling
- SOP No. 8: Monitoring Well Installation and Development

9.2 Boring Abandonment Procedures

9.2.1 Equipment and Materials List

The following is an equipment and materials list for boring abandonment:

High solids bentonite grout or granular bentonite

Potable water

Logbook

Boring log sheets

Waterproof and permanent marking pens

Appropriate health and safety equipment

9.2.2 Abandonment Procedures

Borings:
Following completion of the soil borings, each boring must be abandoned and plugged to provide a low-permeability zone that would retard movement of water through the boring backfill. Where water was not encountered and the boring sidewalls are stable the boring may be backfilled using granular bentonite. The dry granular bentonite is poured into the boring from the ground surface filling the boring in 1-foot lifts. Hydration of the bentonite with 1 gallon of water is necessary for each lift.

Monitoring Wells:
All abandonment of monitoring wells, shall be performed in accordance with 6 NYCRR Part 360-2.11 (a)(8)(vi) and the 1996 version Ground-Water Monitoring Well Decommissioning Procedures, Sections 2.2, 9.0, and 10.0. NYSDEC approved abandonment methods into grout and pull, grout in place or over drilling. These are described below:

Grout and Pull
Well casing is pulled out of the ground using a drill rig and a slurry is applied to bore hole.

Grout in place
Well casing remains in the ground; however, a slurry is applied to the well to close all potential pathways.

Over Drilling
Well casing is over drilled by a drilling company. An auger is advanced to the bottom of the well and a slurry is applied to bore hole.

When slurry is used, a mud balance and/or Marsh Funnel shall be used to ensure that the density (lbs/gal) of the abandonment mud mixture conforms to the manufacturer's specification. All abandoned monitoring wells shall be checked 24 to 48 hours after mud/solid bentonite emplacement to determine whether curing is occurring properly. More specific curing specifications or quality assurance checks may be recommended by the manufacturer and shall be followed. Additionally, if significant settling has occurred, a sufficient amount of mud/solid bentonite shall be added to attain its initial level. These slurry/solid bentonite curing checks and any addition of mud/solid bentonite shall be recorded in the field logs.

9.2.3 Pavement Repair

Where borings or monitoring wells penetrate concrete or asphalt pads, it will be necessary to patch the pavement surface following backfilling. Concrete pavements should be filled with low slump (less than 4 inches) concrete mix. Asphaltic or concrete pavements should be filled with asphaltic concrete patch mix and thoroughly compacted by ramming. The surface of any patch should be level upon completion. In freezing weather the concrete mix must be protected from freezing for 48 hours after placement.

9.3 Documentation

Observations and data acquired in the field during boring abandonment will be recorded to provide a permanent record. These observations will be recorded with waterproof black ink in a bound weatherproof field book with consecutively numbered pages. A note shall be placed on the boring log for the boring that was abandoned and backfilled that identifies the date and method of abandonment. The type of material used to patch a pavement surface (if done) will also be noted on the boring log and the field book.

10 Equipment Calibration

10.1 Purpose and Scope

This SOP describes the procedures for equipment calibration and documentation. This SOP is intended to be used with the UFP-QAPP, FSP and with other SOPs listed below:

SOP No. 1, Soil Sampling

SOP No. 2, Groundwater Sampling

SOP No. 3, Surface water Sampling

SOP No. 8, Monitoring Well Installation and Development

10.2 Equipment and Materials List

The following section provide a list of equipment that may be needed to perform equipment calibration.

Horiba U-22 and Horiba U-52:

Horiba U-22

Horiba U-52

Auto calibration solution pH 4

Calibration cup

Calibration log for Horibas

YSI 556

YSI 556

Calibration cup

Calibration log for YSI

DI water

Conductivity solution (1.413 µS/cm)

pH 4 solution

pH 7 solution

ORP solution (240 mV)

PID, miniRAE

PID, miniRAE

Tedlar bag

Isobutylene (100 ppm)

Calibration log for PID

10.3 Equipment Calibration Procedures

The following provides the procedures for the calibration of the Horiba U-22 and U-52, YSI 556, and PID miniRAE.

Horiba U-22:

- Turn on Horiba.
- Place probe in auto calibration solution (pH 4.00).
- Press Cal button.
- Press Ent button, calibration begins.
- END appears when calibration is complete.
- Press MEAS button and collect pH reading.
- The acceptable pH range is 3.96 to 4.04.
- If any errors appear, refer to Horiba U-22 manual.

Horiba U-52:

- Turn on Horiba.
- Place probe in auto calibration solution (pH 4.00).
- Press Cal button.
- Press Ent button, calibration begins when the parameters on screen start to blink.
- When parameters stop blinking, calibration is complete.
- Collect pH reading.
- The acceptable pH range is 3.96 to 4.04.
- If any errors appear, refer to Horiba U-52 manual.

YSI 556:

- Turn on YSI 556.
- Press ESC which will lead to main menu.
- Scroll to Calibrate and press ENT.
- Scroll to DO, press enter, scroll to DO%
- Enter barometric pressure.
- Place probe in DI water (in calibration cup) and loosely tighten probe to calibration cup.
- Press enter, and then enter again.
- DO% is instantly calibrated.
- Acceptable range is 95% to 105%.
- Press ESC to return to calibration menu.
- Scroll to Conductivity, press enter, scroll to Conductivity in list and press enter
- Enter standard, 1.413 µs/cm.

- Fill calibration cup with conductivity solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.
- Conductivity is instantly calibrated.
- Acceptable range is 1.408 to 1.418 µs/cm.
- Press ESC to return to calibration menu.
- Scroll to pH, press enter, scroll to 2-point calibration and press enter
- Enter 1st standard, 4.00.
- Fill calibration cup with pH 4.00 solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.
- pH is instantly calibrated.
- Acceptable range is 3.95 to 4.05.
- Press enter.
- Enter 2nd standard, 7.00.
- Fill calibration cup with pH 7.00 solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.
- pH is instantly calibrated.
- Acceptable range is 6.95 to 7.05.
- Press ESC to return to calibration menu.
- Scroll to ORP, press enter
- Enter standard, 240 mV.
- Fill calibration cup with ORP solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.
- Conductivity is instantly calibrated.
- Acceptable range is 235 to 245 mV.
- If any errors appear, refer to YSI 556 manual.

PID miniRAE:

Zero Calibration

Turn on PID to Zero Calibration menu.

Press [Y/+] to start calibration.

Press [MODE] to quit and return to the main calibration display.

Zero calibration starts.

When Zero calibration is complete, you see this message: Zeroing is done!, Reading = 0.000 ppm.

Span Calibration

Turn on PID to Scan Calibration menu.

The span gas is first be filled into a Tedlar bag.

Connect the calibration adapter to the inlet port of the instrument, and connect the tubing to the regulator or Tedlar bag.

Press [Y/+] to enter Span calibration.

Turn on your span calibration gas.

Press [Y/+] to initiate calibration.

Span calibration starts and displays this message: Calibrating...

When Span calibration is complete, you see this message: Span 1 is done!, Reading = 100.0 ppm

Per the Mini RAE manual, there is no set range of what is allowed above or below 100 ppm. The Manual simply states that the “reading should be very close to the span gas value”.

10.4 Documentation:

Documentation for equipment calibration forms which are included in Daily CQCRs. The calibration forms include:

Equipment model and number

Date

Calibration personnel

Standard calibration values

Scan gas concentration for PID calibration

Standard calibration solution parameters for water quality

Attachment 1
Field Forms

HTW DRILLING LOG

1. COMPANY NAME	2. DRILLING CONTRACTOR	HOLE NO. SHEET 1 OF 2 SHEETS
3. PROJECT	4. LOCATION	
5. NAME OF DRILLER	6. MANUFACTURER'S DESIGNATION OF DRILL	
7. SIZES & TYPES OF DRILLING & SAMPLING EQUIPMENT	8. HOLE LOCATION	
	9. SURFACE ELEVATION	
	10. DATE STARTED / 11. DATE COMPLETED	
12. OVERBURDEN THICKNESS	15. DEPTH GROUNDWATER ENCOUNTERED	
13. DEPTH DRILLED INTO ROCK	16. DEPTH TO WATER AND ELAPSED TIME AFTER DRILLING COMPLETED	
14. TOTAL DEPTH OF HOLE	17. OTHER WATER LEVEL MEASUREMENTS (SPECIFY)	

18. GEOTECHNICAL SAMPLES	DISTURBED	UNDISTURBED	19. TOTAL NUMBER OF CORE BOXES				
20. SAMPLES FOR CHEMICAL ANALYSIS	VOC	METALS	OTHER (SPECIFY)	OTHER (SPECIFY)	OTHER (SPECIFY)	21. TOTAL CORE REC %	
22. DISPOSITION OF HOLE	BACKFILLED	MONITORING WELL	OTHER (SPECIFY)	23. SIGNATURE OF INSPECTOR			

ELEV. a	DEPTH b	DESCRIPTION OF MATERIALS c	Field Screening Results d	Geotech Sample or Core Box No. e	Analytical Sample No. f	Blow Counts g	REMARKS h

HTW DRILLING LOG

HOLE NO.

PROJECT

INSPECTOR

SHEET 2
OF 2 SHEETS

ELEV. a	DEPTH b	DESCRIPTION OF MATERIALS c	Field Screening Results d	Geotech Sample or Core Box No. e	Analytical Sample No. f	Blow Counts g	REMARKS h

PROJECT

HOLE NO.

WELL PURGING & SAMPLING FORM

Project: _____ Sampled by: _____

Location and Site Code (**SITEID**): _____

Well No. (**LOCID**): _____ Well Diameter (**SDIAM**): _____

Date (**LOGDATE**): _____ Weather: _____

CASING VOLUME INFORMATION:

Casing ID (inch)	1.0	1.5	2.0	2.2	3.0	4.0	4.3	5.0	6.0	7.0	
Unit Casing Volume (A) (gal/ft)	0.04	0.09	0.16	0.2	0.37	0.65	0.75	1.0	1.5	2.0	2.6

PURGING INFORMATION:

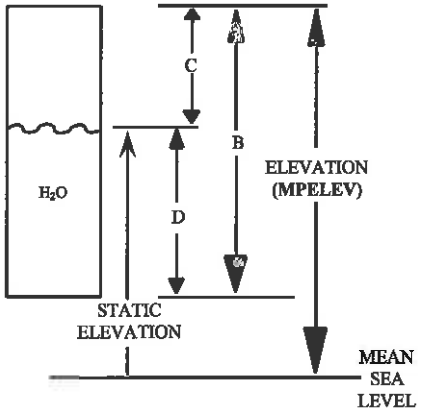
Measured Well Depth (B) (**TOTDEPTH**) _____ ft.

Measured Water Level Depth (C) (**STATDEP**) _____ ft.

Length of Static Water Column (D) = _____ (B) - _____ (C) = _____ (D) ft.

Casing Water Volume (E) = _____ (A) x _____ (D) = _____ gal

Minimum Purge Volume = _____ gal (3 well volumes)



Purge Date and Method: _____

Physical Appearance/Comments: _____

FIELD MEASUREMENTS:

Allowable Range: ± 0.1 ± 5% ±1°C

Time	Volume Removed (gal)	pH	EC (mS/cm)	Temp. (F or C)	Turbidity (NTU)	D.O. (mg/L)	ORP (mV)

Sample Time: _____ Sample ID: _____

Note: Attempt to get at least 5 sets of field measurements during purging. Sample may be collected after 3 to 5 well volumes have been removed and parameters have stabilized. Sample may be collected after 6 well volumes if parameters do not stabilize. VOC and gas sensitive (e.g. alkalinity, Fe^{2+}, CH_4, H_2S) parameters should be sampled first.

WELL PURGING & SAMPLING FORM
(LOW FLOW)

Project: _____ Sampled by: _____

Location and Site Code (**SITEID**): _____

Well No. (**LOCID**): _____ Well Diameter (**SDIAM**): _____

Date (**LOGDATE**): _____ Weather: _____

CASING VOLUME INFORMATION:

Casing ID (inch)	1.0	1.5	2.0	2.2	3.0	4.0	4.3	5.0	6.0	7.0	8.0
Unit Casing Volume (A) (gal/ft)	0.04	0.09	0.16	0.2	0.37	0.65	0.75	1.0	1.5	2.0	2.6

PURGING INFORMATION:

Measured Well Depth (B) (**TOTDEPTH**) _____ ft. (optional)

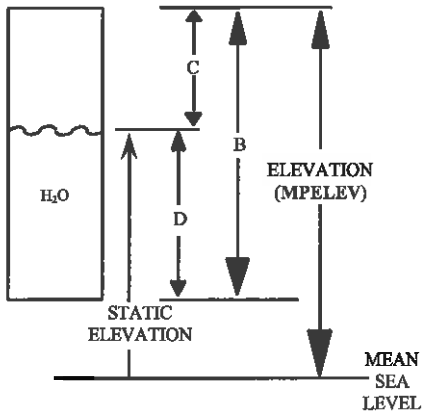
Measured Water Level Depth (C) (**STATDEP**) _____ ft.

Length of Static Water Column (D) = _____ (B) - _____ (C) = _____ (D) ft. (optional)

Pump Intake Depth (ft): _____

Depth during Purging/Sampling: _____ ft
(provide range)

Comments (re: Depth during purging/sampling): _____



Purge Date and Method: BLADDER PUMP _____

Physical Appearance/Comments: _____

Dissolved Ferrous Iron (mg/L): _____

FIELD MEASUREMENTS:

Allowable Range:		± 0.1	± 3%		± 10%	± 10%	± 10mV	
Time	Depth to Water (ft BTOC)	pH	EC (mS/cm)	Temp. (F or C)	Turbidity (NTU)	D.O. (mg/L)	ORP (mV)	Flow Rate (mL/min)

Sample Time: _____ Sample ID: _____

Note: Maintain a flow rate of 200-500 mL/min during purging. Collect samples at a flow rate between 100-250 mL/min. VOC and gas sensitive (e.g. alkalinity, Fe^{2+}, CH_4, H_2S) parameters should be sampled first.

SOIL / SEDIMENT SAMPLING FORM

Project: _____ Sampled by: _____

Site and Site Code (**SITEID**): _____

Sampling Location ID. (**LOCID**): _____

Date (**LOGDATE**): _____ Time: _____

FIELD OBSERVATIONS:

Sample Depth or Interval	**Material Description/ Color**

Comments/Observations:

_____

_____

_____

_____

Sample Time: _____ Sample ID: _____

SOIL VAPOR PROBE MONITORING FORM

DATE: _____ TIME: _____

SAMPLE IDENTIFICATION: _____

SAMPLE DEPTH: _____

FIELD PERSONNEL: _____

INSTRUMENTS (model and serial number):

PUMP: _____

CGI: _____

TRACER GAS VERIFIED: ☐ Yes ☐ No TRACER GAS CONC. (%): _____

SAMPLE PURGE VOLUME: _____

VOLUME OF SOIL VAPOR EXTRACTED: _____

SUMMA CANISTER: VACUUM BEFORE SAMPLING: _____

VACUUM AFTER SAMPLING: _____

APPARENT MOISTURE CONTENT: (DRY/MOIST/SATURATED/ETC.)

_____

Comments/Observations during sampling (odor, other instrument readings):

_____

_____

If sampling near an industrial/commercial building, VOCs used during normal operations of facility:

_____

_____

Weather conditions: Outdoor temperature: _____

Barometric pressure: _____

Wind speed/direction: _____

SUB-SLAB VAPOR PROBE MONITORING FORM

DATE: _____ TIME: _____

SAMPLE IDENTIFICATION: _____

SAMPLE DEPTH: _____

FIELD PERSONNEL: _____

INSTRUMENTS (model and serial number):

PUMP: _____

CGI: _____

TRACER GAS VERIFIED: ☐ Yes ☐ No TRACER GAS CONC. (%): _____

SAMPLE PURGE VOLUME: _____

VOLUME OF SOIL VAPOR EXTRACTED: _____

SUMMA CANISTER: VACUUM BEFORE SAMPLING: _____

VACUUM AFTER SAMPLING: _____

APPARENT MOISTURE CONTENT: (DRY/MOIST/SATURATED/ETC.)

_____

Comments/Observations during sampling (spills, floor stains, odors, other instrument readings):

VOCs used during normal operations of facility:

_____

_____

Weather conditions: Outdoor temperature: _____

Barometric pressure: _____

Precipitation: _____

Ventilation conditions: _____

Heating System Active? ☐ Yes ☐ No Indoor Air Temp: _____

Location in relation to sample location: _____

Windows Closed? ☐ Yes ☐ No

INDOOR/OUTDOOR AIR MONITORING FORM

DATE: _____ TIME: _____

SAMPLE IDENTIFICATION: _____

SAMPLE DEPTH: _____

FIELD PERSONNEL: _____

INSTRUMENTS (model and serial number):

PUMP: _____

CGI: _____

TYPE OF SAMPLE: ☐ INDOOR ☐ OUTDOOR

DURATION OF AIR SAMPLING: _____

VOLUME OF AIR SAMPLED: _____

SUMMA CANISTER: VACUUM BEFORE SAMPLING: _____

VACUUM AFTER SAMPLING: _____

Comments/Observations during sampling (spills, floor stains, odors, other instrument readings):

_____

_____

VOCs used during normal operations of facility: _____

_____

Weather conditions: Outdoor temperature: _____

Barometric pressure: _____

Precipitation: _____

Ventilation conditions: _____

Heating System Active? ☐ Yes ☐ No Indoor Air Temp.:_____

Location in relation to sample location: _____

Windows Closed? ☐ Yes ☐ No

WEATHER OBSERVATION FORM

LOCATION: _____

DATE: _____

FIELD PERSONNEL: _____

INSTRUMENTS (model and serial number):

Thermometer: _____

Anemometer: _____

	Time (military)	Precip. (in)	Atmospheric pressure (in)	Temp. (degrees F)	Wind (mph)	Comments
Prior to Sampling						
Mid Day						
End of Sampling						

Notes: Additional measurements should be taken in case of weather condition changes.
Air sampling will be postponed if conditions move outside the acceptable range.

Sampling Event Acceptable Range:
1. Precipitation: dry while conducting sampling.
2. Atmospheric pressure: 29.7 – 30.4 in Hg.
3. Temperature: 35 – 95 degrees F. The ground must be completely thawed.
4. Wind: <10 mph.

NEW YORK STATE DEPARTMENT OF HEALTH
INDOOR AIR QUALITY QUESTIONNAIRE AND BUILDING INVENTORY
CENTER FOR ENVIRONMENTAL HEALTH

This form must be completed for each residence involved in indoor air testing.

Preparer's Name _____ Date/Time Prepared _____

Preparer's Affiliation _____ Phone No._____

Purpose of Investigation_____

1. OCCUPANT:

Interviewed: Y / N

Last Name: _____ First Name: _____

Address: _____

County: _____

Home Phone: _____ Office Phone: _____

Number of Occupants/persons at this location _____ Age of Occupants _____

2. OWNER OR LANDLORD: (Check if same as occupant ___)

Interviewed: Y / N

Last Name: _____ First Name: _____

Address: _____

County: _____

Home Phone: _____ Office Phone: _____

3. BUILDING CHARACTERISTICS

Type of Building: (Circle appropriate response)

Residential	School	Commercial/Multi-use
Industrial	Church	Other: _____

If the property is residential, type? (Circle appropriate response)

Ranch	2-Family	3-Family
Raised Ranch	Split Level	Colonial
Cape Cod	Contemporary	Mobile Home
Duplex	Apartment House	Townhouses/Condos
Modular	Log Home	Other:_____

If multiple units, how many? _____

If the property is commercial, type?

Business Type(s) _____

Does it include residences (i.e., multi-use)? Y / N If yes, how many? _____

Other characteristics:

Number of floors_____ Building age_____

Is the building insulated? Y / N How air tight? Tight / Average / Not Tight

4. AIRFLOW

Use air current tubes or tracer smoke to evaluate airflow patterns and qualitatively describe:

Airflow between floors

_____

_____

_____

Airflow near source

_____

_____

_____

Outdoor air infiltration

_____

_____

_____

Infiltration into air ducts

_____

_____

_____

5. BASEMENT AND CONSTRUCTION CHARACTERISTICS (Circle all that apply)

a. Above grade construction:	wood frame	concrete	stone	brick
b. Basement type:	full	crawlspace	slab	other _____
c. Basement floor:	concrete	dirt	stone	other _____
d. Basement floor:	uncovered	covered	covered with _____	
e. Concrete floor:	unsealed	sealed	sealed with _____	
f. Foundation walls:	poured	block	stone	other _____
g. Foundation walls:	unsealed	sealed	sealed with _____	
h. The basement is:	wet	damp	dry	moldy
i. The basement is:	finished	unfinished	partially finished	
j. Sump present?	Y / N			
k. Water in sump?	Y / N / not applicable			

Basement/Lowest level depth below grade: _____(feet)

Identify potential soil vapor entry points and approximate size (e.g., cracks, utility ports, drains)

_____

_____

6. HEATING, VENTING and AIR CONDITIONING (Circle all that apply)

Type of heating system(s) used in this building: (circle all that apply – note primary)

Hot air circulation	Heat pump	Hot water baseboard	
Space Heaters	Stream radiation	Radiant floor	
Electric baseboard	Wood stove	Outdoor wood boiler	Other _____

The primary type of fuel used is:

Natural Gas	Fuel Oil	Kerosene
Electric	Propane	Solar
Wood	Coal	

Domestic hot water tank fueled by: _____

Boiler/furnace located in:	Basement	Outdoors	Main Floor	Other_____
Air conditioning:	Central Air	Window units	Open Windows	None

Are there air distribution ducts present? Y / N

Describe the supply and cold air return ductwork, and its condition where visible, including whether there is a cold air return and the tightness of duct joints. Indicate the locations on the floor plan diagram.

_____

_____

_____

_____

7. OCCUPANCY

Is basement/lowest level occupied? Full-time Occasionally Seldom Almost Never

Level	**General Use of Each Floor (e.g., familyroom, bedroom, laundry, workshop, storage)**
Basement	_____
1st Floor	_____
2nd Floor	_____
3rd Floor	_____
4th Floor	_____

8. FACTORS THAT MAY INFLUENCE INDOOR AIR QUALITY

a. Is there an attached garage? Y / N

b. Does the garage have a separate heating unit? Y / N / NA

c. Are petroleum-powered machines or vehicles stored in the garage (e.g., lawnmower, atv, car) Y / N / NA Please specify_____

d. Has the building ever had a fire? Y / N When?_____

e. Is a kerosene or unvented gas space heater present? Y / N Where?_____

f. Is there a workshop or hobby/craft area? Y / N Where & Type? _____

g. Is there smoking in the building? Y / N How frequently? _____

h. Have cleaning products been used recently? Y / N When & Type? _____

i. Have cosmetic products been used recently? Y / N When & Type? _____

j. Has painting/staining been done in the last 6 months? Y / N Where & When? _____

k. Is there new carpet, drapes or other textiles? Y / N Where & When? _____

l. Have air fresheners been used recently? Y / N When & Type? _____

m. Is there a kitchen exhaust fan? Y / N If yes, where vented? _____

n. Is there a bathroom exhaust fan? Y / N If yes, where vented? _____

o. Is there a clothes dryer? Y / N If yes, is it vented outside? Y / N

p. Has there been a pesticide application? Y / N When & Type? _____

Are there odors in the building? Y / N
If yes, please describe: _____

Do any of the building occupants use solvents at work? Y / N
(e.g., chemical manufacturing or laboratory, auto mechanic or auto body shop, painting, fuel oil delivery, boiler mechanic, pesticide application, cosmetologist

If yes, what types of solvents are used? _____

If yes, are their clothes washed at work? Y / N

Do any of the building occupants regularly use or work at a dry-cleaning service? (Circle appropriate response)

Yes, use dry-cleaning regularly (weekly) No
Yes, use dry-cleaning infrequently (monthly or less) Unknown
Yes, work at a dry-cleaning service

Is there a radon mitigation system for the building/structure? Y / N Date of Installation: _____
Is the system active or passive? Active/Passive

9. WATER AND SEWAGE

Water Supply:	Public Water	Drilled Well	Driven Well	Dug Well	Other: _____
Sewage Disposal:	Public Sewer	Septic Tank	Leach Field	Dry Well	Other: _____

10. RELOCATION INFORMATION (for oil spill residential emergency)

a. Provide reasons why relocation is recommended: _____

b. Residents choose to: remain in home relocate to friends/family relocate to hotel/motel

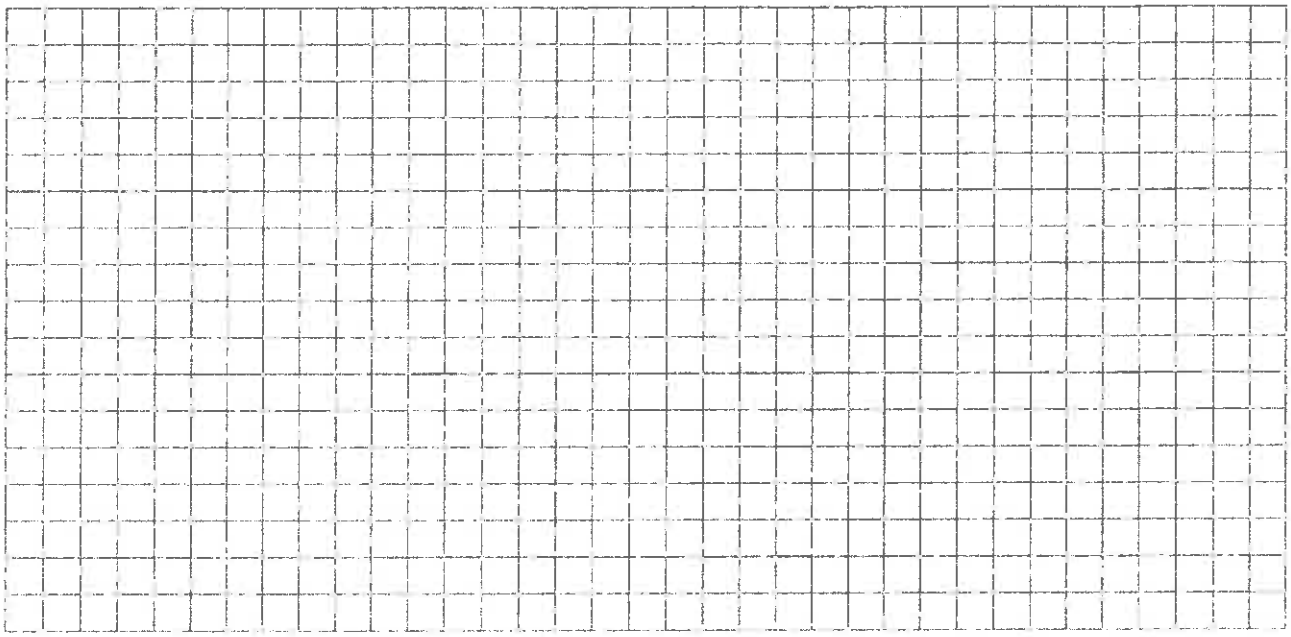
c. Responsibility for costs associated with reimbursement explained? Y / N

d. Relocation package provided and explained to residents? Y / N

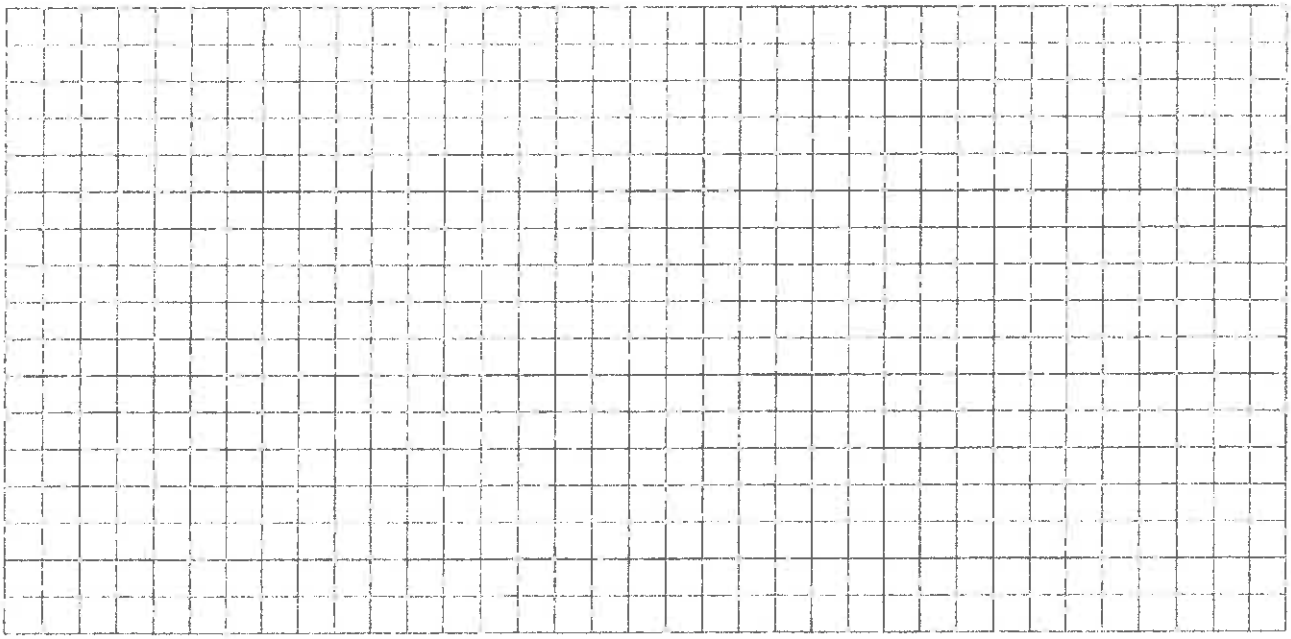
11. FLOOR PLANS

Draw a plan view sketch of the basement and first floor of the building. Indicate air sampling locations, possible indoor air pollution sources and PID meter readings. If the building does not have a basement, please note.

Basement:



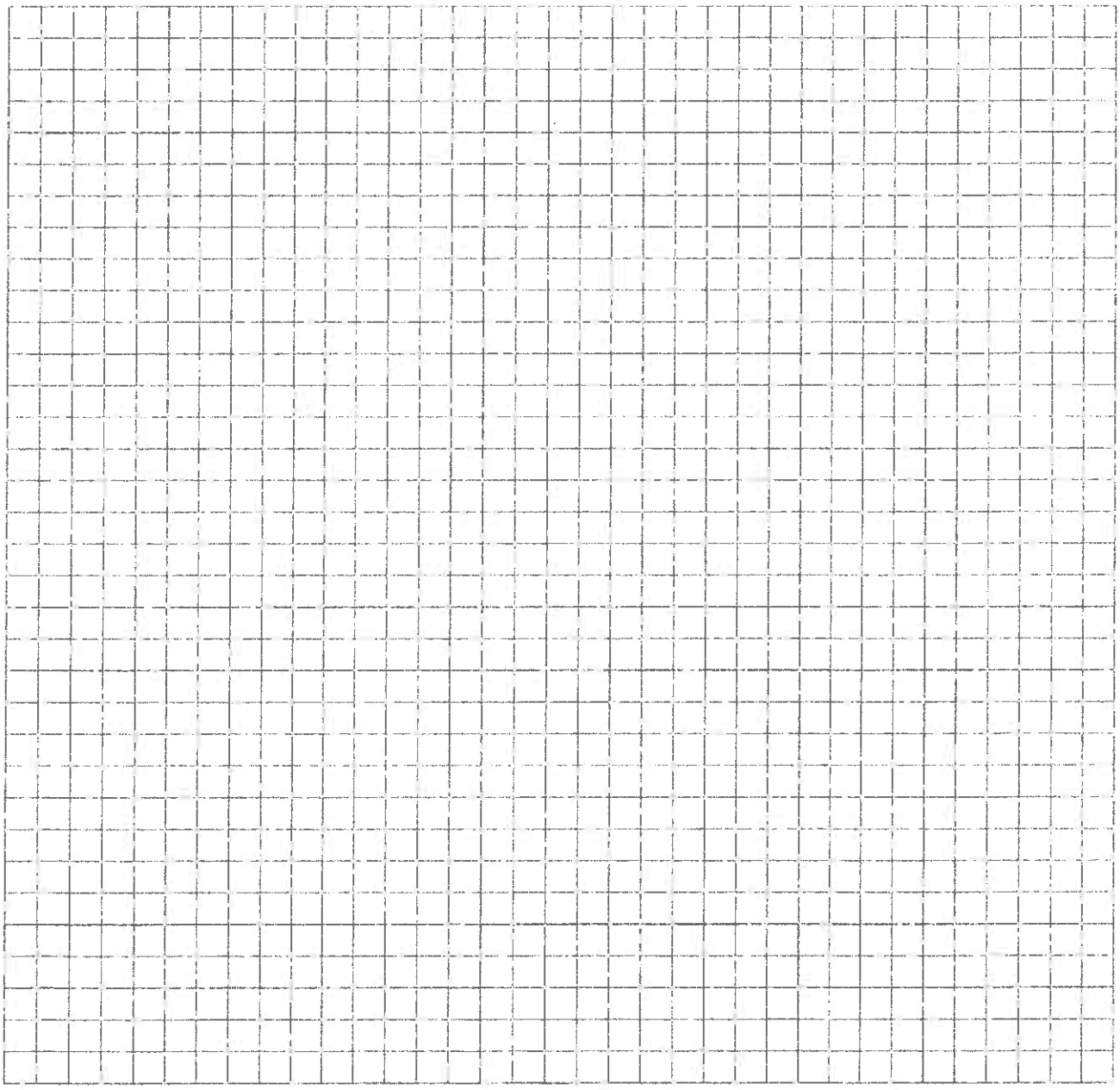
First Floor:



12. OUTDOOR PLOT

Draw a sketch of the area surrounding the building being sampled. If applicable, provide information on spill locations, potential air contamination sources (industries, gas stations, repair shops, landfills, etc.), outdoor air sampling location(s) and PID meter readings.

Also indicate compass direction, wind direction and speed during sampling, the locations of the well and septic system, if applicable, and a qualifying statement to help locate the site on a topographic map.



13. PRODUCT INVENTORY FORM

Make & Model of field instrument used: _____

List specific products found in the residence that have the potential to affect indoor air quality.

Location	Product Description	Size (units)	Condition*	Chemical Ingredients	Field Instrument Reading (units)	Photo ** Y / N

* Describe the condition of the product containers as **Unopened (UO), Used (U),** or **Deteriorated (D)**
** Photographs of the **front and back** of product containers can replace the handwritten list of chemical ingredients. However, the photographs must be of good quality and ingredient labels must be legible.

Daily Health and Safety Meeting Form

Date: _____ *Time :* _____

Location: FPM office (sample room)

Weather Conditions: _____

Meeting Type: Daily Health and Safety

Personnel Present:

_____

_____

Visitors Present: _____

Visitor Training: _____

PPE Required: Modified D

Possible risks, injuries, concerns:

_____

_____

_____

Anticipated Releases to Environment (if so, describe and detail response action/control measures implemented):

_____

_____

Property Damage:

_____

_____

Description (include sequence of events describing step by step how incident happened):

_____

_____

Analysis for, and Implementation of Corrective/Preventative Procedure to Prevent Future Occurrences (to be formulated by SSHO + FOM, approved by PM, and SSHO implemented):

_____

_____

_____

Report made by (Name): _____

SSHP Organization Title: Site Safety and Health Officer

Daily Health and Safety Inspection Form

Date:_____ *Time:* _____

Location: _____

Personnel Present: _____

_____

_____

*Visitors Present:*_____

_____

Behavior, approach or practice that was found unacceptable:

_____

_____

_____

*Possible risks, injuries, concerns, deviations from H&S Plan:*_____

_____

_____

_____

Anticipated releases to environment or anticipated future Health and Safety risks: _____

_____

_____

*Analysis for, and Implementation of Corrective/Preventative Procedure to Prevent Future Occurrences (to be formulated by SSHO and FOM, approved by PM, and implemented by SSHO):*_____

_____

_____

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Report made by (Name): _____

SSHP Organization Title: ____Site Safety and Health Officer_____

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Well Decommissioning Form

Date	Well ID	Depth to Water (TOIC)	Total Depth (TOIC)	Method of Decommissioning	Physical Description (Well Type, etc.)	Grout Added (gallons)

Appendix B

APPENDIX B

LABORATORY STANDARD OPERATING PROCEDURES

APPENDIX B

LABORATORY STANDARD OPERATING PROCEDURES

Title: Determination of VOCs in Ambient Air EPA Compendium Methods TO14 and TO15

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Approval Date: August 16, 2012

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

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1.0 Scope and Application

This SOP describes the laboratory procedure for the analysis of polar and non-polar volatile organic compounds (VOCs) in ambient air. The procedure is applicable to those VOCs that have been evaluated by the laboratory for their consistent performance in meeting the control criteria put forth in Compendium Method TO-15. While the compendium method is specifically written for the analysis of samples collected in leak-free passivated stainless steel canisters, it may be applied to the analysis of samples that have employed the use of other collection devices such as Tedlar bags and solid absorbents.

1.1 Analytes, Matrix(s), and Reporting Limits

The target compound list and reporting limits for each compound are provided in Table 1.

2.0 Summary of Method

An aliquot of sample is pulled from the canister through a solid multi sorbent bed trap which reduces the water content of the sample. The sample is thermally desorbed and the VOCs are carried onto a GC column coupled to a mass spectrometer. Compounds are identified by comparison of the mass spectra for individual peaks in the total ion chromatogram to the fragmentation patterns of ions corresponding to VOCs including the intensity of primary and secondary ions as well as the patterns of stored spectra acquired under similar conditions. The concentration of the target compound is calculated by internal standard technique using the average response factor of that compound as determined by the initial calibration.

This procedure is based on EPA Compendium Method TO-15 "Determination of Volatile Organic Compounds in Ambient Air using Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry", US EPA, January, 1999.

If the laboratory has modified the method, a list of these modifications may be found in Section 16.0.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Contamination may occur if canisters or other equipment is not properly cleaned before use. The laboratory procedures for canister and flow controller cleaning procedures are provided in Appendices C and D.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples

and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The analytical system contains zones with elevated or depressed temperatures that are capable of causing injury upon direct contact. The analyst needs to be aware of the locations of those zones, and allow them to return to room temperature prior to maintenance activities or take measures to avoid contact with hot and/or cold surfaces. There are areas of high voltage in the analytical system. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

Liquid nitrogen (LN2) is used for cryogenic purposes. In addition to avoiding contact with LN2 cooled surfaces, analysts must be aware of the potential for oxygen depletion in a confined space in the event of an unexpected large release of the product. Users should evacuate a confined space in which large amounts of LN2 have been released.

Sample canisters are occasionally pressurized for cleaning or sample dilution purposes. Lab systems are designed to ensure that the cans are not pressurized above 40 psi. Eye protection must be worn when cans are pressurized in the event of a canister failure.

5.2 Primary Materials Used

There are no materials used in this method which have a serious or significant hazard rating **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment meets the specification of this SOP.

6.1 Sampling Equipment

- 6L, 3L, and 1L SUMMA® Canisters: Leak-Free, Passivated Stainless Steel, with Swagelok DSS4 Valves, or equivalent.

- 6L SUMMA® Canisters: Silicon lined-Leak-Free, Passivated Stainless Steel, with Swagelok DSS4 valves or equivalent.

- Flow Controllers: Restek Catalog #24239 or equivalent.

- Flow Controller Orifice: Various sizes ranging from 0.008” to 0.060”, Restek or equivalent.

- Flow Controller Vacuum Gauges: Capable of measuring vacuum to an absolute vacuum of -30” of HG, and pressure up to 30 psi, Grainger Catalog #5WZ37 or equivalent.

- Rain Guard: Stainless Steel Tubing ¼", 10ft. Grainger or Equivalent. Cut 8" and bend into a J shape using a pipe bender.

- Stainless Steel Pre-Filter (7 um): Swagelok Catalog# SS-4F-T7-7 or equivalent.

- Teflon Tape: Home Depot Brand or equivalent.

6.2 Analytical System

- Mass Spectrometer: Agilent 5973 or 5972 MSD or equivalent.
- Gas Chromatograph: Agilent 6890 or equivalent.
- VOC Autosampler: Entech 7016CA or equivalent.
- Cryogenic Concentrator: Equipped with an electronic mass flow controller that maintains a constant flow for carrier gas and sample over a range of 0-200 cc/min. Entech 7100A or equivalent.
- Low Pressure Liquid Nitrogen: Air Gas or equivalent.
- Glass Bead Cryotrap: Capable of effectively removing water while trapping polar and non-polar compounds. Entech catalog# 01-04-11320.
- TENAX Sorbent Trap: Capable of removing CO2 and trapping the polar and non-polar compounds. Entech catalog # 01-04-11330.Primary Column: Fused silica capillary column (60 m x 0.32 mm x 1.8 μm), Restek RTX-624 or equivalent.
- Data System: PC software for Entech instrumentation. Hewlett-Packard ChemStation data acquisition software and Hewlett-Packard ChemServer, Target 3.5 data processing software or TestAmerica Chrom and TestAmerica LIMS (TALS).

6.3 Cleaning System

- Canister Cleaner Module and Software: Capable of filling canisters with humidified air and evacuating canisters to 50 mtorr, Entech Model 3100A or equivalent.

- Vacuum Pump: Capable of evacuating sample canisters to full vacuum. Vacuubrand or equivalent.

- Cleaning Manifold: Equipped with stainless steel and Teflon transfer lines and connections for cleaning up to twelve canisters simultaneously.

- Heating Belts: Individual thermal-stated heating belts used to heat canisters to 100ºC during the manifolds cleaning cycles. Entech or equivalent.

- Cleaning oven: Capable of cleaning 6 Summa Cans simultaneously at a temperature of 100ºC. Entech or equivalent.

- Flow Controller Cleaning Manifold: Capable of flushing hot Nitrogen through 24 flow controllers simultaneously for cleaning.

6.4 Miscellaneous Supplies

- Mass Flow Controller, NIST Traceable: Capable of flow rate of 70 mL/min, McMillan Company 80SD or equivalent. Use for the preparation of calibration and working standards.

- Zero Air Generator: Ballston Model HPZA-3000 or equivalent.
- Syringes: Gas tight with a Luer-Lok tip, assorted sizes ranging from 1.0 mL to 1.0 L, SGE or equivalent.

- Digital Pressure Gauges, NIST Traceable: Capable of measuring pressure in the range of -30" Hg to 100 psi, Dwyer Models DPGA-12 and 67100 or equivalent.

- Digital Flow Meter, NIST Traceable: Alltech or equivalent.

7.0 Reagents and Standards

7.1 Reagents

- Ultra Pure Humidified Zero Air - Pass ambient laboratory air through a zero air generator. The zero air generator humidifies the air to a relative humidity of >20 percent.

7.2 Standards

Purchase the following stock standard mixtures from commercial vendors:

- Mixed Gas Stock Standard: - Commercially prepared standard that includes internal standard and tune standard compounds: Bromofluorobenzene, Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d5, and at a concentration of 100 ppbv each. Spectra Gas or equivalent.

- Calibration Stock Standard: - Commercially prepared custom gaseous stock standard used by all network facilities that includes all target analytes at a concentration of 1.0 ppmv. Spectra Gases or equivalent.

- Calibration Ethanol Neat Material. >99.5 %

- ICV / LCS Stock Standard: - Custom made gaseous stock standard prepared from a different lot(s) of the source material(s) used to manufacture the calibration stock standard. The ICV/LCS stock standard includes all target analytes at a concentration of 1.0 ppmv. Spectra Gases.

- ICV/LCS Ethanol Neat material. >99.5% from a source other than the calibration source.

Prepare calibration and working standards mixtures by diluting a known volume of the stock standard in humidified ultra pure zero air to a specified volume. The formulations for standard preparation are provided in Appendix B along with recommended expiration dates and storage conditions.

Each stock standard is assigned a 1 year expiration date from manufacture and recertified annually. The ethanol neat material is assigned the expiration date given by the manufacturer.

The recertification procedure is as follows:

Internal Standard Mixed Gas Stock: Assay the active internal standard cylinders against a new, vendor certified cylinder purchased from Spectra Gas. Verify that the % difference between new, vendor certified cylinder and the active cylinder is within 20%. If this criterion is not met, replace the cylinder.

Calibration Stock Standard and the ICV/LCS Stock Standard: Return one of the cylinders to Spectra for recertification (rotate a different cylinder each year). When the re-certified stock standard cylinder is returned, assay against all active cylinders. The difference between the recertified value and the assay values should be within 15%. If these criteria are not met, return the cylinder that was not re-certified to the vendor for recertification or purchase a new stock standard.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Air	1L ,3L or 6L Passivated Summa Canister	1L	NA	30 days from collection	EPA TO-15

All samples should be collected in passivated stainless steel canisters that have been certified clean prior to sampling. The laboratory will provide certified clean canisters to the client upon request. The procedures for clean canister certification are provided in Laboratory SOP BR-AT-011.

The laboratory can also provide flow controllers set to the appropriate flow rate for the sampling time required by the client.

The laboratory ships air canisters in custom made boxes. The boxes are equipped with custom-made foam inserts to hold the pre-set flow-controllers. The custom shipping materials are designed to prevent damage of equipment to and from the sampling site. The laboratory checks the equipment to ensure it is in proper working order before shipment to the client and additional checks are performed on return of the equipment to the laboratory. Sampling instructions are provided with each sampling kit. The sampling crew is advised to handle the sampling equipment using the instructions provided by the laboratory to ensure optimum performance.

Samples should be stored at ambient temperature.

The analytical holding time is 30 days from the date of sample collection.

9.0 Quality Control

9.1 Sample QC

The following quality control samples are prepared with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Table 3
Internal Standard (ISTD)	Every Sample	See Table 3
Laboratory Control Sample Duplicate (LCSD)	Client request	See Table 3
Sample Duplicate (SD)	Client Request	See Table 3
Trip Blank (TB)	Client Request	See Table 3

NOTE: The compendium reference method does not require the analysis of a laboratory control sample (LCS) or provide criteria for the evaluation of an LCS. The laboratory performs an LCS at the above mentioned frequency as an evaluation of percent recovery in a blank matrix. The control limits set by the laboratory for the LCS (70-130) are those specified in Section 11.4 of the reference method for the audit accuracy evaluation.

The compendium method does not require analysis of a LCSD. Evaluations for precision should be derived from field samples. Duplicate precision should be measured by the analysis of a sample duplicate. Replicate precision should be measured from separate aliquots taken from the same sample canister. The laboratory will perform an LCSD to measure precision only per client request and analysis of the LCSD is considered a billable. The acceptance criteria for duplicate and replicate precision is <25%. Unless otherwise specified by the client during project initiation, the LCSD will be used to measure precision only. The LCS will be used for evaluations for percent recovery and to determine if corrective action is necessary.

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Tune Standard (BFB)	Each Analytical Window	See Section 10.0
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Section 10.0
Initial Calibration Verification (ICV)	Once, after each ICAL	See Section 10.0
RT Window Establishment	Once per ICAL	See Section 10.0
Relative Retention Time (RRT)	With each sample	See Section 10.0
Continuing Calibration Verification (CCV)	Daily, after each BFB	See Section 10.0

10.0 Procedure

10.1 Support Equipment Calibration

Verify the calibration of the mass flow controller used to prepare standards, the calibration of the digital flow meter used to set and check the flow rates of the FC(s) used for sample collection, and the calibration of the digital pressure gauges used to check return canister pressure is current to the year. Immediately notify the QA department if the calibration is not current and

wait for further instruction. Equipment whose calibration has expired may not be used without documented approval from the QA department.

NOTE: The QA department schedules the annual calibrations of the support equipment and maintains all Certificates of Calibration. The flow controllers are checked against a NIST traceable standard. This check is performed by the manufacturer of the equipment, when possible, or by an approved vendor that provides certification service.

10.2 Instrument Calibration

10.2.1 Tune Standard

Analyze a tune standard (BFB) prior at the beginning of each analytical window. The tune standard is a commercially prepared mixed gas stock standard that includes bromofluorobenzene (BFB) at a concentration of 100 ppbv.

To analyze the tune standard:

1) Establish the instrument operating conditions specified in Section 10.4.1.

2) Attach the mixed gas stock standard to the Entech concentrator by attaching the cylinder to the line dedicated for introduction of the internal standard (ISTD). The concentrator directly injects 20 mL of the 100 ppbv stock standard onto the instrument to yield an on column concentration of 10 ppbv.

3) Acquire the data and evaluate the results against the acceptance criteria given in Table 2. Criteria must be met prior to further analysis. The official start time of the 24 hour analytical window is the injection time of a passing tune standard. All samples must be injected within 24 hours of that time.

NOTE: The data processing system averages three scans (apex scan, scan prior, and scan following) and performs background subtraction of the single scan prior to the elution of BFB.

10.2.2 Initial Calibration (ICAL)

The instrument must be calibrated with a minimum of five calibration standards for each target analyte at concentrations that span the working range of the method.

The laboratory routinely analyzes 8 standards at the recommended concentrations of 0.04, 0.20, 0.50, 5.0, 10.0, 15.0, 20 and 40 ppbv, except for Ethanol. For Ethanol, a 6 point curve is analyzed at the following concentrations: 5, 10, 15, 20, 40, and 100 ppbv. Even though seven calibration standards are routinely analyzed not every calibration standard is used for each analyte. Each analyte has been assigned to an analyte group that includes a calibration range of at least five standards. The analyte group associations for each target analyte are provided in Table 1. The calibration range for each analyte group is as follows:

- Group A: This analyte group is associated with a seven point calibration curve. The calibration range is 0.20 to 40 ppbv with the 0.04 ppbv standard routinely excluded. The limit of quantitation (LOQ) for this group of analytes is 0.20 ppbv

- Group B: This analyte group is associated with a six point calibration curve. The calibration range is 0.50 to 40 ppbv with the 0.04 and 0.20ppbv standards routinely excluded. The limit of quantitation (LOQ) for this group of analytes is 0.50 ppbv.

- Group C: This analyte group is associated with a five point calibration curve. The calibration range is 5.0 to 40 ppbv with the 0.20, 0.50, and 0.04 ppbv standards routinely excluded. The limit of quantitation (LOQ) for this group of analytes is 5.0 ppbv.

- Group D: This analyte group is an eight point calibration curve. The calibration range is 0.04 40 ppbv. The limit of quantitation (LOQ) for this group of analytes is 0.04 ppbv.

- Group E: (Ethanol : This analyte has a six point calibration curve. The calibration range is 5 to 100 ppbv. The limit of quantitation (LOQ) for this analyte is 5 ppbv.

Prepare the calibration standards using the formulations provided in Appendix B.

Analyze the standards in a sequence from lowest to highest concentration using the instructions provided in Section 10.4.2.

The data processing system calculates a relative response factor (RRF), for each analyte and isomer pair using the assigned internal standard. The internal standard associations for each target analyte are provided in Table 1. The data processing system also calculates a mean relative response factor, relative standard deviation (RSD), relative retention time (RRT) and the mean RRT.

The following criteria must be met for a calibration to be considered acceptable:

- The RSD for each target analyte must be <30% with at most 2 exceptions up to a limit of 40%**.**

- The area response for the primary quantitation ion for the internal standard for each ICAL standard must be within 40% of the mean area response over the calibration range for each internal standard.

- The RRT for each target compound at each calibration level must be within 0.06 RRT units of the mean RRT for the compound. The retention time shift for each of the internal standards at each calibration level must be within 20 seconds of the mean retention time over the initial calibration range for each internal standard.

If these criteria are not met inspect the system for problems and perform corrective action. Recommended corrective actions are provided in Section 10.2.5 and in Table 3.

Repeat initial calibration whenever instrument operating conditions are changed, a new column is installed, when significant instrument maintenance has been performed, and when the result of the CCV indicate the calibration is no longer valid.

10.2.3 Second Source Calibration Verification (ICV)

Immediately following an acceptable initial calibration verify the accuracy of the calibration by the analysis of the second source calibration verification standard (ICV).

Prepare the ICV following the formulation provided in Appendix B.

Analyze the ICV following the instructions provided in Section 10.4.2.

The percent recovery (%R) for each target analyte must be within 70-130%. If criteria are not met, perform corrective action. Recommended corrective actions are provided in Table 3. If corrective action is not successful, remake your standards and recalibrate.

If after successful analysis of the ICV, time remains in the 24-hour analytical window, QC and field samples may be analyzed without analysis of a continuing calibration verification check standard. If time does not remain in the analytical window, a new analytical sequence must be initiated with a Tune Standard followed by daily calibration (CCV).

10.2.4 Continuing Calibration Verification (CCV)

Analyze the CCV immediately after the tune standard unless the analytical window includes ICAL, in which case, a CCV is not required.

Prepare the CCV standard using the formulation given in Appendix B. The recommended concentration of the CCV for each target analyte is 10.0 ppbv.

Analyze the CCV following the instructions provided in Section 10.4.2. The data system calculates a response factor for each analyte and calculates the percent difference (%D) of the RRF relative to the mean RRF in the most recent initial calibration.

- The %D for each target analyte must be within ±30%. If the above criteria are not met, repeat the analysis of the CCV once. If the second CCV meets criteria, continue with the analytical sequence. If it fails, evaluate the data to determine if one of the following conditions is met. If these conditions are not met corrective action must be taken. Guidance for troubleshooting is provided in Section 10.2.5. After corrective action the analytical sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If these two CCVs do not meet the criteria, recalibration is required prior to further analysis.

  - If the CCV criteria are exceeded high, indicating a high bias, and the associated samples have non-detects for those analytes, the analytical data may be considered usable. In the absence of instructions otherwise, proceed with analysis.

  - If the CCV criteria are exceeded low, indicating a low bias, analytical results may be reported if those results exceed the project's regulatory decision level. In other words, if the analytical results are sufficiently high to counter the low bias, results may be reported. Consult with the project manager to determine if the exception is allowable for each project.

10.2.5 Troubleshooting

Check the following items in case of calibration failures:

- Loss of sensitivity or unstable ISTD recoveries are usually the result of a leak. Check the union between the GC column and Entech transfer line.

- Loss of sensitivity for individual compounds may be a result of either an active site in a transfer line or a bad trap. Troubleshoot and perform maintenance as necessary.

- Poor chromatography usually requires GC column maintenance, perform as necessary.

- Carryover is usually caused by excessive amounts of analyte introduced to the system. Analyze blanks until the system is cleaned or replace the traps and transfer lines if necessary.

Refer to corporate policy CA-Q-S-005 for additional information of procedures to establish and troubleshoot initial calibration curves.

10.3 Sample Preparation

10.3.1 Post Sampling Canister Pressure Check Procedure

Perform the post-sampling canister pressure check within 1 business day of receipt of canisters in the laboratory so that any problems found are quickly identified and communicated to the client. Record the date and time the post-receipt check is performed is in the analytical record.

To perform the post- sampling canister pressure check:

1) Inspect the condition of equipment for signs of damage. If damage is observed, immediately notify the PM and await further instruction.

2) Record your checks on the Air Canister Post-Sampling Pressure Check Record.

3) Check to see if the sampling FC(s) were returned with the canister(s). If so, check the paperwork (Canister ID Tag, Field Test Data Sheet or Chain of Custody (COC) to determine if the sampling record identifies which FC was used for each canister. If the paperwork does not include this information, record the omission on the post-sampling check record.

   NOTE: The laboratory's sample acceptance policy for air samples in canisters requires that the sampling crew record the ID of the FC used for sample collection on the tag attached to each canister, but the association may also be recorded on the Field Test Data Sheet or a COC. With this information the laboratory can review the history of use of the FC as needed to troubleshoot potential equipment problems. Without the association, the history of use of the FC is unknown. The laboratory strongly recommends that field samplers be instructed to provide this information for each sampling event.

4) Check the pressure of each canister using a digital pressure gauge. Verify that the calibration of the gauge has not expired prior to use. If the calibration sticker indicates calibration checks are past due, notify department management and remove the gauge from service.

Attach the gauge to the canister inlet. Check for the presence of burr or thread damage when attaching the pressure gauge to the canister inlet. If damage is observed, record the observation on the post-sampling check record

Open the canister valve and record the pressure reading.

The pressure should be between -10" Hg to -1"Hg, except for "grab" samples and samples whose sampling time is <200 mL/min, which do not have a return pressure criteria range.

NOTE: The return canister pressure criteria of -10"Hg to -1"Hg was established based on the recommendations provided in Section V of Appendix I of the Vapor Intrusion Guidance Document prepared by the New Jersey Department of Environmental Protection (NJDEP). These sections of this document describe canister and quality assurance requirements for USEPA Methods TO-15 and TO-17. This document explains that due to recent advances in technology in concentrator units, such as with the Entech concentrators used by the laboratory, it is now possible to remove sample from canisters with a negative pressure of -10"Hg without having to add makeup air. Previous models of concentrators (such as NuTechs) required a pressure of at least -5"Hg. When the return negative pressure of a canister is greater than -10"Hg, the laboratory may need to add makeup air to the canister in order to provide a sufficient amount of sample for analysis. The need for the addition of makeup air depends on the concentrator unit. Some concentrator units used by the laboratory can pull sample without the addition of make-up air when the return negative pressure is up to -15"Hg. The amount of air added to the canister depends on the return pressure reading and will vary with each canister. See section 10.3 for the procedure of the determination of amount of makeup air needed. Except for "grab" samples and samples whose sampling time is set for <200 mL/min, the return pressure of a canister should never be zero negative pressure or a pressure equal to ambient pressure. If it is, the PM must consult with the client and obtain authorization for the laboratory to proceed with analysis.

5) If the return pressure is within range for all samples, photocopy the post-sampling canister pressure check record and attach the record to the screen worksheet.

   If the return pressure is not within range, initiate an anomaly report and perform one of the following actions:

   - Action 1: If the FC was not returned with the canister, attach a copy of the post-sampling pressure check record to the anomaly report and forward the paperwork to the PM who will notify the client of the situation and request further instruction. The PM will record any decisions made regarding the sample on the anomaly report and return the packet to you. Attach a photocopy of the complete anomaly report to the screen worksheet and forward the original anomaly report to the QA department.

   - Action 2: If the FC was with the canister, perform a leak check on the FC gauge and re-check the FC's flow rate as follows. Record all measurements on the original Flow Controller Set Flow Rate & Leak Check Record. The protective sticker on the back of the FC will provide the page number that corresponds to the logbook record.

   To check the flow rate and check the FC gauge for leaks:

1) Remove the stem from the FC.

2) Attach a dust cap where the stem was, and attach the FC to the control gauge/vacuum manifold.

3) Turn the calibrated digital flow meter on and zero the meter.

4) Turn on the vacuum pump and read the vacuum of the control gauge and the FC gauge and record these readings.

5) Turn off the vacuum. Wait ~30 seconds and read the vacuum of the control gauge and the FC gauge and record these readings.

6) The difference between the initial and final readings for the control gauge should be zero. If it is not, there is a leak in the manifold system. Stop work and correct the problem.

7) The difference between the initial and final readings for the FC gauge should be zero. If it is not, there is a leak in the FC assembly. Record the presence of a leak in the record and set aside the FC for service and repair.

8) Remove the dust cap from the FC and re-attach the stem.

9) Attach the flow meter tube to the stem of the FC.

10) Measure the flow rate. The flow rate should be within the ranges specified in the Set Flow Rate Table (See Table 4) for the sampling time requested. If the flow rate is not within range, record the situation on the anomaly report.

11) Attach a photocopy of the Flow Controller Set Flow Rate & Leak Check Record and a photocopy of the post sampling check record to the anomaly report and forward the paperwork to the PM who will notify the client of the situation and request further instruction. The PM will record any decisions made regarding the sample on the anomaly report and return the packet to you. Attach a photocopy of the complete anomaly report to the screen worksheets and forward the original anomaly report to the QA department.

10.3.2 Sample Screening

At the laboratory's discretion unknown samples may be screened prior to initial analysis to determine if the sample requires dilution. Unless otherwise requested by the client the laboratory does not provide screen data with the data package report even when primary dilutions are performed based on the results of the screen analysis.

To prepare a sample for screen analysis connect the sample canister to the autosampler connected to screening instrument and analyze 20 mL of sample. Acquire and evaluate the results. If the results of screen analysis indicate that a target compound is above its upper

range of calibration. Calculate a recommended dilution factor (DF) by dividing the concentration of analyte found by 30. Record the recommended DF on the screen worksheet.

NOTE: Samples are screened on a GC/MS instrument that is programmed with the operating conditions given in Section 10.4.1 of this SOP. The calibration is checked weekly with a single point calibration standard at a concentration of 10 ppbv for all target analytes. The calibration is checked more frequently when the results of instrument analysis do not correlate well with the results of the screen analysis.

10.3.3 Sample Dilutions & Pressure Adjustment

Field samples should be diluted prior to initial analysis when the screen results indicate that the concentrations are above calibration range and when the laboratory has sufficient knowledge of the sample (history) to know that the sample will require dilution. Field samples must be reanalyzed at a dilution initial analysis when the concentration of target compounds in initial analysis exceed of the upper range of calibration.

When the return negative pressure of a canister is greater than -15”Hg, make-up air is added to provide sufficient volume of make-up air in order to have an adequate sample volume for analysis. The addition of make-up air is considered a canister dilution.

To dilute the sample:

1) Attach the sample canister to the zero air line equipped with a pressure gauge that reads negative pressure in (“Hg) and positive pressure in (psig).

2) Ensure the valve of the zero air line is closed then open the valve of the sample canister. Record the negative pressure reading in the Canister Dilution Worksheet or on the canister’s tag.

3) Slowly open the valve of the zero air line and fill the canister until canister pressure gauge reads -10”Hg. Do not open the valve to such an extent that the zero air line pressure drops below 15 psig and do not allow the zero air line to reach equilibrium otherwise you will contaminate the zero air line.

4) When the desired pressure is achieved, close the canister valve and the valve on the zero air line; wait 15 seconds.

5) Open the canister valve and record the final pressure reading in psig.

6) Close the canister valve and remove the valve from the zero air line.

7) Record the initial and final pressure readings in the TALS canister dilution tracking module. If the final pressure is below ambient, the “HG reading must be converted to psig by dividing the value by 2 prior to entry into the TALS worksheet.

When the return pressure of a canister is positive, the pressure must be adjusted to near ambient (0”Hg) prior to analysis. To adjust the pressure to ambient, vent the canister to ambient in a fume hood by opening the canister value for ~4-5 seconds, close the valve. For higher

pressure canisters, open the valve and listen for a release of air then close the valve when the sound recedes.

If a trip blank is provided, pressurize the trip blank canister to 10 psig. The pressurization of the trip blank is not considered a dilution.

10.3.4 QC Sample Preparation

To prepare the method blank (MB): Fill a clean canister that has never been used to collect environmental samples and has never left the laboratory to 20 psig with zero air. Continue to use this canister as the MB until the pressure of the canister reaches 0 psig, at which time, recharge with zero air to 20 psig and reuse.

To prepare the LCS: Follow the instructions provided in Appendix B for preparation of the working ICV/LCS standard. If an LCSD and replicate precision is requested, the aliquot for the LCSD must be taken from the LCS canister. If an LCSD and duplicate precision is requested, prepare another LCS in a separate canister to serve as the LCSD.

10.4 Sample Analysis

10.4.1 Instrument Operating Conditions

Optimize the GC and MS conditions for compound separation and sensitivity.

The recommended operating conditions are as follows:

Thermal Desorb:	Initial Trap #1 Temperature: -110ºC Desorb Temperature from Trap #1 to #2: 0 ºC Total Volume Transfer by Mass Flow Controller: 40 mL Initial Trap #2 Temperature: -15 ºC Desorb Temperature from Trap #2 to #3: 200ºC Transfer time 3.5 minutes Initial Trap #3 Temperature: -165 ºC Injection Trap #3 Temperature: 70ºC Injection Time: 1.5 minutes Trap #3 Temperature after Injection: -165 ºC
Carrier Gas:	Helium, Ultra High Purity
Cryogenic Focusing Gas:	Liquid Nitrogen
Flow Rate:	~1.5 mL/min
Temperature Program:	Initial Temperature: 40ºC Initial Hold Time: 4 minutes Ramp1 Rate: 20ºC/min. to 200ºC. Ramp 2 Rate: 40ºC/min. to 220ºC Final Temperature: 220ºC Final Hold Time: 6.5 minutes
Electron Energy:	70 electron volts
Mass Range:	35-265 amu
Scan Time:	≥1 scan per second

These operating conditions may be changed but once the operating conditions are established for initial calibration the same conditions must be used until a new calibration is performed.

10.4.2 Analytical Sequence

An example analytical sequence that includes initial calibration (ICAL) is provided below. When ICAL is not performed, the sequence begins with the tune standard and is followed by the CCV, LCS, LCSD, and method blank. If sufficient time remains in the 24 hours analytical window after initial calibration, QC and field samples may be analyzed without the CCV and the ICV will serve as the LCS for the sequence. The MB, LCS and LSCD must be analyzed at a frequency of every 20 samples or with each analytical sequence whichever is more frequent.

1. Tune Standard (BFB)
2. ICAL
3. ICV
4. CCV
5. LCS (repeat every 20 samples)
6. LCSD (when requested)
7. MB (repeat every 20 samples)
8. Field Samples (including trip blanks)

Attach the canisters to the autosampler inlet in the order of the analytical sequence then initiate the analytical sequence. The autosampler introduces 200 mL of sample volume from each canister to the instrument system and adds 20 mL of the mixed gas standard to each sample.

Acquire the data and evaluate the results to confirm qualitative identification and quantification.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

The data processing system tentatively identifies target analytes by comparing the retention time of the peaks to the window set around the continuing calibration standard, and searches in that area for the primary ion and up to two secondary ions characteristic of the target analyte.

All tentative identifications made by the computer are reviewed and either accepted or rejected by the primary analyst. The identification made by the system is accepted when the following criteria are met:

- The target analyte is identified by comparison of its background subtracted mass spectrum to a reference spectrum in the user-created database. In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and their relative abundances should agree within 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance if a tentative identification is to be made, and should be evaluated even if they are below 10% relative abundance.

- The GC retention time for the target analyte should be within 0.06 RRT units of the daily standard.

Identification requires expert judgment when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components. When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP BR-QA-006 *Manual Integration*.

11.2 Quantification of Target Analytes

After a compound has been identified, the data system quantifies the on-column concentration of the target compound based on the integrated abundance of the characteristic ion from the EICP. If there is matrix interference with the primary ion, a secondary ion may be used for quantification by calculating a mean RF factor for that ion and using that ion to quantify the analyte in the sample. When secondary ion calculations are required, include this information in the non-conformance report and project narrative.

Final results are calculated in TALS.

11.3 Calculations

Analytical results are calculated as follows:

- **Dilution Factor**

$$DF = \frac{V_2}{V_1} x \frac{V_4}{V_3}$$

Where:
V_1 = Pre-Dilution Canister Volume
V_2 = Post-Dilution Canister Volume
V_3 = Sample Amount (mL)
V_4 = Base Sample Amount (200 mL)

- **Relative Response Factor (RRF)**

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

Where:
A_x = Area of the quantitation ion of the analyte
A_{is} = Area of the quantitation ion of the internal standard

C_x = Concentration of analyte in concentration units (ppbv)
C_{is} = Concentration of internal standard in concentration units (ppbv)

- **Percent Relative Standard Deviation (%RSD)**

$$\%RSD = \frac{SD}{Mean} x100$$

Where:
SD = Standard deviation individual response factors
Mean = Average of five response factors

- **Sample Concentration**

$$C_x = \frac{(A_x)(C_{IS})}{(A_{IS})(\overline{RRF})}(DF)$$

Where:
C_x = Compound concentration (ppbv)
C_{IS} = Concentration of associated internal standard (ppbv)
A_{IS} = Area of quantitation ion for associated internal standard
A_x = Area of quantitation ion for compound
DF = Dilution Factor
Mean RRF = Average Relative Response Factor from initial calibration.

- **Unit Conversion from ppbv to ugm3**

$$\text{Analytical Result (ug/m3)} = \text{Result(ppbv)} \times \left(\frac{mw}{24.45}\right)$$

Where:
mw = molecular Weight

Example:
Benzene Result = 56 ppbv
Benzene mw = 78.108

$$\text{Analytical Result (ug/m3)} = 56\ \text{ppbv} \times \left(\frac{78.108}{24.45}\right)$$

Result(ug/m3) = 178.9 ug/m3 reported as 180 ug/m3

- **Percent Recovery (%R)**

$$\%R = \frac{C_s}{C_n} \times 100\%$$

Where:
C_s = Concentration of the spiked sample (ppbv)
C_n = Nominal concentration of spike added (ppbv)

- **Precision (%RPD)**

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

Where:
C_1 = Measured concentration of the first sample aliquot
C_2 = Measured concentration of the second sample aliquot

11.4 Data Review

11.4.1 Primary Review (Performed by Primary Analyst)

Upload the data files to TALS. Enter batch editor information and add the standards and reagents to the TALS batch. Review the results against acceptance criteria. If acceptance criteria are not met, make arrangements to perform corrective action.

Check the results of samples analyzed immediately after high concentration samples for signs of carry-over. Reanalyze the sample if carry over is suspected.

Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should result in a determination within the upper half of the calibration curve.

Set results to primary, secondary, acceptable or rejected as appropriate.

Verify corrective action was taken for all results not within acceptance criteria. If corrective action is not taken or was unsuccessful, record all instances where criteria are not met with a nonconformance memo (NCM). Be sure to provide explanation of your decision making in the internal comment section of the NCM. The internal comment section should list the reason the NCM is suspected, which action (if any) was taken and why and the outcome of the action taken.

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Set the batch to 1st level review.

Record your review on the data review checklist.

11.4.2 Secondary Review (Performed by Peer Reviewer)

Review the project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify project requirements

were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Review the TALS batch editor to verify information is complete. Review the batch to verify that the procedures in this SOP were followed. If discrepancy is found, resolve the discrepancy and verify any modifications to the SOP are were approved and are properly documented.

Spot-check 15% of samples in the batch to verify quantitative and qualitative identification.

If manual integrations were performed:

- Review each manual integration to verify that the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-005.

- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in laboratory SOP BR-QA-005.

- Generate a “before” and “after” chromatogram for every manual integration performed on an instrument performance check standard (Tune, ICAL, ICV, CCV), QC sample (MB, LCS) and for any manual integration performed on any surrogate or internal standard in any field sample.

- Generate the Manual Integration Summary Report. Document your review of manual integrations on the summary report and obtain any review signatures of integrations performed during secondary review as required.

  If the reviewer disagrees with the integration performed by the primary analyst, the secondary data reviewer should not change the integration. Instead, he/she should consult with the primary analyst that performed the integration and both the reviewer and the primary analyst should agree the integration should be changed. If consensus between the primary analyst and the peer reviewer cannot be achieved; both should consult with the Technical Manager or department management for resolution. Any changes to the integration should be performed by the primary analyst. If it is necessary for the secondary reviewer to perform the manual integration because the primary analyst is out of the office; the integration made by the peer reviewer must be reviewed by another peer reviewer or by department management to verify the integration was performed and documented in compliance to SOP BR-QA-005. If the original analyst that performed the integration is out of the office, the data reviewer may consult with the Department Manager (DM), Department Supervisor (DS) or the Technical Manager (TM) to verify the change he/she thinks is needed is warranted and should be made.

Verify that the performance criteria for the QC items listed in Table 1 were met. If the results do not fall within the established limits verify that corrective actions were performed. If corrective action was not performed; verify the reason is provided and that the situation is properly documented with an NCM. Set samples to 2nd level review.

Run the QC checker and fix any problems found. Run and review the deliverable. Fix any problems found. When complete set the method chain to lab complete and forward any paperwork to report/project management.

Record second level review on the data review checklist.

11.5 Data Reporting

Data reporting and creation of the data deliverable is performed by TALS using the formatters set by the project manager during project initiation.

Electronic and hardcopy data are maintained as described in laboratory SOP BR-QA-014 Laboratory Records.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Perform a method detection limit (MDL) study at initial method set-up following the procedures specified in laboratory SOP BR-QA-005,

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when there is a significant change in instrumentation or procedure.

Each analyst that performs the analytical procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP for employee training.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*.

The following waste streams are produced when this method is carried out:

- None

15.0 References / Cross-References

- EPA Compendium Method TO-15, "Determination of Volatile Organic Compounds in Ambient Air using Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry", US EPA, January, 1999.
- Laboratory SOP BR-QA-005, Procedures for the Determination of Limits of Detection (LOD), Limits of Quantitation (LOQ) and Reporting Limits (RL).
- Laboratory SOP BR-QA-011 Employee Training
- Laboratory SOP BR-EH-001 Hazardous Waste
- Laboratory SOP BR-QA-014 Laboratory Records
- LaboratorySOP BR-QA-006 Procedures & Documentation Requirements for Manual Integration
- Laboratory Quality Assurance Manual (QAM)

16.0 Method Modifications

Not Applicable.

17.0 Attachments

- Table 1: Target Compound List, RL, Internal Standard and Ion Assignments
- Table 2: Ion Abundance Criteria (BFB)
- Table 3: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables

18.0 Revision History

BR-AT-004r8, Revision 8:

- Title Page: Updated approval signatures
- All sections: Added procedure for ethanol
- Section 10: Changed calibration groups

Revision 7:
Section 11.4.1: Expanded on discussion of carry over.
Appendix C: Removed.
Appendix D: Removed.
Appendix E: Removed.

Table 1: Routine Compound List, Reporting Limit, Internal Standard and Ion Assignments

Analyte	CAS No.	6L RL (ppbv)	1L RL (ppbv)	Quantifier Mass	Qualifier Mass	Qualifier Mass	ISTD Group	Analyte Group
Dichlorodifluoromethane	75-71-8	0.5	5	85	87		1	B
Freon-22	75-45-6	0.5	5	51	67	69	1	B
1,2-Dichlorotetrafluoroethane	76-14-2	0.2	2	85	135	87	1	A
Chloromethane	74-87-3	0.5	5	50	52		1	B
n-Butane	106-97-8	0.5	5	43	41	58	1	B
Vinyl Chloride	75-01-4	0.04	0.40	62	64		1	D
1,3-Butadiene	106-99-0	0.5	5	54			1	B
Bromomethane	74-83-9	0.2	2	94	96		1	A
Chloroethane	75-00-3	0.5	5	64	66		1	B
Isopentane	78-78-4	0.2	2	43	57	56	1	A
Bromoethene (Vinyl Bromide)	593-60-2	0.2	2	106	108	81	1	A
Trichlorofluoromethane	75-69-4	0.2	2	101	103		1	A
Pentane	109-66-0	0.5	5	43	57	72	1	B
EthylEther	60-29-7	0.2	2	59	45	74	1	A
Acrolein	107-02-8	5	50	56	55	37	1	C
Freon TF	76-13-1	0.2	2	101	151	103	1	A
1,1-Dichloroethene	75-35-4	0.2	2	96	61	63	1	A
Acetone	67-64-1	5	50	43	58		1	C
Isopropyl Alcohol	67-63-0	5	50	45	43		1	C
Carbon Disulfide	75-15-0	0.5	5	76			1	B
3-Chloropropene (Allyl Chloride)	107-05-1	0.5	5	41	76		1	B
Acetonitrile	75-05-8	5	50	41	40	39	1	C
Methylene Chloride	75-09-2	0.5	5	49	84	86	1	B
tert-Butyl Alcohol	75-65-0	5	50	59	41	43	1	C
Methyl tert-Butyl Ether	1634-04-4	0.5	5	73	43		1	B
trans-1,2-Dichloroethene	156-60-5	0.2	2	61	96		1	A
n-Hexane	110-54-3	0.5	5	57	86		1	B
1,1-Dichloroethane	75-34-3	0.2	2	63	65	83	1	A
Methyl Ethyl Ketone	78-93-3	0.5	5	72	43		1	B
cis-1,2-Dichloroethene	156-59-2	0.2	2	96	98		1	A
Tetrahydrofuran	109-99-9	5	50	42	72		2	C
Chloroform	67-66-3	0.2	2	83	85		1	A
1,1,1-Trichloroethane	71-55-6	0.2	2	97	99	61	2	A
Cyclohexane	110-82-7	0.2	2	84	56		2	A
Carbon Tetrachloride	56-23-5	0.2	2	117	119		2	A
2,2,4-Trimethylpentane	540-84-1	0.2	2	57	41	43	2	A
1,2-Dichloroethene (total)	540-59-0	0.2	2	61	96		1	A
Benzene	71-43-2	0.2	2	78	77		2	A
1,2-Dichloroethane	107-06-2	0.2	2	62	98		2	A
n-Heptane	142-82-5	0.2	2	43	71		2	A
Trichloroethene	79-01-6	0.04	0.40	95	130	132	2	D
Methyl Methacrylate	80-62-6	0.5	5	69	41	39	2	B
1,2-Dichloropropane	78-87-5	0.2	2	63	41		2	A
1,4-Dioxane	123-91-1	5	50	88	58		2	C

Analyte	CAS No.	6L RL (ppbv)	1L RL (ppbv)	Quantifier Mass	Qualifier Mass	Qualifier Mass	ISTD Group	Analyte Group
Dibromomethane	74-95-3	0.2	2	174	93	172	2	A
Bromodichloromethane	75-27-4	0.2	2	83	85		2	A
cis-1,3-Dichloropropene	10061-01-5	0.2	2	75	110		2	A
Methyl Isobutyl Ketone	108-10-1	0.5	5	43	58		2	B
n-Octane	111-65-9	0.2	2	43	57	114	2	A
Toluene	108-88-3	0.2	2	92	91		3	A
trans-1,3-Dichloropropene	10061-02-6	0.2	2	75	110		2	A
1,1,2-Trichloroethane	79-00-5	0.2	2	83	97	85	3	A
Tetrachloroethene	127-18-4	0.04	0.40	166	168	129	3	D
Methyl Butyl Ketone	591-78-6	0.5	5	43	58		3	B
Dibromochloromethane	124-48-1	0.2	2	129	127		3	A
1,2-Dibromoethane	106-93-4	0.2	2	107	109		3	A
Nonane	111-84-2	0.2	2	57	71	128	3	A
Chlorobenzene	108-90-7	0.2	2	112	77	114	3	A
Ethylbenzene	100-41-4	0.2	2	91	106		3	A
Xylene (m,p)	1330-20-7	0.5	5	106	91		3	A
Xylene (o)	95-47-6	0.2	2	106	91		3	A
Styrene	100-42-5	0.2	2	104	78		3	A
Bromoform	75-25-2	0.2	2	173	175	171	3	A
Cumene	98-82-8	0.2	2	105	120	77	3	A
1,1,2,2-Tetrachloroethane	79-34-5	0.2	2	83	131	85	3	A
Xylene (total)	1330-20-7	0.2	2	106	91		3	A
n-Decane	124-18-5	0.5	5	57	71	142	3	B
n-Propylbenzene	103-65-1	0.2	2	91	120	92	3	A
1,2,3-Trichoropropane	96-18-4	0.5	5	75	110	112	3	B
4-Ethyltoluene	622-96-8	0.2	2	105	120		3	A
1,3,5-Trimethylbenzene	108-67-8	0.2	2	105	120		3	A
2-Chlorotoluene	95-49-8	0.2	2	91	63		3	A
tert-Butylbenzene	98-06-6	0.2	2	119	91	134	3	A
1,2,4-Trimethylbenzene	95-63-6	0.2	2	105	120		3	A
sec-Butylbenzene	135-98-8	0.2	2	105	134	91	3	A
4-Isopropyltoluene	99-87-6	0.2	2	119	134	91	3	A
1,3-Dichlorobenzene	541-73-1	0.2	2	146	111	148	3	A
1,4-Dichlorobenzene	106-46-7	0.2	2	146	111	148	3	A
n-Undecane	1120-21-4	5	50	57	71	156	3	C
Benzyl Chloride	100-44-7	0.2	2	91	126	65	3	A
n-Butylbenzene	104-51-8	0.2	2	91	134	92	3	A
1,2-Dichlorobenzene	95-50-1	0.2	2	146	111	148	3	A
n-Dodecane	112-40-3	5	50	57	71	170	3	C
1,2,4-Trichlorobenzene	120-82-1	0.5	5	180	182		3	B
1,3-Hexachlorobutadiene	87-68-3	0.2	2	225	223		3	A
Naphthalene	91-20-3	0.5	5	128			3	B
1,2,3-Trichlorobenzene	87-61-6	0.2	2	180	182	145	3	A
Propylene	115-07-1	5	50	41	42	39	1	C
Vinyl Acetate	108-05-4	5	50	43	86		1	C

Analyte	CAS No.	6L RL (ppbv)	1L RL (ppbv)	Quantifier Mass	Qualifier Mass	Qualifier Mass	ISTD Group	Analyte Group
Ethyl Acetate	141-78-6	5	50	43	74		1	C
Ethanol	64-17-5	5	50	46	45		1	E
Bromochloromethane	74-97-5	NA	NA	128	49	130	1	NA
1,4-Difluorobenzene	540-36-3	NA	NA	114			2	NA
Chlorobenzene-d5	3114-55-4	NA	NA	117			3	NA

Table 2: Tune Standard Criteria

Mass	Ion Abundance Criteria
50	8.0 to 40.0 percent of mass 95
75	30.0 to 66.0 percent of mass 95
95	Base Peak, 100 percent relative abundance
96	5.0 to 9.0 percent of mass 95
173	Less than 2.0 percent of mass 174
174	50.0 to 120.0 percent of mass 95
175	4.0 to 9.0 percent of mass 174
176	93.0 to 101.0 percent of mass 174
177	5.0 to 9.0 percent of mass 176

Table 3: TO15 QC Summary & Recommended Corrective Action

QC Check	Frequency	Acceptance Critera	Recommended Corrective Action
Tune Standard	Prior to calibration and every 24 hours	See Table 2	Correct Problem. Reanalyze. No sa without a valid tune.
ICAL	Prior to sample analysis and when CCV fails	RSD for each analyte ≤ 30% with 2 exceptions up to 40%	Correct problem and repeat calibrati
ICV	Once after each ICAL	%R for all analytes within 70-130	Correct Problem. Reanalyze, re-ma analyze. If that fails, re-make all sta calibration.
Retention Time Window	Once per ICAL	NA	NA
RRT	With each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct Problem. Repeat ICAL
CCV	Daily before sample analysis after tune standard	%D ≤ 30	Correct Problem. Reanalyze once. 10.2.5 for instruction.
LCS	Each batch or every 20 samples, whichever is sooner.	%R for all analytes within 70-130	Reanalyze LCS, re-prep and reanaly associated samples if sufficient sam If corrective action not successful, in report and qualify sample results.
LCSD	Per Client Request	RPD ≤ 25	Reanalyze LCSD, re-prep and reana associated samples if sufficient sam If corrective action is not successful, report and qualify sample results.
Method Blank	Each batch or every 20 samples, whichever is sooner.	No analytes detected above RL	Reanalyze along with associated sa same compounds found in blank are concentration found in the blank.
Internal Standard	All standards, field and QC samples	+/- 40% area response from last acceptable calibration. RT +/- 0.33 min (20 seconds) from last acceptable calibration.	Inspect system for malfunction. Rea data.
Sample Duplicate	Per Client Request	RPD ≤ 25	Consult with PM. Reanalyze or qual

Appendix A: Terms and Definitions

Acceptance Criteria: Specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: Environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): An analytical standard gas mixture containing all target analytes and internal standard compounds that is used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Cryogen: A refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp -195.8°C) or liquid argon (bp -185.7°C).

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): An analytical standard mixture containing all target analytes and internal standard compounds that are prepared from a source independent of the source of the initial calibration standards. The purpose of the ICV is to verify that the initial calibration is in control.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Internal Standards (IS): Non-target analytes that are similar to the target analytes but are not expected to be found in environmental media (generally, isotopically labeled target analytes are used for this purpose). IS are added to every standard, quality control sample, and field sample at a known concentration prior to analysis. IS responses are used as the basis for quantitation of target analytes.

Laboratory Control Sample (LCS) – A QC sample of known composition spiked with analytes of interest. The LCS evaluates method performance and ability to successfully recover target analytes from a clean matrix. LCS recovery is typically expressed as percent recovery and provides a measure of accuracy. A LCSD is a duplicate LCS prepared and analyzed from a separate canister to provide a measure of replicate precision.

Method Blank (MB): A canister of humidified ultra pure zero air that is treated exactly as a sample. The MBLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Gas Mixture: A Commercially purchased concentrated gas mixture containing one or more method analytes

Appendix B: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance on the preparation of standard solutions.

Prepare all standards using the McMillan Company 80SD mass flow controller. Prepare the standard in zero air, demonstrated to be analyte free. Store the standard at ambient temperature. Unless otherwise specified, assign an expiration date of 30 days from date of preparation unless the parent standard expires earlier, in which case, use the earliest expiration date.

Intermediate Calibration Standard

Parent Standard	Vendor	Stock Standard Concentration (ppmv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Custom Calibration Stock Standard	Spectra Gases Custom Made	1.0	7500	37.5	200

Prepare in 15 L Summa Canister Expiration Period 3months
This standard contains all the target analytes listed in table 1.

Working Calibration Standards

Parent Standard	Calibration Standard	Parent Standard Concentration (ppbv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Cal Standard 20 ppbv	Cal Standard 0.2 ppbv	20	155	15.46	0.2
Cal Standard 20 ppbv	Cal Standard 0.5 ppbv	20	386	15.46	0.5
Intermediate Calibration Standard	Cal Standard 5 ppbv	200	386	15.46	5
Intermediate Calibration Standard	Cal Standard 10 ppbv	200	773	15.46	10
Intermediate Calibration Standard	Cal Standard 15 ppbv	200	1160	15.46	15
Intermediate Calibration Standard	Cal Standard 20 ppbv	200	1546	15.46	20
Intermediate Calibration Standard	Cal Standard 40 ppbv	200	3092	15.46	40

Prepare in 6 L Summa Canister Expiration Period 3 months
Each calibration standard contains all the analytes listed in table 1 at the above concentrations.

Initial Calibration Levels

Calibration Level	Working Calibration Standard	Volume Analyzed (mL)	Concentration on Column (ppbv)
Calibration Level 1	Cal Standard 0.2 ppbv	200	0.2
Calibration Level 2	Cal Standard 0.5 ppbv	200	0.5
Calibration Level 3	Cal Standard 5 ppbv	200	5
Calibration Level 4	Cal Standard 10 ppbv	200	10
Calibration Level 5	Cal Standard 15 ppbv	200	15
Calibration Level 6	Cal Standard 20 ppbv	200	20
Calibration Level 7	Cal Standard 40 ppbv	200	40
Calibration Level 8	Cal Standard 0.2 ppbv	40	0.04

Prepare in 6L Summa Canister

Intermediate ICV/LCS Standard

Parent Standard	Vendor	Stock Standard Concentration (ppmv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)

ICV Stock Standard	Spectra Gases Custom Made	1.0	7500	37.5	200

Prepare in 15L Summa Canister Expiration period 3 months
This standard contains all target analytes listed in table 1.

Working ICV/LCS Standard

Parent Standard	Calibration Standard	Stock Standard Concentration (ppbv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Intermediate ICV/LCS Standard	ICV Standard 10 ppbv	200	773	15.46	10

Prepare in 6L Summa Canister Expiration period 3 months
This standard contains all target analytes listed in table 1.

Intermediate Ethanol Calibration Standard at 500ppbv/v

1) Fill a 44 ml VOA vial with VOA free water. Remove 197ul of water from the vial.
2) Add 197 ul of >99.5% Ethanol neat material
3) Cap and shake/roll vial for 1 minute
4) Inject 10ul of the prepared water/ethanol mix into a fully evacuated 15 liter summa canister
5) Pump the syringe plunger 5 times to insure complete transfer of material
6) Immediately fill the canister to 22 psig with zero air.

Calibration Level	Working Calibration Standard	Volume added (mL)	Concentration on Column (ppbv)
Calibration Level 1	Cal Standard 0.5 ppbv	124	5
Calibration Level 2	Cal Standard 5.0 ppbv	309	10
Calibration Level 3	Cal Standard 10ppbv	464	15
Calibration Level 4	Cal Standard 15 ppbv	618	20
Calibration Level 5	Cal Standard 20 ppbv	1237	40
Calibration Level 6	Cal Standard 40 ppbv	3092	100

Title: HOMOGENIZATION OF BIOTA & TISSUE

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Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure for the whole body homogenization and extraction of biota and tissue samples by tissuemizer in preparation for analysis by a variety of chromatographic procedures.

NOTE: Pre-preparation of tissue such as dissection, sportsman filet, sample compositing, shucking, etc. may be performed by the laboratory per customer specification. These procedures are project specific and instructions are not included in this SOP.

2.0 Summary of Method

2.1 Homogenization

Tissue samples are homogenized using a titanium blade homogenizer. Biota samples are homogenized using stainless steel knives and/or a food processor. The homogenized sample(s) are transferred to labeled glass jars and stored in a freezer maintained at a temperature of -15ºC (±5ºC) in preparation for extraction.

The laboratory's standard procedure is to perform whole body homogenization of the tissue sample. Any customer specifications for dissection or homogenization of certain parts of the tissue sample, compositing multiple samples or other must be negotiated with the laboratory during project initiation and specific instructions for sample processing must be prepared and provided to the extraction laboratory by the laboratory PM. In the absence of instruction, the laboratory will homogenize the entire sample.

The homogenization procedure is a laboratory developed procedure based on the procedures described in the Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, National Status and Trends Program for Marine Environmental Quality.

2.2 Extraction

A portion of homogenized sample is mixed with anhydrous sodium sulfate then macerated for 3 minutes in an appropriate extraction solvent using the Tissumizer. The solvent layer decanted poured through sodium sulfated and collected in a collection vessel. The extraction is repeated two more times with fresh portions of extraction solvent. After extraction, the combined extracts are concentrated to an appropriate final volume using K-D Technique. Percent lipids are determined following procedures given in laboratory SOP BR-EX-016 Percent Lipid Determination and extract cleanup is performed when necessary.

The extractionprocedure is a laboratory developed procedure based on the procedures described in the Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, National Status and Trends Program for Marine Environmental Quality.

3.0 Definitions

- Biota: flora and fauna. For this SOP, all reference to "biota" refers to plant material.

- Tissue: an aggregate of cells usually of a particular kind together with their intercellular substance that form one of the structural materials of a plant or animal. For this SOP, all reference to "tissue" refers to structural materials from an animal.

A list of general terms and definitions are provided in Appendix A.

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference. All glassware must be cleaned in accordance with laboratory SOP BR-EX-017 Glassware Cleaning, and rinsed with acetone and methylene chloride prior to use.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Homogenization Equipment

- Cutting Board- High density polyethylene 16X23"

- Homogenizer equipped with 55 mm Titanium Blade Omni International or equivalent.
- Food Processor Cuisinart or equivalent
- Stainless steel knives
- Glass Jars, wide mouth; 125 mL-1000 mL. ESS or equivalent.
- Meat Grinder

6.2 Extraction Equipment

- Tissuemizer equipped with a 20 mm x 195 Generator probe. Omni International PowerGen 700 or equivalent.
- Filter Funnels – 100 mm diameter for filtration/drying. Fisher Scientific or equivalent.
- No. 54 Filter paper. Whatman 18.5 cm, or equivalent.
- Beakers – 400 mL. Fisher Scientific or equivalent.

6.3 Extract Concentration (KD Apparatus)

- Concentrator Tube, 10 mL Graduated: ChemGlass Catalog # CG-1316-11 or equivalent
- Snyder Column: Three ball macro, AMK Catalog # SC2-01 or equivalent
- Snyder Column: Two ball micro, AMK Catalog # SC3-01 or equivalent
- Evaporation Flask: 500 mL attached to concentrator tube with clip, AMK Catalog Number KDF-500 or equivalent.
- Boiling Chips: Silicon carbide, approximately 10/40 mesh, solvent extracted in methylene chloride, Troemner Catalog # 133B or equivalent.
- Heating Mantle: Rheostat controlled for water bath capable of temperature control (±5°C). ChemGlass Catalog # PL3122 or equivalent.
- Water Bath, capable of temperature control to ±5°C. Barnstead Corporation Catalog # HM0500-HS1 or equivalent.
- Solvent Vapor Recovery System, Kontes K-54000-1006, K-547300-000, Ace Glass Catalog # 6614-30 or equivalent.

6.4 Miscellaneous

- Disposable Glass Pasteur Pipette and bulb: Fisher Scientific or equivalent.
- Top Loading balance: Capable of measuring to 0.01 gram accuracy, Mettler Model # PM4800 or equivalent.
- Vials and caps: 2, 4, 8, and 16 mL with Teflon lined septa and screw caps, Fisher Scientific or equivalent.
- Teflon and Stainless Steel Spatulas, Fisher Scientific or equivalent.
- Adjustable Pipette: Finnpipette or equivalent
- 0.5 mL – 2.0 mL Hamilton Gastight® syringes or equivalent.
- Paper towels

7.0 Reagents and Standards

7.1 Reagents

- Sodium Sulfate (Na_2SO_4), Granular Anhydrous: J.T. Baker or equivalent. Purify by heating at 400°C for at least 4 hours.
- Methylene Chloride (CH_2C_{12}): Pesticide Quality, J.T Baker or equivalent.

- Hexane, (C_6H_{14}): Pesticide Quality, J.T. Baker or equivalent.
- Acetone, ($(CH_3)_2CO$): Pesticide Quality, J.T. Baker or equivalent.
- Reagent Water: RO water filtered through a Nanopure System.
- Alkaline Liquid Detergent: Contrex or equivalent.

Methylene Chloride/Acetone (1:1): In a 4 L amber glass bottle mix 2 L methylene chloride with 2 L acetone. Store the solution in a fume hood. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

Hexane /Acetone (1:1): In a 4 L amber glass bottle mix 2 L hexane with 2 L acetone. Store the solution in a fume hood. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

7.2 Standards

Purchase certified stock standards from commercial vendors and from these prepare surrogate and spiking solutions by diluting a known volume of the stock standard solutions in an appropriate solvent. Record the preparation of all standards in the LIMS module. The formulations for the preparation of surrogate and spiking standards are provided in analytical SOPs.

Unless otherwise specified in the analytical SOP store prepared in glass containers at 4°C or below and assign an expiration date of 6 months from the date of preparation unless the parent standards expire earlier in which case use the earliest expiration date.

Assay surrogate and spike solutions before each use to verify the made to concentration is within specifications. Maintain records of the assay per the procedure established for the work section.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP.

The laboratory recommends that tissue and biota samples be collected in glass jars or sealable plastic bags. Immediately following collection, biota samples should be iced to a temperature of 4°C (±2°C) and tissue samples should be frozen and maintained at a temperature of -15°C (±5°C) until the time of homogenization. After homogenization, all homogenized samples must be stored in a freezer maintained at a temperature of -15°C (±5°C).

Tissue and biota samples should be extracted within 14 days after the sample was thawed or removed from frozen storage. The remaining sample should be returned immediately to the freezer after extraction. As long as the samples are frozen, a six-month holding time will apply.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Analytical SOP
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Analytical SOP
Matrix Spike(s) MS/MSD	With every batch if sufficient volume is available	See Analytical SOP
Sample Duplicate (SD)	Client Request	See Analytical SOP

10.0 Procedure

10.1 Instrument Calibration

Check the calibration of the balance each day of use prior to use.

Check the calibration of the adjustable pipettes quarterly.

Perform periodic maintenance on the tissuemizer's generator probe as necessary. Maintenance may include but is not limited to the replacement of Teflon bearings and rotor shafts when a loud squealing noise is heard. Refer to the PowerGen 700 Homogenizer Instruction Manual for further guidance and for the manufacturer's recommended maintenance program.

10.2 Whole Body Homogenization of Tissue Samples

Remove the samples from storage and let the sample(s) thaw completely.

Wash all equipment with detergent and hot water, then rinse with reagent water and acetone prior to use and also after each sample.

Prepare a equipment blank to ensure the equipment is clean. To prepare the blank, transfer a representative amount of reagent water (similar to the size of the samples) to a clear glass jar. Let the water come into contact with all equipment that will be used to homogenize the samples. This process should be done randomly during homogenization to verify the cleaning process.

If possible obtain a glass jar large enough to accommodate the entire sample. Label the jar with the sample's lab ID and place the jar on the analytical balance. Tare the balance. Put on a pair of nitrile gloves and with your hands transfer the sample from the storage container to the labeled jar. Record the pre-homogenization weight of the sample in the worksheet.

Transfer the sample to a pre-cleaned cutting board. Cut the sample into 1-3" sections using a stainless steel knife then place the sections of samples in the labeled jar.

Insert the titanium blade into the jar and homogenize the sample at 2000-4000 RPM until the sample becomes slurry. Manual mixing with a stainless steel or Teflon spatula may be required to insure complete homogenization. Remove the blade from the sample jar and scrape any remaining sample from the blade into the labeled jar. Place the jar on the top-loading balance and record the post homogenization weight in the worksheet.

NOTE: Samples greater than 12 inches or samples with weight measurements that exceed 1-2 pounds should be homogenized in a stainless steel meat grinder prior to use of the titanium

blade. If the sample is extremely large homogenization with a knife may be necessary. If the homogenized tissue sample cannot fit into a single container, pass the sample through the meat grinder multiple times, homogenize the slurry and transfer to multiple containers.

10.3 Homogenization of Biota Samples

Wash all equipment with detergent and hot water, then rinse with reagent water and acetone prior to use and also after each sample.

Prepare a equipment blank to ensure the equipment is clean. To prepare the blank, transfer a representative amount of reagent water (similar to the size of the samples) to a clear glass jar. Let the water come into contact with all equipment that will be used to homogenize the samples. This process should be done randomly during homogenization to verify the cleaning process.

Remove the samples from storage and let warm to ambient temperature. Select a glass jar large enough to accommodate the entire sample. Label the jar with the sample's lab ID and place the jar on the top-loading balance. Tare the balance. Remove the biota sample from the storage container and place in the labeled jar. Record the pre-homogenization weight of the sample into the worksheet.

Remove the sample and place on a pre-cleaned cutting board. Slice the material into very fine sections using a stainless steel knife or a food processor. Return homogenized sample back into the jar and place on the top-loading balance. Record the post homogenization weight into the worksheet.

10.4 Extraction

Clean all glassware prior to use following the procedure given in laboratory SOP BR-EX-017. Label all glassware with field and QC samples ID numbers clearly and unambiguously during each step of the extraction procedure. Solvents will erase grease pens and "sharpie ink", so caution must be taken to ensure that the labels are not obliterated during the procedure.

Assemble a KD apparatus set-up and prepare a glass funnel for each sample to be extracted. Fold a 185 mm Whatman® 54 filter into quarters and place a filter in each funnel. Fill each funnel ~3/4 full with purified granular anhydrous sodium sulfate. Rinse the funnel with ~30 mL acetone and methylene chloride each and discard the solvent rinse. Place a prepared funnel onto each K-D setup.

Assemble the Tissuemizer by attaching the 20 mm x 195 mm-generator probe to the Tissumizer motor. Place the Tissuemizer in the fume hood and attach to the aluminum staging using clamps. Clean the Tissuemizer prior to use by running the generator probe for 10 seconds in a 400 mL beaker filled with ~ 200 mL of reagent water. Discard the reagent water and repeat with another aliquot of reagent water. Repeat the cleaning step two more times each with ~250 mL of acetone.

Mix the sample using a stainless steel or Teflon spatula. Place a labeled 400 mL beaker onto the top-loading balance and depress the "tare" button. Referring to the extraction condition spreadsheet, weigh out the appropriate amount of sample and record sample weight $\pm$ 1 gram into TALS. Repeat for all samples. Transfer two additional aliquots of the sample selected for the MS and MSD into labeled 400 mL beakers. Transfer the same weight of sodium sulfate each

into labeled 400 mL beakers to serve as the method blank (MB) and laboratory control sample (LCS).

Add a sufficient volume of granular sodium sulfate to each sample and mix thoroughly with a stainless steel spatula until a free-flowing mixture is formed.

Add the appropriate volume of surrogate spike to each field sample and QC sample. Add the appropriate volume of spike solution to the laboratory control samples and the MS/MSD.

Add 100 mL of the appropriate extraction solvent to each beaker. Use 1:1 $MeCl_2$/Acetone for samples to be analyzed by GC/MS and 1:1 Hexane/Acetone for GC/ECD.

Refer to the Extractions Condition Worksheet to determine the type and amounts of solution added and the extraction solvent used for each test method.

Immerse the generator probe in the first sample beaker so that it is approximately ½" into the extraction solvent. Turn on the Tissuemizer. Adjust the speed on the motor until the solvent begins to vortex in the beaker, but does not splash out of the beaker. Extract the sample for 3 minutes. During extraction move the beaker in a circular motion to ensure that the entire sample is subject to extraction. Remove the beaker and decant the extraction solvent into the sample's corresponding funnel and K-D apparatus. Repeat the extraction 2 more times with ~100 mL of extraction solvent. After the 3rd extraction, pour the entire contents of the beaker into the funnel, rinse the beaker with more of the extraction solvent, and pour this into the funnel as well.

Rinse the funnel with ~30 mL of extraction solvent and allow the solvent to completely drain into the K-D apparatus. Remove the funnel from the K-D apparatus and discard the contents of the funnel. Clean the generator probe and repeat the extraction for each sample.

Concentrate the extracts following the procedure given in section 10.5 in preparation for percent lipids determination and extract cleanup. After concentration and prior to extract cleanup, set aside a 1 mL aliquot of the concentrated extract and determine the percent lipids following procedures given in laboratory SOP BR-EX-016 *Percent Lipids Determination.*

Perform extract cleanup as appropriate following procedures given in laboratory SOPs BR-EX-002 and BR-EX-011. Refer to extraction condition spreadsheet for details. After cleanup, concentrate the extracts following the procedure given in section 10.5.

Enter the extraction data into the TALS. Assemble any associated paperwork and submit the extracts to the supervisor for a final project check. After review is complete, relinquish the extracts to the appropriate analytical department and place in the refrigerated storage area.

Note: Immediately following concentration, all sample extracts must be stored in a refrigerator maintained at a temperature of 4ºC (±2°C) in order to maintain thermal preservation.

10.5 Extract Concentration (KD Apparatus)

10.5.1 Macro Concentration

Macro Snyder Column (K-D)

Add one or two clean boiling chips to the K-D evaporation flask and attach a three-ball Snyder column to the flask. Add ~1 mL of methylene chloride to the top of the column then place the K-D apparatus in a hot water bath (60-70°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot water vapor.

Attach the solvent vapor recovery glassware to the Snyder column. Adjust the vertical position of the apparatus and check the water bath temperature. The water bath temperature should be between 54.8 – 74.8°C when methylene chloride is the extraction solvent and 84-89°C when hexane is the extraction solvent. Higher water bath temperatures may be used so long as the recovery of target analytes is not impacted. The boiling point of each solvent is provided in the following table:

Solvent	Boiling Point	Water Bath Temperature
Hexane	69°C	84 – 89°C
Methylene Chloride	39.8°C	54.8 – 74.8°C

Monitor the concentration and do not let the extract evaporate to dryness. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with solvent.

When the apparent volume of the extract reaches desired amount remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

Micro Synder Column (K-D)

Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column to the tube. Place the concentrator tube into the water bath so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the concentrator tube and check the temperature of the water bath to ensure the proper temperature for the extract solvent.

Continuously monitor the distillation process to ensure sample extracts do not evaporate to dryness. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with solvent. Remove setup when desired sample volume is reached.

Nitrogen Blowdown

Nitrogen blow down may be used to concentrate extracts as needed.

Place the concentrator tube in a warm water bath maintained at a temperature of 35°C. Apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

11.0 Calculations / Data Reduction

11.1 Data Review

11.1.1 Primary Review

Review the TALS worksheet for correctness and completeness. Record any problems encountered during the extraction process into TALS or complete a NCM, when necessary. Set aside the extracts and paperwork for secondary review.

11.1.2 Secondary Review

Review the TALS worksheet against the extraction conditions spreadsheet and/or project notes to ensure the extraction performed is consistent with project specifications. Authorize release of the extracts to the appropriate analytical department.

For additional guidance regarding the laboratory's protocol and required elements for data review refer to laboratory SOP BR-QA -019 *Data Review*.

12.0 Method Performance

12.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ)

Refer to the SOP for the test method for requirements for determination of LOD and LOQ. These procedures are not included in sample preparation SOPs.

12.2 Demonstration of Capabilities (DOC)

Each analyst must complete an Initial Demonstration of Capability prior to unsupervised performance of this method.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Organic Solvents - Satellite container: 55 gallon covered and vented drum.
- Vials containing extracts - Satellite container: 5 gallon covered bucket in fume hood.
- Methylene Chloride-Waste-Satellite Container: 55 Gallon Waste Drum
- Sulfuric Acid Waste-Satellite Container: 2.5L Waste Bottle Labeled with appropriate acid type (sulfuric).

- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)

15.0 References / Cross-References

- Comprehensive Descriptions of Trace Organic Analytical Methods given in the Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, National Status and Trends Program for Marine Environmental Quality.

- GERG Trace Organic Contaminant Analytical Techniques published in NOAA Technical Memorandum NOS Orca 71, Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, Comprehensive Descriptions of Trace Organic Analytical Methods, July 1993.

- CW-E-M-001 *Corporate Environmental Health and Safety Manual*
- BR-EX-016 *Percent Lipid Determination*
- BR-EX-017 *Glassware Cleaning*
- BR-EX-002 *Extract Cleanup Procedure*
- BR-EX-011 *Gel-Permiation Cleanup*
- BR-QA -019 *Data Review*
- BR-QA-005 *Determination of LOD, LOQ, & RLs*
- BR-EH-001 *Hazardous Waste*

16.0 Method Modifications

Not applicable.

17.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions

18.0 Revision History

Revision 5, Effective Date 5/20/08:
- Title Page: Updated approval signatures.
- Section 6.0: Inserted vendor information
- Section 8.0: Inserted table
- Section 15.0: Added cross referenced methods with the SOP
- All Sections: Fixed typographical errors

Revision 6, Effective Date 05/20/10:
- Title Page: Updated approval signatures
- Section 6.1: Addition of Meat Grinder to equipment list
- Section 10.1: Changed pipette calibrations to be done quarterly
- All Sections: Fixed typographical errors
- All Sections: Changed benchsheets reference to extraction condition spreadsheet
- All Sections: Changed LIMS references to TALS

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Appendix A: Terms and Definitions

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Title: DETERMINATION OF PERCENT LIPIDS

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Approval Date: March 16, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

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1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of percent lipids in biological tissue.

2.0 Summary of Method

A 1 mL aliquot of extract is evaporated to dryness and weighed to a constant weight. The residual weight of the dried aliquot is used to calculate percent lipids.

This procedure is based on EPA SOP ASB P100, ASB E100 for the Determination of Organics in Fish and NOAA Technical Memorandum NOS ORCA 130 Method for the Determination of Percent Lipid in Tissue, Sampling and Analytical Methods of the National Status and Trends Program Mussel Watch Project: 1993-1996 Update.

The following exceptions to the NOAA reference method are noted:

- The laboratory does not include the dry weight determination of the tissue sample in the percent lipids calculation.

- The tissue sample may be extracted with hexane instead of dichloromethane when hexane is an appropriate extraction solvent for the determinative method.

- The percent lipids are determined from the extract using a gravimetric procedure without the additional filtering or concentration steps.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference. All glassware must be cleaned in accordance with laboratory SOP BR-EX-017 *Glassware Cleaning Procedure*, and rinsed with acetone and methylene chloride prior to use.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1 includes a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Extraction Equipment

- Aluminum Weigh Boat(s)
- Analytical Balance
- Adjustable Pipette(s)
- Drying Oven
- Stainless Steel Tongs

7.0 Reagents and Standards

7.1 Reagents

- Sodium Sulfate, granular anhydrous (Na_2SO_4): J.T. Baker or equivalent. Purify by heating at 400°C for at least 4 hours.

7.2 Standards

Not Applicable

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

- Sample extracts used for percent lipid determination must be protected from light and maintained at a temperature of 4(±2)°C.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Below
Sample Duplicate (SD)	1 in 20 or fewer samples	See Below

A method blank comprised of sodium sulfate is extracted with each batch of 20 or less samples and it is taken through the percent lipids determination in the same manner as sample extracts. The calculated percent lipids in the method blank should be <0.1%.

The relative percent difference (RPD) in the percent lipids determination between replicate samples should be less than or equal to 25%. If it is not, the original and duplicate sample extracts should be re-weighed. If the RPD is still not within specification, the percent lipids procedure should be repeated using new extracts.

9.2 Instrument QC

Not Applicable
.

10.0 Procedure

10.1 Instrument Calibration

Check the calibration of the balance each day of use prior to use.

Check the calibration of the adjustable pipettes each day of use prior to use.

10.2 Lipid Determination

Samples are extracted using an appropriate tissue extraction method. The samples are then concentrated to approximately 2 mL and filtered using a Whatman 0.45um Autovial Filter before concentration to 10.0 mL.

Tare the balance and weigh an aluminum boat to the nearest 0.0001 g and record the weight measurement on the Percent Lipid Benchsheet. Transfer 1 mL of the 10 mL extract (pre-cleanup) to the aluminum weigh boat. Using tongs transfer the weigh boat to a drying oven maintained at a temperature of 104˚C (±2°C) and allow sufficient time for the solvent to evaporate to dryness, approximately 1 hour. Allow the weigh boat/sample to cool to room temperature.

Re-weigh the weigh boat to the nearest 0.0001 g and record the weight measurement on the Percent Lipid Benchsheet.

Calculate the percent lipids using the equation given in Section 11.0.

Evaluate the results of the method blank and sample duplicate against the criteria given in Section 9.0 Quality Control and perform corrective action as necessary.

11.0 Calculations / Data Reduction

11.1 Calculations

Percent Lipid Calculation

$$\%Lipids = \frac{RW}{AV} \otimes \frac{EV}{WS} \otimes 100$$

Where:
RW = the residue weight *(residue + weigh boat)* minus the weight of the weigh boat
AV = the volume of the aliquot used (mL)
EV = the final extract volume (mL)
WS = the original weight of sample extracted (g)

Relative Percent Difference (RPD)

$$\frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \otimes 100$$

Where:
C_1 = Result of Parent Sample
C_2 = Result of Duplicate Sample

11.2 Data Review

Primary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Enter the batch information into LIMS and complete the batch editor and worksheet. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to 1st level review.

Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Not Applicable

12.2 Demonstration of Capabilities (DOC)

Not Applicable

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Organic Solvents - Satellite container: 55 gallon covered and vented drum.
- Vials containing extracts - Satellite container: 5 gallon covered bucket in fume hood.
- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)

15.0 References / Cross-References

- *SOP for the Determination of Organics in Fish.* SOP ID: ASB P100, Mod1.0 October 1990; ASB E100, Mod1.0, October 1990. USEPA Region 4 Science and Ecosystem Support Division, Analytical Support Branch.
- *Determination of Percent Lipid in Tissue.* Geochemical and Environmental Research Group, Texas A&M University College Station, TX. NOAA Technical Memorandum NOS ORCA 130 National Status and Trends Program for Marine Environmental Quality Sampling and Analytical Methods of the National Status and Trends Program Mussel Watch Project: 1993-1996 Update
- Laboratory SOP BR-EX-017 *Glassware Cleaning Procedure*
- Laboratory SOP BR-QA-019 *Data Review Procedure*
- Laboratory SOP BR-EH-001 *Hazardous Waste*
- Corporate SOP CW-E-M-001*Corporate Environmental Health and Safety Manual*

16.0 Method Modifications

There are no modifications from referenced method.

17.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions

18.0 Revision History

BR-EX-016, Revision 8:

- Title Page: Updated approval signatures.

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Appendix A: Terms and Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Title: Polychlorinated Biphenyls (PCBs) by GC/ECD (SW-846 8082)

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Approval Date: November 3, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure used to determine the concentration of polychlorinated biphenyls (PCBs) as Aroclors using dual column gas chromatography with electron capture detectors (GC/ECD).

This SOP is applicable to instrument analysis only. Extraction and extract cleanup procedures are provided in separate SOPs.

1.1 Analytes, Matrices, and Reporting Limits

This procedure may be used for a variety of matrices including: water, soil, sediment and tissue.

The list of target compounds that can be determined from this method along with the associated reporting limits (RL) is provided in Table 1.

2.0 Summary of Method

2 uL of extract is injected into a dual capillary column gas chromatograph equipped with electron capture detectors (GC/ECD). The chromatographic data is used to determine the list of analytes provided in Table 1.

This SOP is based on the following reference method:

- SW-846 Method 8082 Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, December 1996.

If the laboratory procedure is modified from the above reference method, a list of modifications will be provided in Section 16.0 of this SOP.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

- Method interference may be caused by contaminants in the extraction solvent. Solvents should be stored away from organochlorine compounds to minimize contamination.

- Non-target compounds co-extracted from the sample matrix can also cause interference, the extent of which will vary depending on the nature of the samples. Elemental sulfur is often found in sediment samples and its presence will result in broad peaks. Samples are screened prior to analysis, and those samples that contain high levels of sulfur are subject to sulfur cleanup (SW-846 3660B). Cleanup procedures that may be used for this method include: GPC (SW-846-3640A), silica gel (SW-846 3630C), Florisil (SW-846 3620B), and Sulfuric acid Cleanup (SW-846 3665A).

- Phthalate esters introduced during sample preparation can pose a problem in the determination of target analytes. Common flexible plastics contain varying amounts of

phthalate esters. These phthalate esters can be easily extracted or leached during extraction. To minimize this interference, avoid contact with any plastic materials.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats, and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst must be aware of the locations of those zones and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off or disconnect it from its source of power.

5.2 Primary Materials Used

Table 2 lists materials used in this method which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Miscellaneous

- Autosampler Vials, National Scientific or equivalent.
- Hydrogen Generator: Parker Balston.
- Volumetric Syringes, Class “A” (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.

6.2 Analytical System

- Computer Hardware/Software: GC Acquisition Platform - VAX 4505 (GVAX) Multichrom V2.11. Data Processing - Hewlett-Packard 9000-series computers, an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5 or higher.

- GC/ECD: with dual columns, dual ECDs, and auto-sampler capable of a 2-µl injection split onto two columns: HP 5890 with Leap Technology CTC A200SE and A200S Fisons autosamplers, Agilent Technologies 6890N with 7683 Series injector, or equivalent.
- GC Columns: A dual fused silica capillary column system that will provide simultaneous primary and confirmation analyses:

    - RTX-5, (30m x 0.25 mmID x 0.25um)
    - RTX-35, (30m x 0.25 mmID x 0.25um)

Equivalent columns may be used provided the elution orders are documented and compound separations are maintained.

7.0 Reagents and Standards

7.1 Reagents

- Hexane, Ultra-Resi Analyzed, JT Baker or equivalent.

7.2 Standards

Purchase stock standard solutions from commercial vendors and from these prepare calibration and working standards by diluting a known volume of stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The recommended formulation for each standard used in this procedure is provided in Appendix B along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection, so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method. Listed below are minimum sample size, preservation, and holding time requirements needed for this test.

Matrix	Sample Container	Minimum Sample Size	Preservation	Extract Holding Time	Reference
Water	Glass	1 L	Chilled to 4°C	40 Days	SW-846 8082
Solid	Glass	50 g	Chilled to 4°C	40 Days	SW-846 8082

[1]Analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Table 3

Matrix Spike(s) MS/MSD	Client Request	See Table 3
Sample Duplicate (SD)	Client Request	See Table 3

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Table 3
Second Source Calibration Verification (ICV)	Once, after each ICAL	See Table 3
Continuing Calibration Verification (CCV)	Daily, every 10 samples, end of sequence	See Table 3
Retention Time Windows	As Needed	See Table 3

10.0 Procedure

10.1 Instrument Operating Conditions

Install a five meter deactivated guard column into the injection port and connect the guard column to the separate analytical columns using a glass "Y". The analytical columns are installed into independent ECD detectors.

The recommended instrument operating conditions are as follows:

Initial Temperature:	130°C for 1 minute Temperature Program: 20°C per minute to 190°C to 5°C per minute to 225°C to 20.0°C per minute to 300°C. Hold for 6 minutes.
Detector Temperature	300°C
Injector Temperature:	200°C
Injection volume:	2µL
Carrier Gas:	Hydrogen (supplied by hydrogen generators)

Optimize the flow rate of the carrier gas by injecting an un-retained substance onto the column at an isothermal oven state and adjusting the flow to obtain the recommended dead volume time.

10.2 Retention Time Window Establishment

Whenever a new GC column is installed, establish RT windows for each analyte by analyzing three standards over a 72-hour period. Calculate the mean RT and Standard Deviation (SD). The RT window is calculated as the mean RT $\pm$ 3SD. If the SD is <0.01 minutes, a default SD of 0.01 minutes may be used.

If this procedure results in RT windows that are too tight, favoring false negatives, the laboratory may opt to use an alternate method to determine the RT windows. An alternate method consists of using a RT window of $\pm$ 0.05 minutes. The center of the RT window is set at the midpoint calibration level in the initial calibration sequence. RT windows are then updated daily (minimum frequency), re-centering the windows on the retention times established in a CCV.

10.3 Instrument Calibration

10.3.1 Initial Calibration (ICAL)

Clean the injection port and column with a hexane instrument blank prior to calibration.

To calibrate the instrument analyze a standard containing a mixture of Aroclor 1016 and Aroclor1260 (AR1660) at a minimum of five concentrations and use this multi-point calibration to determine the concentration of AR1016 and AR1260 in sample.

The mixed AR1660 standard includes most of the peaks represented in the other Aroclors so the multi-point calibration can also be used to demonstrate linearity of the instrument and that a sample does not contain peaks that represent the other Aroclors but it is not sufficient for pattern recognition. For the remaining Aroclors analyze a single-point standard at a concentration near the mid-point of the calibration and use these standards for pattern recognition and calculation of a single-point calibration factor. The laboratory does not perform a multi-point calibration for the remaining Aroclors unless requested for the project or by regulatory requirement.

Prepare the calibration standards using the formulations provided in Appendix B then transfer ~100 ugL to an autosampler vial insert. Place the vials in the autosampler, set the autosampler to inject 2-µl of each standard onto the instrument and initiate the analytical sequence.

A minimum of 3 peaks must be chosen for each Aroclor, and preferably 5 peaks. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016/1260 mixture.

The data processing system calculates the Calibration Factor (CF), mean CF, and Percent Relative Standard Deviation (%RSD) for each analyte on both columns. The %RSD for each target analyte must be less than or equal to 20% in order to use the mean CF for quantification. This evaluation is performed for each quantitation peak chosen for each Aroclor. All peaks must pass the 20% evaluation, not the average of the 5 peaks chosen for quantitation. If this criterion is not met, use another suitable quantification method for that analyte or correct the problem and repeat the calibration. Once a method of quantification is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is performed.

The calibration factor is used to determine the linearity of the calibration.

Alternate Quantification Option:

Linear Regression: Generate a curve of concentration vs. response for each analyte and calculate the correlation coefficient. The calibration must have a correlation coefficient (r) $\geq$ 0.995. If this criterion is not met, correct the problem and repeat the calibration. The use of linear regression requires a minimum of 5 calibration points.

10.3.2 Second Source Calibration Verification (ICV)

Immediately after each calibration and prior to the analysis of any QC or field samples, verify the accuracy of the initial calibration by analyzing a second source ICV.

Prepare the ICV using the formulation provided in Appendix B. Inject 2 µl of the ICV standard onto the instrument in the same manner as performed for the initial calibration standards.

The percent recovery of the average concentration of the peaks chosen for quantitation must be within ± 20% of the expected value (%R 80-120). If this criterion is not met, correct the problem and reanalyze the ICV. If reanalysis fails, remake the calibration standards and/or perform instrument maintenance and recalibrate. The acceptance criteria must be met on both columns.

10.3.3 Continuing Calibration Verification (CCV)

Analyze a CCV (1660) at or below the mid-calibration range each day before sample analysis, after every ten sample injections and at the end of each analytical sequence.

The laboratory does not perform a CCV for the remaining Aroclors unless requested for the project or by regulatory requirement.

The data system calculates the calibration factor (CF) and percent difference using the average percent difference of the peaks chosen for quantitation.

The percent difference or drift must be within ±15% and the retention time (RT) must be within the established RT window. Acceptance criteria must be met on both columns.

If the CCV fails, it may be repeated once. If repeat analysis fails, corrective action must be taken. The sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples must be bracketed by passing CCVs. Samples analyzed before and after CCV failures must be reanalyzed, unless the CCV is high and there are no detects in the associated samples.

10.4 Troubleshooting

Check the following items in case of calibration failures:

- ICAL Failure – Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if the chromatography has degraded as evidenced by peak shapes.
- CCV Failure – Perform Injection port maintenance; if injection port maintenance does not restore CCV, install a new guard column and remove one or more loops from each analytical column.
- Needle crushed during injection - Replace the needle and check the injection port for obstructions and check the autosampler for misalignment.
- Auto-sampler failure - Reset the auto-sampler.
- Power failure - Reset run in Multichrom and re-acquire or re-initiate run sequence.

10.5 Analysis

Remove the extract from refrigerated storage and warm to room temperature.

Transfer approximately 100 uL of extract to an autosampler vial and place the vials in the autosampler in a sequence that begins with the calibration standards followed by the analysis of an ICV, QC samples, field samples and continuing calibration verification standards (CCVs).

Enter the sample ID's into the data acquisition program in the order that the samples were placed in the autosampler tray and initiate the analytical sequence.

An example analytical sequence that includes calibration is as follows:

Injection Number	Lab Description
1	Instrument Blank
2	Instrument Blank
3	Instrument Blank
4	AR1221 (200 ppb)
5	AR1232 (200 ppb)
6	AR1242 (200 ppb)
7	AR1248 (200 ppb)
8	AR1254 (200 ppb)
9	AR1262 (200 ppb)
10	AR1268 (200 ppb)
11	AR1660 (50 ppb)
12	AR1660 (100 ppb)
13	AR1660 (200 ppb)
14	AR1660 (400 ppb)
15	AR1660 (800 ppb)
16	Instrument Blank
17	ICV
18-27	10 injections
28	CCV (AR1660 200ppb)
	Repeat steps 18-28

Cleaning blanks (IBLK) consisting of hexane may be analyzed after high-level samples at the discretion of the analyst.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

The data processing system identifies the target analytes by comparing the retention time of the peaks to the established retention time windows.

Review and accept or reject the qualitative identifications made by the data processing system using the following guidelines:

Compare the retention time of the peak to the established RT window, taking into account the shift of the surrogate peaks. If the surrogate peaks have shifted, open the retention time window in the direction of the shift. The processing system identifies the peak in the retention time window that is closest to the expected retention time set in the Target method, so the peak may need to be re-identified if a shift has occurred. The data system does not recognize Aroclor

patterns. The analyst manually identifies Aroclors by comparing the pattern in the samples to the patterns in the initial calibration standards. Weathering of PCB's in the environment may alter the PCB's to the point that the pattern no longer matches the pattern established for that Aroclor in the initial calibration. The laboratory takes the best pattern match approach to the identification and quantification of weathered PCB's.

Look for shoulders on the side of large peaks that may be peaks of interest. The processing system does not always automatically integrate shoulders from larger peaks, so manual integration (split) of the shoulder may be necessary.

Each target analyte must be detected on each column for qualitative identification to be made.

11.2 Quantitative Identification

Using an average of the chosen quantification peaks per Aroclor the data system calculates the corrected concentration for each target analyte using the equations given in Appendix C. If sample interference is suspected, the laboratory may remove up to two quantification peaks per column. The higher value between the two columns is reported as the primary result unless there is evidence of chromatographic anomalies, in which case the lower value will be reported. This deviation must be noted in the project narrative.

11.3 Calculations

See Appendix C.

11.4 Data Review

See laboratory SOP BR-QA-019 for data review requirements.

11.4.1 Primary Review

Review project documents to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Confirm qualitative and quantitative identification criteria using the criteria provided in Sections 11.1 and 11.2. If the data system does not properly integrate the peaks perform manual integration in accordance with laboratory SOP BR-QA-006.

Upload the data files from the data processing system to the laboratory information management system (TALS). Complete the batch information for standards and reagents and verify ICAL and QC sample associations. Review the results and set results to primary, secondary, acceptable or rejected as appropriate. Dilute and reanalyze samples whose results exceed the calibration range. The dilution analysis should result in a determination within the calibration range, preferably in the upper half of the calibration range. A more concentrated analysis is not necessary unless the project requires it. Dilution analyses may be performed to minimize matrix interference.

If a sample was analyzed immediately following a high concentration sample, review the results of the sample for any sign of carryover. If carryover is suspected, reanalyze the sample.

Create a non-conformance report (NCM) for any calibration, QC and sample data that is reported outside established acceptance criteria and/or schedule necessary corrective action. Set batch to 1st level review and complete the data review checklist.

11.4.2 Secondary Data Review

Verify quantitative and qualitative identification in the initial calibration standards and spot check such for ~15% of the remaining data in the batch.

If manual integrations were performed:

- Review each integration to verify that the integration meets the requirements for manual integration as specified in laboratory SOP BR-QA-006. If an error is suspected or found consult with the analyst that performed the integration analyst and request correction or notify the Department Manager, Technical Director or QA Manager. Do not "fix" the integration. Reintegration by a secondary data reviewer must not be performed except in limited circumstances as approved by the department supervisor or other laboratory management. If those instances where the secondary reviewer performs the integration, this person is now considered the primary analyst and each integration performed by the secondary reviewer must be subsequently reviewed by a peer analyst or the department supervisor to verify the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-006.

- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in laboratory SOP BR-QA-005.

Review project documents to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Verify that the acceptance criteria for the calibration and QC items listed in Table 1 were met. If the results do not fall within the established limits verify the recommended corrective actions were performed. If not, initiate corrective actions and/or verify an NCM was created to document the criteria exception. Verify analytical results are qualified accordingly. Set batch to 2nd level review and complete the data review checklist.

11.5 Data Reporting

The report format, application of data qualifiers and creation of the data deliverable is performed by the LIMS using the formatter set by the project manager during log-in.

Records of electronic and hardcopy data are maintained as described in laboratory SOP BR-QA-014.

12.0 Method Performance

12.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ)

Establish a LOD and LOQ at initial method set up following the procedures specified in laboratory SOP BR-QA-005. Verify the LOD and LOQ at the frequency established for the method using the procedures specified in same SOP. The frequency of LOD and LOQ verification depends on

the strictest frequency of the regulatory program for which the method supports. The frequency requirement is documented in a spreadsheet maintained by the QA Department.

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when there is a significant change in instrumentation or procedure.

Each analyst that performs the analytical procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP for employee training.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of the SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Vials containing sample extracts: Satellite container: 15 gallon bucket connected to a fume hood.
- Solvent Waste: Satellite container: 1 L glass bottle located in fume hood.

15.0 References / Cross-References

- SW-846 Method 8082 Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, December 1996.
- Corporate Environmental Health and Safety Manual (CW-E-M-001)
- Laboratory SOP BR-QA-011
- Laboratory SOP BR-LP-011

- Laboratory SOP BR-QA-014
- Laboratory SOP BR-QA-006
- Laboratory SOP BR-QA-005

16.0 Method Modifications

Not applicable.

17.0 Attachments

- Table 1: Target Compound List and Reporting Limit
- Table 1A: Accuracy and Precision Limits
- Table 2: Primary Materials Used
- Table 3: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables
- Appendix C: Equations

18.0 Revision History

BR-GC-005, Rev. 10:

- Updated approval signatures
- Section 10: Inserted note regarding multi-point calibrations for other Aroclors.

BR-GC-005, Rev. 9

- Updated reference method in Section 2.0.
- Changed QC criteria for %D from 15% to 20%.
- Added language to Section 10.2 to allow for updating RT windows using CCVs.
- Added language to Section 11.4.1 to allow for dilution to minimize matrix interference.
- Added standard preparation tables to Appendix B to allow for the preparation of 5 point calibrations for each of the Aroclors

Table 1: Routine Target Analyte List & Reporting Limits (RL)

ANALYTE	Routine Reporting Limit (RL)[1,2]	
	Water (ug/L)	Solid (ug/Kg)
AR1016	0.50	17
AR1221	0.50	17
AR1232	0.50	17
AR1242	0.50	17
AR1248	0.50	17
AR1254	0.50	17
AR1260	0.50	17
AR1262	0.50	17
AR1268	0.50	17

[1]*The routine RL is the unadjusted value that can be achieved in a blank matrix.*
[2]*The RL for tissue matrix is project defined.*

Table 1A: Routine Accuracy and Precision Limits[1]

Analyte	In-House Limits (%R)		Precision (RPD) (≤)
	Water	Solid	
AR1016	55-120	55-120	30
AR1260	60-125	55-125	30
Surrogate: Decachlorobiphenyl (DCB)	30-150	45-125	NA
Surrogate:TCX (Advisory)[2]	55-120	30-130	NA

[1] *The limits in this table are those used as of the effective date of this SOP. Current limits are stored in the LIMS database.*
[2] *The control limits for TCX are advisory. Corrective action is not performed when recovery is outside limits.*

Table 2: Primary Materials Used

Material [1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	Option 1: RSD for each analyte ≤ 20% Option 2: Linear Regression: r ≥ 0.995	Correct problem, reanalyze, repeat calibration.
ICV	After each initial calibration	(% R) ± 20% from expected value	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±15%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and bracketed samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	Target Analyte < RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	Per client request	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate	All field and QC samples	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]*The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are only produced in this range and qualified with the proper data reporting flag when a project requires this type of data reporting.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Appendix B: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in hexane using Class A volumetric glassware and Hamilton syringes. Unless otherwise specified for a standard solution, assign an expiration date of 6 months from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. Store the prepared solutions under refrigeration and protected from light at a temperature of 4ºC (±2). See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance.

Intermediate Calibration Standards (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
AR1660[1]	Restek #32039	Aroclor 1016 Aroclor 1260	1000	0.40	40	10
AR1254	Restek #32011	Aroclor 1254	1000	0.40	40	10
AR1248	Restek #32010	Aroclor 1248	1000	0.40	40	10
AR1242	Restek #32009	Aroclor 1242	1000	0.40	40	10
AR1232	Restek #32008	Aroclor 1232	1000	0.40	40	10
AR1221	Restek #32007	Aroclor 1221	1000	0.40	40	10
AR1262	Restek #32409	Aroclor 1262	1000	0.40	40	10
AR1268	Restek #32410	Aroclor 1268	1000	0.40	40	10

[1] *Standard is a mix of AR1016/AR1260. Concentration shown is the concentration of each Aroclor in the mixed standard.*

Intermediate ICV Standard (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
AR1660	Ultra Scientific PPM8082	Aroclor 1016 Aroclor 1260	1000	0.40	40	10

Surrogate Solution (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Pesticide Surrogate	Restek #3200	TCX DCB	1000	0.40	40	10

Working ICV Standard (200 ug/L)

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
Intermediate ICV	Laboratory Prepared	Aroclor 1016 Aroclor 1260	10	0.80	40	200
Surrogate	Laboratory Prepared	TCX DCB	10	0.080		20

AR1660 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1660 Intermediate	Laboratory Prepared	Aroclor 1016 Aroclor 1260	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1660 calibration standards*

AR1660 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1660 Level 5	AR1660 CAL Level 4	800	20	40	400
AR1660 Level 5	AR1660 CAL Level 3	800	10	40	200
AR1660 Level 5	AR1660 CAL Level 2	800	5.0	40	100
AR1660 Level 5	AR1660 CAL Level 1	800	2.5	40	50

AR1221 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1221 Intermediate	Laboratory Prepared	Aroclor 1221	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1221 calibration standards*

AR1221 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1221 Level 5	AR1221 CAL Level 4	800	20	40	400
AR1221 Level 5	AR1221 CAL Level 3	800	10	40	200
AR1221 Level 5	AR1221 CAL Level 2	800	5.0	40	100
AR1221 Level 5	AR1221 CAL Level 1	800	2.5	40	50

AR1232 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1232 Intermediate	Laboratory Prepared	Aroclor 1232	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1232 calibration standards*

AR1232 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1232 Level 5	AR1232 CAL Level 4	800	20	40	400
AR1232 Level 5	AR1232 CAL Level 3	800	10	40	200
AR1232 Level 5	AR1232 CAL Level 2	800	5.0	40	100
AR1232 Level 5	AR1232 CAL Level 1	800	2.5	40	50

AR1242 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1242 Intermediate	Laboratory Prepared	Aroclor 1242	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1242 calibration standards*

AR1242 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1242 Level 5	AR1242 CAL Level 4	800	20	40	400
AR1242 Level 5	AR1242 CAL Level 3	800	10	40	200
AR1242 Level 5	AR1242 CAL Level 2	800	5.0	40	100
AR1242 Level 5	AR1242 CAL Level 1	800	2.5	40	50

AR1248 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1248 Intermediate	Laboratory Prepared	Aroclor 1248	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1248 calibration standards*

AR1248 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1248 Level 5	AR1248 CAL Level 4	800	20	40	400
AR1248 Level 5	AR1248 CAL Level 3	800	10	40	200
AR1248 Level 5	AR1248 CAL Level 2	800	5.0	40	100
AR1248 Level 5	AR1248 CAL Level 1	800	2.5	40	50

AR1254 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1254 Intermediate	Laboratory Prepared	Aroclor 1254	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1254 calibration standards*

AR1254 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1254 Level 5	AR1254 CAL Level 4	800	20	40	400
AR1254 Level 5	AR1254 CAL Level 3	800	10	40	200
AR1254 Level 5	AR1254 CAL Level 2	800	5.0	40	100
AR1254 Level 5	AR1254 CAL Level 1	800	2.5	40	50

AR1262 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1262 Intermediate	Laboratory Prepared	Aroclor 1262	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1262 calibration standards*

AR1262 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1262 Level 5	AR1262 CAL Level 4	800	20	40	400
AR1262 Level 5	AR1262 CAL Level 3	800	10	40	200
AR1262 Level 5	AR1262 CAL Level 2	800	5.0	40	100
AR1262 Level 5	AR1262 CAL Level 1	800	2.5	40	50

AR1268 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1268 Intermediate	Laboratory Prepared	Aroclor 1268	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1268 calibration standards*

AR1268 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1268 Level 5	AR1268 CAL Level 4	800	20	40	400
AR1268 Level 5	AR1268 CAL Level 3	800	10	40	200
AR1268 Level 5	AR1268 CAL Level 2	800	5.0	40	100
AR1268 Level 5	AR1268 CAL Level 1	800	2.5	40	50

Appendix C: Equations

Calibration Factor (CF_x) = $\frac{\text{Peak area or height}_{(x)}}{\text{Standard concentration}_{(ug/L)}}$

Mean Calibration Factor ($\overline{CF}$) = $\frac{\sum_{i=1}^{n} CF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Calibration Factor (SD) = $\sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Calibration Factor = $\frac{SD}{\overline{CF}} \times 100\%$

Percent Difference (%D) = $\frac{CF_v - \overline{CF}}{\overline{CF}} \times 100\%$ Add absolute value signs

where: CF_v = Calibration Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $\frac{\text{Calculated Concentration – Theoretical Concentration}}{\text{Theoretical Concentration}}$ X 100%

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (RPD) = $\frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$C_{extract}$ (ug/L) = $\frac{\text{Peak Area (or Height)}}{\overline{CF}}$

Note: The concentrations of the 3-5 peaks chosen for quantification is calculated and the average is then taken for final calculation.

Water

$$C_{sample}(ug/L) = C_{extract}(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample volume (L)}} \times DF$$

Solid

$$C_{sample}(ug/Kg) = C_{extract}(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample weight (Kg)}} \times \frac{100}{\%\,\text{solids}} \times DF$$

Title: Chlorinated Pesticides by GC/ECD (SW-846 8081A)

Approvals (Signature/Date):

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Approval Date: May 4, 2009

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure used to determine the concentration of certain organochlorine pesticides using dual column gas chromatography with electron capture detectors (GC/ECD). This SOP is applicable to the analytical procedure. The extraction procedures are given in laboratory SOPs:

- BR-EX-005 Separatory Funnel Extraction
- BR-EX-008 Ultrasonic Extraction
- BR-EX-002 Extract Cleanup Procedures
- BR-EX-011 Gel Permeation Chromatography (GPC)

1.1 Analytes, Matrix(s), and Reporting Limits

This procedure may be used for a variety of matrices including: water, soil, sediment, air, and tissue.

The list of target compounds that can be determined from this method along with the associated reporting limit (RL) is provided in Tables 1 and 1A.

2.0 Summary of Method

A measured volume or weight of sample is extracted using an appropriate matrix-specific extraction technique. After extraction, the extract may be subject to cleanup depending on the nature of sample matrix and the target analytes. After cleanup, the extract is analyzed by injecting a 2 uL aliquot into a dual capillary column GC/ECD.

This SOP is based on the following reference method:

SW-846 Method 8081A, Organochlorine Pesticides by Gas Chromatography, Test Methods for the Chemical Analysis of Water and Wastes, Revision 1, December 1996.

Method modifications are listed in Section 16.0.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

- Method interference may be caused by contaminants in the extraction solvent. Solvents should be stored in an area away from organochlorine compounds to minimize contamination.

- Non-target compounds co-extracted from the sample matrix can also cause interference, the extent of which will vary considerably depending on the nature of the samples. Elemental sulfur is often found in sediment samples and its presence will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Samples are screened before analysis and those samples that contain high levels of sulfur are subject to cleanup using activated copper before analysis (SW-846 3660B). Waxes, lipids, other high molecular weight materials and co-eluting organophosphorous pesticides may be removed by extract

cleanup with GPC (SW-846 3640A). Co-eluting chlorophenols can be eliminated by cleanup with silica gel (SW-846 3630C), or Florisil (SW-846 3620B).

- Phthalate esters introduced during sample preparation can pose a problem in the determination of pesticides. Common flexible plastics contain varying amounts of phthalate esters, and these can be easily extracted or leached during extraction. To minimize this interference, avoid contact with any plastic materials.

- The presence of PCBs may interfere with the analysis of organochlorine pesticides. This interference is most severe for multi-component analytes such as Chlordane and Toxaphene.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.1 Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

Table 3 lists those materials used in this procedure that have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Miscellaneous

- Autosampler Vials, National Scientific or equivalent.

- Hydrogen Generator: Whatman.

- Volumetric Syringes, Class “A” (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.

6.2 Analytical System

Computer Hardware/Software: GC Acquisition Platform - VAX 4505 (GVAX) Multichrom V2.11. Data Processing - Hewlett-Packard 9000-series computers, an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5 or higher.

GC/ECD: with dual columns, dual ECDs, and auto-sampler capable of a 2 µL injection split onto two columns: Agilent Technologies 6890N with 7683 Series injector, or equivalent.

GC Columns: A dual fused silica capillary column system that will provide simultaneous primary and confirmation analyses.

- RTX-CLPesticides (30m x 0.32 mmID x 0.50um), Restek or equivalent or
- RTX-CLPesticides (30m x 0.32 mmID x 0.32um), Restek or equivalent
- RTX-CLPesticides II, (30m x 0.32 mm ID x 0.25um), Restek or equivalent.

Equivalent columns may be used so long as elution orders are documented and compound separations are maintained.

7.0 Reagents and Standards

7.1 Reagents

- Acetone, Ultra-Resi Analyzed. JT Baker or equivalent.
- Hexane, Ultra-Resi Analyzed. JT Baker or equivalent.
- Methanol, Ultra-Resi Analyzed. JT Baker or equivalent.

7.2 Standards

Purchase stock standard solutions from commercial vendors and from these prepare calibration and working standards by diluting a known volume of stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The recommended formulation for each standard used in this procedure is provided in Appendix B along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Water	Glass	1 L	Chilled to 4°C	7 Days: Extraction 40 Days: Analytical	SW-846 8081A
Solid	Glass	50 g	Chilled to 4°C	14 days: Extraction 40 Days: Analytical	SW-846 8081A
Tissue	Glass/other	50 g/available	Chilled to 4°C or Frozen to -15°C	14 days: Extraction 40 Days: Analytical	SW-846 8081A

[1]Analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts

are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Table 4
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Table 4
Matrix Spike(s) (MS/MSD)	1 pair per extraction batch when sufficient sample volume is provided or per client request	See Table 4
Sample Duplicate (SD)	Client Request	See Table 4

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Table 4
Second Source Calibration Verification (ICV)	Once, after each ICAL	See Table 4
Continuing Calibration Verification (CCV)	Daily, every 10 samples, end of sequence	See Table 4
Retention Time Windows	As Needed	See Table 4
Breakdown Check Standard (BCS)	Prior to ICAL, prior to each CCV	See Table 4

10.0 Procedure

10.1 Instrument Operating Conditions

Install a five meter deactivated guard column to the injection port and connect the guard column to the separate analytical columns using a glass “Y”. Then attach the analytical columns to the dual ECD detectors.

The recommended instrument operating conditions are as follows:

Initial Temperature: 120°C
Temperature Program:
Columns Set: RTX-CLPesticides (30m x 0.32 mmID x 0.50um) and RTX-CLPesticides II, (30m x 0.32 mm ID x 0.25um)
45°C per minute to 200°C, 15°C per minute to 230°C, 15°C per minute to 300°C. Hold for 2 minutes.
Columns Set: RTX-CLPesticides (30m x 0.32 mmID x 0.32um and RTX-CLPesticides II, (30m x 0.32 mm ID x 0.25um)
35°C per minute to 200°C, 25°C per minute to 230°C, 30°C per minute to 300°C. Hold for 2 minutes.
Detector Temperature 300°C

Injector Temperature:	200°C
Injection volume:	2-µL
Carrier Gas:	Hydrogen (supplied by hydrogen generators)
Flow:	
Columns Set:	RTX-CLPesticides (30m x 0.32 mmID x 0.50um) and RTX-CLPesticides II, (30m x 0.32 mm ID x 0.25um) Constant Flow of 2.3ml/min used. Split/Splitless injection used. Pulsed injection of 29psi for 0.5min, 0.3min purge time.
Columns Set:	RTX-CLPesticides (30m x 0.32 mmID x 0.32um and RTX-CLPesticides II, (30m x 0.32 mm ID x 0.25um) Constant Flow of 2.4ml/min used. Split/Splitless injection used. Pulsed injection of 29psi for 0.5min, 0.3min purge time.

10.2 Retention Time Window Establishment

Whenever a new GC column is installed establish RT windows for each analyte by analyzing three standards over a 72-hour period and calculating the mean RT and Standard Deviation (SD). Calculate the RT window as mean RT ± 3SD of the three standards. If the SD is <0.01 minutes, a default SD of 0.01 minutes may be used. If, in the professional judgment of the analyst, this procedure results in an RT window that is too tight and would favor false negatives, the laboratory may opt to use an alternate method to determine the RT windows. An alternative method is as follows: using the RT of the midpoint initial calibration standard, calculate the RT window using ± 0.05 minutes from the midpoint of the RT in the initial calibration.

10.3 Instrument Calibration

10.3.1 Initial Calibration (ICAL)

Perform initial calibration of the instrument during initial method set-up, whenever a new column is installed, when significant instrument maintenance has been performed and when the result of the continuing calibration verification (CCV) indicates the calibration is no longer valid. Significant instrument maintenance includes changing the lengths of the analytical columns and baking or installing detectors.

Before calibration, or if the instrument has been idle for longer than 8 hours, prepare and analyze a Column Prime Standard. The formulation for the preparation of the standard is provided in Appendix B.

To initiate the calibration, prepare and analyze the breakdown check standard (BCS), see Section 10.3.3. The formulation for the preparation of the BCS is provided in Appendix B. Evaluate the results. The individual breakdown values for DDT and Endrin must be ≤ 15%. If the breakdown criteria are not met, correct the problem and reanalyze the BCS. Repeat the analysis of the BCS at the start of each sequence, every 10 samples, and at the end of the sequence.

Prepare the calibration standards using the formulation provided in Appendix B. Inject 2-µL of each calibration standard onto the instrument using the same technique used for sample extracts as described in Section 10.5. Unless otherwise specified for a specific project, the calibration for single component pesticides is established with a minimum of five calibration points. Multi-component pesticides use a single-point calibration at or near the low-point of the calibration range for pattern recognition.

When a multi-component pesticide is detected in a field sample, a five-point curve is established for the analyte and the extract is re-analyzed and quantified from the five-point calibration.

The data processing system calculates the Calibration Factor (CF), mean CF and Percent Relative Standard Deviation (% RSD) for each analyte on both columns. The equations used are provided in Appendix C. The RSD for each target analyte must be less than or equal to 20% in order to use the mean CF for quantification. If this criterion is not met use another suitable quantification method for that analyte or correct the problem and repeat the calibration. Once a method of quantification is chosen for a specific compound, it must be consistently used throughout the entire analytical sequence until a new initial calibration is performed.

Alternate Quantification Option:
Linear Regression & Weighted Linear Regression: Generate a curve of concentration vs. response for each analyte and calculate the correlation coefficient. The calibration must have a correlation coefficient (r) $\geq$ 0.995 (or $r^2 \geq 0.990$). If this criterion is not met, correct the problem and repeat the calibration. The use of linear regression requires a minimum of 5 calibration points.

NOTE: Unless otherwise specified for the project, for DoD QSM work, the quantification option (mean CF or linear regression) must meet criteria for each target analyte otherwise the calibration must be repeated. The DoD QSM prohibits the reporting of samples against an ICAL that does not meet criteria even when associated analytical results are flagged.

10.3.2 Second Source Calibration Verification (ICV)

Immediately after each calibration and prior to the analysis of any other QC or field samples, verify the accuracy of the initial calibration by analyzing a second source ICV.

Prepare the ICV using the formulation provided in Appendix B. Inject 2 µL of the ICV standard onto the instrument in the same manner as performed for the initial calibration standards.

The percent recovery of each analyte must be within ±20% of the expected value (%R 80-120) for each analyte. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and/or perform instrument maintenance and recalibrate. The acceptance criteria must be met on both columns.

10.3.3 Breakdown Check Standard (BCS)

Analyze the breakdown check standard (BCS) prior to initial calibration and prior to all CCV analyses. The formulation for the preparation of the BCS is provided in Appendix B. Evaluate the results against the criteria in Table 4. The breakdown values for DDT and Endrin must each be ≤ 15%. If the breakdown criteria are not met, correct the problem and reanalyze the BCS.

10.3.4 Continuing Calibration Verification (CCV)

Analyze a CCV each day before sample analysis (after analysis of the BCS) after every ten samples and at the end of each analytical batch to monitor instrument drift. The CCV should include all single component analytes and the concentration of the CCV standard should be within the calibration range. When a multi-component analyte is detected in a field sample, analyze a multi-component CCV.

The data system calculates the percent difference for each analyte on both columns. The percent difference must be within ±15% of the expected value from the ICAL for each analyte and the retention time (RT) for each analyte must be within the established RT window. The acceptance criteria must be met on both columns. If Technical Chlordane and Toxaphene are analyzed as CCVs, the average of the 3-5 analytical peaks chosen must past acceptance criteria.

If the CCV fails, it may be repeated once. If it still fails, corrective action must be taken. The sequence may be continued only if two immediate, consecutive CCVs , one at the midpoint of the calibration range and one below the midpoint, are within acceptance criteria. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples must be bracketed by passing CCVs, and samples before and after CCV failure must be reanalyzed unless the CCV is high and there are no detects in the associated samples.

10.4 Troubleshooting:

Check the following items in case of calibration failures:

- ICAL Failure – Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if the chromatography has degraded as evidenced by poor peak shapes.
- CCV Failure – Perform injection port maintenance; if injection port maintenance does not restore CCV, install a new guard column and remove one or more loops from each analytical column.
- BCS Failure - Perform injection port maintenance; if injection port maintenance does not restore CCV, install a new guard column and remove one or more loops from each analytical column.
- Needle crushed during injection - Replace the needle and check the injection port for obstructions and check the autosampler for misalignment.
- Autosampler failure - Reset the autosampler.
- Power failure - Reset run in Multichrom and re-acquire or re-initiate run sequence.

10.5 Sample Preparation

Remove the sample extract from refrigerated storage and warm to room temperature.

Transfer approximately 100 uL of extract to an autosampler vial then place the vials in the autosampler using the analytical sequence specified in the next section.

10.6 Sample Analysis

Analytical Sequence

An example analytical sequence that includes initial calibration (ICAL) and subsequent sample analysis is provided below.

Injection Number	Lab Description	
1	Column Priming Standard	Column Prime
2	Breakdown Check Standard	BCS
3	Single Component Level 1	INDAB-1

4	Single Component Level 2	INDAB-2
5	Single Component Level 3	INDAB-3
6	Single Component Level 4	INDAB-4
7	Single Component Level 5	INDAB-5
8	Technical Chlordane -50 ppb	T.CHLOR500
9	Toxaphene - 500 ppb	TOX50
10	ICV – Second Source Standard	ICV
11-20	10 injections	QC and Field Samples
21	Breakdown Check Standard	BCS
22	Continuing Calibration Verification Standard	CCV
23-32	10 injections	QC and Field Samples
33	Breakdown Check Standard	BCS
34	Continuing Calibration Verification Standard	CCV
Repeat ending with BCS and CCV		

When 2,4-DDE, 2,4-DDD and 2,4-DDT are required, analyze the five point calibration standards (refer to Appendix B) immediately after the single component calibration standards. Analyze a 2,4-DDX CCV after each single component CCV as described in section 10.3.3.

Cleaning blanks (CBLK) consisting of hexane may be analyzed after high-level samples at the discretion of the analyst.

Enter the sample ID's into the data acquisition program in the order the samples were placed in the autosampler and initiate the analytical sequence.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

The data processing system identifies the target analytes by comparing the retention time of the peaks to the retention times of the initial calibration standards.

Review and accept or reject the qualitative identifications made by the data processing system using the following guidelines:

Compare the retention time of the peak to that defined by the midpoint of the calibration, taking into account the shift of the surrogate peak. If the surrogate peak has shifted, open the retention time window in that direction. The processing system identifies the peak in the retention time window that is closest to the expected retention time set in the calibration, so the peak may need to be re-identified if a shift has occurred.

Multi-component pesticides are identified by pattern recognition and quantified using 3-5 major peaks. The data system calculates a calibration factor for each of the 3-5 major peaks for each calibration level using height or area. The average CF is used to calculate the concentration for each of the 3-5 major peaks, and the resulting concentrations are averaged to provide the final result in sample concentration.

Look for shoulders on the side of a large peak that may be the peak of interest. The processing system does not always automatically integrate the shoulder from the larger peak, so manual integration (split) of the shoulder may be necessary.

Each target analyte must be detected above the reporting limit for that compound on each column for qualitative identification to be made.

11.2 Quantitative Identification

The data system calculates the corrected concentration for each target analyte from the calibration curve using the equations given in the next section. If sample interference is suspected, the laboratory may choose to report the value from the result that is not affected by interference.

11.3 Calculations

See Appendix C.

11.4 Data Review

11.4.1 Primary Review

Confirm quantitative and qualitative identification criteria using the criteria provided in Section 11.1 and Section 11.2. If the data system does not properly integrate a peak, perform manual integration in accordance with laboratory SOP BR-QA-006 *Manual Integration*.

Review the instrument QC against the acceptance criteria given in Section 10.0 and summarized in Table 4. If the results do not fall within acceptance criteria perform the recommended corrective action. If corrective action is not taken document the situation with a nonconformance report (NCR) and provide technical justification for the decision to proceed with analysis in the NCR. If corrective action is not successful, provide explanation as appropriate in the NCR.

Review the MB against the acceptance criteria given in Table 4. If criteria are not met, investigate the source of contamination, eliminate the problem and remake and reanalyze the MB along with associated samples if the concentration of a target analyte in the MB is above the RL and is greater than 1/10 the concentration measured in any sample. If the concentration of the MB is less than 1/10 the concentration measured in any sample, corrective action is not required unless otherwise specified on a project basis.

In the absence of project-specific control limits, use the in-house control limits specified in Table 2 for the evaluation of the LCS, MS/MSD and sample duplicate (SD). For DoD QSM work, use the DoD limits specified in the same table. If results are outside control limits initiate a nonconformance report (NCR) and evaluate for marginal exceedance (See Appendix D) or correct the problem and remake and reanalyze the LCS along with associated samples. If corrective action is not taken provide technical justification for the decision to proceed with analysis in the NCR.

Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should result in a determination within the upper half of the calibration curve. If an initial dilution is performed based on screen data, the diluted analysis should result in a determination within the upper half of the calibration curve. A more concentrated analysis is not necessary unless the result is not within the upper half of the calibration range or when the project requires that all samples be analyzed undiluted or more concentrated regardless of screen results.

If a sample was analyzed immediately following a high concentration sample, review the results of the sample for any sign of carry over. If carry over is suspected, reanalyze the sample.

Compare results from each column to verify confirmation of each target analyte.

Generate chromatograms and quantitation reports for the calibration, calibration check standards, sample and associated QC analyses, associated summary forms, manual integration reports, primary review checklist, and compile the package.

11.4.2 Secondary Data Review

Check each qualitative identifications and spot check quantitative values using the criteria provided in Section 11.1 and Section 11.2.

If manual integrations were performed:

- Review each manual integration to verify that the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-006 *Manual Integration*. If a problem is found, immediately consult with the primary analyst or notify the Technical Director or QA Manager. Check that each manual integration is included in the manual integration summary and that each instance has an associated manual integration code. Also, check to ensure that a "before and after" report is present for each manual integration of reported analyte.

- Reintegration (by secondary data reviewers) should not be performed except in limited circumstances such as when the primary analyst who performed the initial integration is not available to correct any errors found during secondary review. If reintegration is performed, each integration performed by the secondary reviewer must be reviewed by a peer analyst or the department supervisor to verify the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-006 *Manual Integration*.

Verify that the performance criteria for the QC items listed in Table 4 were met. If the results do not fall within the established limits verify the recommended corrective actions were performed. If corrective action was not taken or is unsuccessful, ensure the situation is documented with a nonconformance report (NCR) and ensure data is qualified accordingly. Report the nonconformance in the narrative note program.

11.5 Data Reporting

Unless otherwise specified in a project plan, report the higher result between the two columns. If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If there is no evidence of chromatographic problems, report the higher result. If there is evidence of chromatographic problems, report the lower value for that compound and note this in the narrative. Report analytical results above the reporting limit (RL) as the value found. Report analytical results less than the RL, to the adjusted RL with a "U" data qualifier. Adjust the RL for sample dilution/concentration. The unadjusted RL for each target analyte is provided in Table 1.

NOTE: The laboratory does not routinely report estimated values for this test method. If a project requires such reporting, evaluate the results to the verified MDL (vMDL) for each target compound. Report any value between the vMDL and the adjusted RL as the value found qualified with a "J" data qualifier. Estimated values are evaluated to the MDL value derived from

the MDL study.

Evaluate the results of the MB and apply "B" flags to the sample data as follows:

- Estimated Value ("J") Flag Reporting & DoD Protocol: If any target analyte is detected in the MB greater than the MDL, apply a "B" flag to all sample results.

- RL Reporting: If any target analyte is detected in the MB greater than the RL, apply a "B" flag to all sample results.

If the sample was analyzed at multiple dilutions, report the result from the appropriate dilution (i.e. no target analyte above calibration range and the result for the analyte for which the dilution was performed is in the upper half of the calibration range). Provide results for the undiluted or more concentrated analyses when requested.

Review project documents such as the environmental test request (ETR) analytical worksheets, Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Generate the summary forms and compile the data report in the deliverable format specified by the laboratory PM and release the report to the report management group.

Retain, manage and archive electronic and hardcopy data as specified in laboratory SOP BR-QA-014 *Laboratory Records*.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Perform a method detection limit (MDL) study at initial method set-up following the procedures specified in laboratory SOP BR-QA-005 *Determination of LOD, LOQ and RL*.

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when there is a significant change in instrumentation or procedure.

Each analyst that performs the analytical procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP BR-QA-011 *Employee Training.*

12.3 Training Requirement

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Vials containing sample extracts: Satellite container: 15 gallon bucket connected to a fume hood.
- Solvent Waste: Satellite container: 1 L glass bottle located in fume hood.

15.0 References / Cross-References

- SW-846 Method 8081A, Organochlorine Pesticides by Gas Chromatography, Test Methods for the Chemical Analysis of Water and Wastes, Revision 1, December 1996.
- Corporate Environmental Health and Safety Manual (CW-E-M-001)
- Laboratory SOP BR-EX-005 *Separatory Funnel Extraction*
- Laboratory SOP BR-EX-008 *Ultrasonic Extraction*
- Laboratory SOP BR-EX-002 *Extract Cleanup Procedures*
- Laboratory SOP BR-EX-011 *Gel Permeation Chromatography (GPC)*
- Laboratory SOP BR-QA-005 *Determination of LOD, LOQ and RL*
- Laboratory SOP BR-QA-011 *Employee Training*
- Laboratory SOP BR-LP-011 *Hazardous Waste*
- Laboratory SOP BR-QA-014 *Laboratory Records*
- Laboratory SOP BR-QA-006 *Manual* Integration
- Laboratory SOP BR-QA-002 *Standard & Reagent Preparation*

16.0 Method Modifications

Modification Number	Method Reference	Modification
1	SW846 8081A	The laboratory has chosen a subset of the compounds listed in 8081A to be our routine reporting list. In addition to this list, we have a list of added compounds that may be reported by this method (see Table 1 and 1A). The added compounds 2,4'-DDE, 2,4'-DDD and 2,4'-DDT are not listed in 8081A.
2	SW846 8081A	Air and tissue matrices have been added.
3	SW846 8081A	Retention time window studies typically result in RT windows that are too narrow. RT windows of ± 0.05 minutes centered around

		the mid-level calibration standard RTs are employed.
4	SW846 8081A	Narrow-bore capillary columns are used for dual column analyses.
5	SW846 8081A	A single calibration mix is used for the single component pesticides.
6	SW846 8081A	The laboratory analyzes a single point calibration at the low-point of the calibration range for Toxaphene and Chlordane. When either compound is detected in a sample, a five point initial calibration is established and the sample is reanalyzed.
7	SW846 8081A	Analysis of an ICV is required after calibration and before sample analyses.
8	SW846 8081A	Chlordane is quantitated by choosing 5 quantitation peaks. A calibration factor is calculated for each peak and the average of the 5 concentration values is reported.
9	SW846 8081A	If corrective action was performed due to CCV failure, the sequence may continue only if two consecutive CCVs are within acceptance criteria.

17.0 Attachments

- Table 1: Target Compound List and Reporting Limit
- Table 1A: Added Compound List and RLs
- Table 2: Accuracy and Precision Limits
- Table 2A Accuracy and Precision Limits for Added Compounds
- Table 3: Primary Materials Used
- Table 4: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables
- Appendix C: Equations
- Appendix D: Marginal Exceedance Evaluation

18.0 Revision History

BR-GC-006, Rev. 9
- SOP updated to the new TestAmerica format.
- Section 1.0: SOP references were updated.
- Section 10.3.4: Update to reflect current laboratory practice
- Section 10.6: Added a reference to 2,4-DDX calibration.
- Table 2A: Created for added compounds.
- Appendix B: Updated standard formulations.

Table 1: Routine Target Analyte List & Reporting Limit (RL)[1]

Analyte	Water (ug/L)	Solid (ug/Kg)
alpha-BHC	0.05	1.7
beta-BHC	0.05	1.7
delta-BHC	0.05	1.7
gamma-BHC (Lindane)	0.05	1.7
Heptachlor	0.05	1.7
Aldrin	0.05	1.7
Heptachlor Epoxide	0.05	1.7
Endosulfan I	0.05	1.7
Dieldrin	0.10	3.3
4,4-DDE	0.10	3.3
Endrin	0.10	3.3
4,4-DDD	0.10	3.3
Endosulfan II	0.10	3.3
Endosulfan sulfate	0.10	3.3
4,4-DDT	0.10	3.3
Methoxychlor	0.50	17
Endrin ketone	0.10	3.3
Endrin aldehyde	0.10	3.3
alpha-Chlordane	0.05	1.7
gamma-Chlordane	0.05	1.7
Technical Chlordane	0.50	17
Toxaphene	5.0	170

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.

Table 1A: Added Compound List and Reporting Limit (RL)

Analyte	Water (ug/L)	Solid (ug/Kg)
Mirex	0.10	3.3
2,4-DDE	0.10	3.3
2,4-DDD	0.10	3.3
2,4-DDT	0.10	3.3

Table 2: Routine Accuracy and Precision Limits[1]

Analyte	In-House Limits[2] (%R)		Precision (RPD) (≤)	DoD QSM Limit[3]	
	Water	Solid		Water	Soil
alpha-BHC	70-135	**55-130**	30	25-140	60-125
beta-BHC	70-135	60-**130**	30	65-125	60-125
delta-BHC	70-135	**50-135**	30	45-135	55-130
gamma-BHC (Lindane)	70-135	55-**135**	30	25-135	60-125
Heptachlor	60-125	60-135	30	40-130	50-140
Aldrin	60-125	55-135	30	25-140	45-140
Heptachlor Epoxide	70-135	60-130	30	60-130	65-130
Endosulfan I	60-125	55-125	30	50-110	15-135
Dieldrin	70-**135**	**60-145**	30	60-130	65-125
4,4-DDE	70-135	**60-145**	30	35-140	70-125
Endrin	70-135	60-**145**	30	55-135	60-135
4,4-DDD	70-135	60-**145**	30	25-150	30-135
Endosulfan II	60-125	55-125	30	30-130	35-140
Endosulfan sulfate	70-135	**40-140**	30	55-135	60-135
4,4-DDT	70-135	60-**155**	30	45-140	45-140
Methoxychlor	75-140	65-145	30	55-150	55-145
Endrin ketone	70-135	65-**145**	30	75-125	65-135
Endrin aldehyde	70-135	**15**-110	30	55-135	35-145
alpha-Chlordane	70-135	**60-140**	30	65-125	65-120
gamma-Chlordane	70-135	**60**-140	30	60-125	65-125
Surrogates:					
Tetrachloro-m-xylene (TCX)	55-120	**55-130**	NA	25-140	70-125
Decachlorobiphenyl (DCB)	40-130	60-**135**	NA	30-135	55-130

[1] The limits in this table are those used as of the effective date of this SOP.
*[2] Any limits that appear in **bold** text are those where the in-house limit is outside of the DoD QSM limit.*
[3] Limits are taken from Appendix D of DoD QSM. If no limit listed in this table, no limit was listed in the DoD QSM.

Table 2A: Added Compound Accuracy and Precision Limits[1]

Analyte	In-House Limits[2] (%R)		Precision (RPD) (≤)	DoD QSM Limit[3]	
	Water	Solid		Water	Soil
2,4-DDE	50-150	50-150	30	NA	NA
2,4-DDD	50-150	50-150	30	NA	NA
2,4-DDT	50-150	50-150	30	NA	NA
Mirex	50-150	50-150	30	NA	NA

[1] Limits are default limits set in lieu of available recovery data for these compounds

Table 3: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 4: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
Breakdown Check Standard	Before initial calibration and each CCV.	Degradation ≤15% for Endrin and DDT each	Correct problem, reanalyze, repeat calibration.
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	See Section 10.3	Correct problem and repeat initial calibration.
ICV	After each initial calibration	%Difference ± 20% from expected value for each analyte	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±15%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs at different concentrations pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and bracketed samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	< RL DoD: ≤ ½ RL if analyte in any sample ≥ RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	Evaluated against control limits in Table 2, 1 Marginal Exceedance allowed.	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch SD: Per client request	Evaluated against control limits in Table 2	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate Spike	All field and QC samples	Evaluated against control limits in Table 2	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that data quality is known and documented. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Breakdown Check Standard (BCS): standard containing 4,4'-DDT and endrin analyzed to check for degradation problems. Presence of 4,4'-DDE, 4,4'-DDD, endrin ketone or endrin aldehyde indicates breakdown.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: a substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

The qualifiers that are routinely used for this test method are:

U: Compound analyzed for but not detected at a concentration above the reporting limit.
J: Estimated Value
P: There is greater than 40% relative percent difference for detected concentrations between two GC columns.
C: Positive result whose identification has been confirmed by GC/MS
B: Compound is found in the sample and the associated method blank.

E: Compound whose concentration exceeds the upper limit of the calibration range.
D: Concentration identified from a dilution analysis.
X,Y,Z: Laboratory defined flags that may be used alone or combined as needed. If used, provide a description of the flag in the project narrative.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are only produced in this range and qualified with the proper data reporting flag when a project requires this type of data reporting.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Appendix B: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in hexane using Class A volumetric glassware and Hamilton syringes. Stock standards must be replaced after one year or sooner if QC tests indicate a problem. For working standards, unless otherwise specified, assign an expiration date of 6 months from date of preparation unless the stock standard expires sooner in which case the earliest expiration date is used. See laboratory SOP BR-QA-002 *Standard & Reagent Preparation* for further guidance. All standards are made in hexane, unless otherwise specified. Standards should be stored in a refrigerator in PTFE-sealed containers.

Intermediate 2,4-DDX Standard

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
2,4-DDX	Restek #32098	2,4-DDD	1000	0.5	50	10
	Restek #32098	2,4-DDE	1000	0.5		10
	Restek #32200	2,4-DDT	1000	0.5		10

Prepared in Acetone

Intermediate Technical Chlordane Standard

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Technical Chlordane	Restek#32021	Technical Chlordane	1000	0.40	40	10

Intermediate Toxaphene Standard

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Toxaphene	Restek #32005	Toxaphene	1000	1.0	20	50

Surrogate Stock Solution

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Pesticide Surrogate	Restek #3200	TCX and DCB	1000	0.40	40	10

Mirex Stock Solution

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Mirex	Ultra # PST-720S	Mirex	100	1.0	10	10

WORKING STANDARDS

Breakdown Check Standard (BCS) – Varied Concentrations

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
Pesticide Performance Evaluation Mix	Ultra #CLP-250	Alpha-BHC	10	0.10	100	10
		Beta-BHC	10			10
		Gamma-BHC	10			10
		4,4-DDT	100			100
		Endrin	50			50
		Methoxychlor	250			250
		TCX	20			20
		DCB	20			20

ICV – Second Source Standard – Varied Concentrations

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
Pesticide Mix A	Ultra # CLP-216	Apha-BHC	5	0.40	100	20
		4,4'-DDT	10			40
		Gamma-BHC	5			20
		Dieldrin	10			40
		Endrin	10			40
		Methoxychlor	50			200
		4-4'-DDD	10			40
		Heptachlor	5			20
		Endosulfan I	5			20
		TCX*	5			20*
		DCB*	10			40*
Pesticide Mix B	Ultra # CLP-226B	Heptachlor Epoxide	5	0.40		20
		Endosulfan Sulfate	10			40
		Aldrin	5			20
		Beta-BHC	5			20
		Delta-BHC	5			20
		Endosulfan II	10			40
		alpha-chlordane	5			20
		gamma-chlordane	5			20
		Endrin Ketone	10			40
		4-4'-DDE	10			40
		Endrin Aldehyde	10			40
		TCX*	5			20*
		DCB*	10			40*

*Compounds present in multiple standard solutions.

Column Prime Standard – Varied Concentrations

Parent Standard	Vendor	Component	Parent Standard Concentration	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)

			(mg/L)			
Organochlorine Pesticide Mix AB #2	Restek #32292	Alpha-BHC	8.0	1.0	20	400
		Beta-BHC	8.0			400
		Delta-BHC	8.0			400
		Gamma-BHC	8.0			400
		Heptachlor	8.0			400
		Aldrin	8.0			400
		Heptachlor Epoxide	8.0			400
		Endosulfan I	8.0			400
		Dieldrin	16			800
		4-4'-DDE	16			800
		Endrin	16			800
		Endosulfan II	16			800
		4-4'-DDD	16			800
		Endosulfan Sulfate	16			800
		4,4'-DDT	16			800
		Methoxychlor	80			4000
		Endrin Ketone	16			800
		Endrin Aldehyde	16			800
		gamma-chlordane	8.0			400
		alpha-chlordane	8.0			400
Surrogate Stock Solution	Laboratory Prepared	TCX and DCB	10	0.80		400

CALIBRATION STANDARDS

INDAB-5: Pesticide Calibration Level 5 – Varied Concentrations

Parent Standard	Vendor	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
Organochlorine Pesticide Mix AB #2	Restek #32292	8-80	1.0	100	80-800
Mirex Stock Standard	Laboratory Prepared	10	1.6		160
Surrogate Stock Solution	Laboratory Prepared	10	0.80		80

INDAB Calibration Standard(s): CAL Levels 1- 4 - Varied Concentrations

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
INDAB-5	INDAB-4	80-800	20	40	40-400
INDAB-5	INDAB-3	80-800	10	40	20-200
INDAB-5	INDAB-2	80-800	5.0	40	10-100
INDAB-5	INDAB-1	80-800	2.5	40	5-50

Individual Pesticide Concentrations in INDAB Levels 1-5

Analyte*	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)
TCX (surrogate)	5	10	20	40	80
DCB (surrogate)	5	10	20	40	80
Alpha-BHC	5	10	20	40	80

Beta-BHC	5	10	20	40	80
Delta-BHC	5	10	20	40	80
Gamma-BHC	5	10	20	40	80
Heptachlor	5	10	20	40	80
Aldrin	5	10	20	40	80
Heptachlor Epoxide	5	10	20	40	80
Endosulfan I	5	10	20	40	80
Dieldrin	10	20	40	80	160
4-4'-DDE	10	20	40	80	160
Endrin	10	20	40	80	160
Endosulfan II	10	20	40	80	160
4-4'-DDD	10	20	40	80	160
Endosulfan Sulfate	10	20	40	80	160
4,4'-DDT	10	20	40	80	160
Methoxychlor	50	100	200	400	800
Endrin Ketone	10	20	40	80	160
Endrin Aldehyde	10	20	40	80	160
gamma-chlordane	5	10	20	40	80
alpha-chlordane	5	10	20	40	80
Technical Chlordane	50	100	200	400	800
Toxaphene	500	1000	2000	4000	8000

2,4-DDX-5: Pesticide Calibration Level 5

Parent Standard	Vendor	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
2,4-DDX	Laboratory Prepared	10	1.6	100	160
Surrogate Stock Standard	Laboratory Prepared	10	0.80		80

2,4-DDX Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
2,4-DDX-5	2,4-DDX-4	160	20	40	80
2,4-DDX-5	2,4-DDX-3	160	10		40
2,4-DDX-5	2,4-DDX-2	160	5		20
2,4-DDX-5	2,4-DDX-1	160	2.5		10

Toxaphene Working Standard - Calibration Level 5

Parent Standard	Vendor	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
Toxaphene Intermediate Standard	Laboratory Prepared	50	16	100	8000
Surrogate	Laboratory Prepared	10	0.80		80

Toxaphene Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
Toxaphene Calibration Level 5	Toxaphene Level 4	8000	20	40	4000
Toxaphene Calibration Level 5	Toxaphene Level 3	8000	10	40	2000
Toxaphene Calibration Level 5	Toxaphene Level 2	8000	5.0	40	1000
Toxaphene Calibration	Toxaphene Level 1	8000	2.5	40	500

Level 5					

Technical Chlordane Working Standard - Calibration Level 5

Parent Standard	Vendor	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
Technical Chlordane Intermediate Standard	Laboratory Prepared	10	8.0	100	800
Surrogate	Laboratory Prepared	10	0.80		80

Technical Chlordane Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
Technical Chlordane Calibration Level 5	Technical Chlordane Level 4	800	20	40	400
Technical Chlordane Calibration Level 5	Technical Chlordane Level 3	800	10	40	200
Technical Chlordane Calibration Level 5	Technical Chlordane Level 2	800	5.0	40	100
Technical Chlordane Calibration Level 5	Technical Chlordane Level 1	800	2.5	40	50

Appendix C: Equations

Calibration Factor (CF_X)

$$CF_x = \frac{\text{Peak area or height (x)}}{\text{Standard concentration (ug/L)}}$$

Mean Calibration Factor ($\overline{CF}$)

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where:
n = number of calibration levels

Standard Deviation of the Calibration Factor (SD)

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)2}{n-1}}$$

Where:
n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Calibration Factor

$$\%RSD \text{ of the Calibration Factor} = \frac{SD}{\overline{CF}} \times 100\%$$

Percent Difference (%D)

$$\%D = \frac{CF_v - \overline{CF}}{\overline{CF}} \times 100\%$$

Where:
CF_v = Calibration Factor from the Continuing Calibration Verification (CCV)

Percent Drift

$$\text{Percent Drift} = \frac{\text{Calculated Concentration - Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100\%$$

Percent Recovery (%R)

$$\%R = \frac{C_s}{C_n} \times 100\%$$

Where:
C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD

$$\%R\ (MS/MSD) = \frac{C_s - C_u}{C_n} \times 100\%$$

Where:
C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD)

$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$$

Where:
C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$$C_{extract}\ (ug/L) = \frac{\text{Peak Area (or Height)}}{\overline{CF}}$$

Note: The concentrations of the 3-5 peaks chosen for quantificaton is calculated and the average is then taken for final calculation.

Water

$$C_{sample}\ (ug/L) = C_{extract}\ (ug/L) \times \frac{\text{extract volume (L)}}{\text{sample volume (L)}} \times DF$$

Solid

$$C_{sample}\ (ug/Kg) = C_{extract}\ (ug/L) \times \frac{\text{extract volume (L)}}{\text{sample weight (Kg)}} \times \frac{100}{\%\ \text{solids}} \times DF$$

% DDT Breakdown

$$\%\,\text{DDT Breakdown} = \frac{\text{Sum of peak area or height (DDE} + \text{DDD)}}{\text{Sum of peak area or height (DDE} + \text{DDD} + \text{DDT)}} \times 100\%$$

% Endrin Breakdown

$$\%\,\text{Endrin Breakdown} = \frac{\text{Sum of peak area or height (Endrin aldehyde} + \text{Endrin ketone)}}{\text{Sum of peak area or height (Endrin aldehyde} + \text{Endrin ketone} + \text{Endrin)}} \times 100\%$$

Appendix D: Marginal Exceedance Evaluation for LCS

When the number of analytes spiked into an LCS increase, the statistical likelihood of random failure increases. The ME evaluation is a process used by the laboratory to assess for random LCS failure to determine subsequent needs for corrective action.

Corrective action for LCS may not be required when all 3 of the following conditions are met:

- The %R exceptions for each analyte that did not meet criteria in the LCS are random.
- The %R for each analyte that did not meet criteria is within the ME limits.
- The number of analytes in the LCS that were not within control limits are within the allowable number of marginal exceedance based on the number of analytes spiked into the LCS.

If each of the above conditions is met, then in the absence of no other QC failure or indicator of bias in the system, the LCS failure may be deemed random and the laboratory has sufficient technical justification to not perform corrective action.

A marginal exceedance (ME) is defined as a percent recovery of an analyte in the LCS that is outside the upper or lower control limit but within the ME Limit.

ME limits are 4 standard deviations around the mean and are calculated as follows:

$$\text{ME Upper Limit} = \left(\frac{UpperControlLimit - LowerControlLimit}{6} \right) + UpperControlLimit$$

$$\text{ME Lower Limit} = \left(\frac{UpperControlLmit - LowerControlLimit}{6} \right) - LowerControlLimit$$

When an LCS is outside control limits but within ME limits corrective action may not be necessary so long as the 3 conditions listed above are met.

The number of allowable exceedance is based on the number of analytes spiked into the LCS, the higher the number of analytes spiked, the higher the number of allowable exceedance.

For example:

The upper and lower control limits for a target analyte are 70-130. Using the equations above the ME limit calculates to 60-140.

If the %R for this analyte in an LCS is 65%, the %R is outside acceptance criteria but it is within the ME limit so this failure should be evaluated for marginal exceedance to determine if the other conditions for marginal exceedance are met.

If the %R for this analyte in an LCS is 59%, the %R is outside acceptance criteria and outside the ME limit so one of the conditions for ME are not met and corrective action for the failing LCS recovery is required.

The other conditions are as follows:

- The marginal exceedance must be random. The condition for random is met when the percent recovery of the same analyte is not outside the upper or lower control limit in 2 out of 3 consecutive LCS. If the %R is outside control limits for the same analyte in consecutive LCS, this condition is not met.

- The percent recovery for the analyte must be within the calculated ME limit.

- The total number of analytes in the LCS that were not within acceptance criteria must be within the total allowable number of marginal exceedance. The total number of allowable exceedance is provided in the table below. Please note that the number of exceptions allowed is based on the total number of analytes spiked into the LCS. The ME evaluation is not based on the number of analytes reported to the client or the number of analytes in the client's sublist.

The number of allowable exceedances based on the number of analytes spiked into the LCS is:

# Analytes Spiked into LCS	Allowable Number of ME
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

If each of the above conditions is met, then in the absence of no other QC failure or indicator of bias in the system, the LCS failure may be deemed random and the laboratory has sufficient technical justification to not perform corrective action.

If each of these conditions is not met, the LCS failure is not considered random and corrective action is required. If corrective action is not taken, the justification for not taking corrective action cannot be attributed to the "marginal exceedance allowance".

The ME evaluation does not negate a nonconformance (NCR) - it provides technical justification to not perform corrective action such as re-extraction or reanalysis.

Use of the Marginal Exceedance Rule must be documented as follows:

1) Initiate a NCR to document the LCS failure.

2) In the column for NCR Situation, record the appropriate NCR option for LCS failure.

3) In the second column "Reason" insert the option code for "OTHER".

4) Record the # of samples and the LAB IDs of the samples affected by the NCR.

5) In the Corrective Action Column, insert the option code for "PROCEED"

6) In the second column "Reason" insert the option code for "OTHER".

7) In the third column “Result” insert the option code for “Not Applicable”.

8) Insert the following blurb In the Explanatory Comment Section:

The %R for analytes (X,Y,Z) were not within the upper or lower control limits of (X,Y,Z) for LCS (insert LCS ID). A marginal exceedance evaluation was performed and the LCS exceptions met the 3 conditions for random failure, thus corrective action was not performed.

The PM should include the following statement in the Project Narrative:

The %R for analytes (X,Y,Z) were not within the upper or lower control limits of (X,Y,Z) for LCS (insert LCS ID). A Marginal exceedance evaluation was performed and the LCS exceptions were found to be attributed to random failure thus corrective action was not performed.

Summary: ME evaluation is a decision making process used to determine needs for corrective action. ME allowances do not replace the upper or lower acceptance limits for the LCS or negate the nonconformance for LCS exceptions.

Each of 3 conditions must be met in order to use ME allowance as technical justification to not take corrective action.

The ME evaluation, when all three conditions are met, provides technical justification for not taking corrective action for the LCS failure because in the absence of other QC failures or problems with the measurement system, the LCS failure is likely an anomaly attributed to the number of analytes spiked into the LCS.

SOP Change in Progress Attachment (CIPA)

SOP Number	SOP Title	SOP Revision	SOP Effective Date	CIPA Effective Date
BR-SM-001	Sample Management	10	09/27/10	01/20/11

The following revisions were made to this standard operating procedure (SOP). These changes are effective as of the CIPA Effective Date. Changes to this document will be incorporated into the document with the next revision. This document change is authorized and issued by the laboratory's QA Department.

Page 8 of 10, 6th Paragraph, Section 5.4.4 Sample Disposal

Insert the text in bold into the following paragraph:

Unless otherwise specified by project or regulation, samples, extracts and digestates are retained for 30 days from the date of invoice and then disposed of following applicable regulations in accordance with laboratory SOP BR-EH-001 for hazardous waste management. The disposal of unused original sample containers is documented in the sample disposal **record. Soil and mixed waste samples designated as foreign source must be disposed of as specified in the compliance agreement. As of the publication date of this CIPA, all samples are incinerated by the laboratory's waste hauler. If the laboratory discontinues the incineration service with the waste hauler, prior to disposal the unused sample portion of the foreign source sample must be heated in a muffle furnace at 250°F for a minimum of 2 hours.**

Title: Sample Management

Approval Signatures

William S. Cicero
Laboratory Director

Steve Timmons
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Kirstin L. McCracken
Quality Assurance Manager

Bryce E. Stearns
Technical Director

Daniel W. Helfrich
Health & Safety Director

Approval Date: September 27, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 PURPOSE

The purpose of this SOP is to ensure that sample integrity and custody are maintained and documented from laboratory receipt to disposal.

This facility does not offer field sampling services therefore procedures for sampling is not included in this SOP. General laboratory procedures for sub sampling are described in laboratory SOP BR-QA-020 and various test method SOPs.

2.0 SCOPE AND APPLICATION

This procedure is applicable to all samples received in the laboratory. Project specific protocol may replace or supplement sections of this SOP as necessary.

The laboratory maintains a sample acceptance policy that describes the conditions under which samples are accepted or rejected. Any compromised samples, nonconformance, abnormality or departure from the laboratory sample acceptance policy is documented, the client is contacted for resolution and/or the situation is noted in the project narrative.

When samples are received in the laboratory, the condition of the shipping container is inspected and the temperature of samples that require thermal preservation is checked with an infrared (IR) thermometer to verify that the arrival temperature is within specified criteria. The containers are unpacked and checked against the chain-of-custody provided. Chemical preservation of inorganic samples is also checked at this time whereas the preservation of samples for volatile and extractable organics is checked in the respective analytical department prior to sample preparation or analysis. Anomalies, observations or exceptions to the laboratory's sample acceptable policy are documented in the job record along with any decisions to proceed or cancel analysis.

Samples are logged into the Laboratory Management Information System (TALS) and subsequently tracked to disposition by the LIMS. A LIMS job is created for each set of samples received and a unique laboratory ID is assigned to each sample and container and placed on each container using a durable label. The job provides the essential information to the laboratory operation for all subsequent laboratory activities including the client and name of project, date and time of laboratory receipt, link between the sample ID assigned by the client and the LAB ID, requested analyses by test method, reporting requirements, deliverable dates, invoicing specifications and the requirements for sample retention and disposal.

Samples are stored in the laboratory in accordance with the environmental conditions specified by the test method. Samples that require thermal preservation are stored under refrigeration and conditions are monitored and recorded daily by laboratory personnel. The laboratory is a secure facility. Entrance doors are locked and access to the facility is controlled by card access entry or by laboratory personnel.

Samples, extracts, digestates, leachates or any other sample preparation output is disposed of in accordance with the laboratory's SOP for hazardous waste in accordance with applicable Federal, State and local regulations.

3.0 SAFETY

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must

be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

Each sample received in the laboratory should be regarded as a potential health hazard and exposure should be minimized as reasonably possible. Material Safety Data Sheets (MSDS) for reagents and chemical are available to all personnel and the sample management staff should read the MSDS for the chemicals used to preserve samples before they handle such samples. Safety glasses, latex gloves and protective clothing (lab coat) should be worn while performing tasks outlined in this SOP.

Coolers that exhibit a strong odor or are suspected to contain highly contaminates samples should be opened in a fume hood.

4.0 DEFINITIONS

4.1 A list of general laboratory terms and definitions is given in Appendix A.

5.0 PROCEDURE

5.1 Bottle Order Requests & Transport to Sampling Location

The laboratory provides sample containers and related materials such as ice packs, packing material, chain-of-custody forms (COC) and custody seals to any client upon their request. Bottle order requests are initiated by a laboratory project manager (PM) and forwarded to sample custodians who compile and pack the sample containers for shipment to the location designated by the client using the procedures specified in laboratory SOP BR-SM-004.

5.2 Sample Collection

The laboratory does not perform field sampling services. The following sections present general guidelines that should be used by those responsible for sample collection. To ensure valid data, it is necessary for sample collectors to be trained, familiar with, and proficient with proper sampling technique for the collection of samples. The laboratory strongly recommends that our customers establish a written sampling protocol that includes specific sampling instructions and documentation requirements for each type of sample collected for each project.

All sample collection records should be made in indelible ink and should identify the person responsible for each activity including transport of the samples to the laboratory.

At the time of collection the following information should be recorded for each sample:

- Sample ID
- Public Water System ID (where applicable)
- Date and Time of Sample Collection
- Sample Type (i.e., compliance, confirmation, etc.)
- Analysis Method Required
- Sample Container Type, Size, and Preservative Type and Concentration
- Sample Volume or Weight Collected
- Name of Sampler and Contact Information
- Sampling Location (where applicable)

- Comments

After sample collection, the samples should be preserved, labeled and packed for shipment to the laboratory. Unless thermal preservation is not required, all samples should be iced at the time of collection to a temperature of 0-6°C, without freezing, unless freezing is the recommended thermal preservation technique. A fixed label should be placed on each sample container and it should include the following information; sample identification, preservative, date and time of collection, collector's initials, and the requested test methods.

The sample containers should be placed in the shipping cooler along with a chain-of-custody record and any other pertinent documentation. The sample containers must be packed to prevent breakage and to comply with any applicable Department of Transportation (DOT) regulations, and all paperwork should be sealed in plastic as protection from moisture. If ice is used in shipping, this should be sealed in plastic so as to contain melt water. The shipping cooler should be sealed in such a manner that any evidence of tampering would be readily evident upon receipt in the laboratory. The transfer of possession of samples from the field custodian to the shipper must be documented on the chain-of-custody record.

5.3 Sample Acceptance Policy & Sample Receipt Protocol

5.3.1 Sample Acceptance Policy

The sample management department is staffed Monday-Friday from 8:00AM - 5:30PM and from 8:00AM - 5:00PM on Saturday. During these times a sample custodian is available to accept delivery from commercial carriers (FED EX, UPS) and/or courier service. The laboratory can also make arrangements to accommodate sample delivery outside this time schedule provided that sufficient advance notification is given. Samples received after normal business hours are received by designated personnel then placed in a refrigerated storage area in order to maintain thermal preservation if thermal preservation is required.

The receipt of samples is acknowledged on the chain of custody (COC) form with the signature and date/time of the sample custodian. The condition of samples upon receipt is documented on checklists designated for this purpose. Any deficiencies identified during sample receipt are recorded and communicated to the laboratory project manager (PM), who will contact the client and fully document any decision to proceed with analysis in the project record. Consultation with the client should be immediate and timely (next business day or as specified in the project plan). Correspondence records and/or records of conversations concerning the decision to proceed with analysis and/or the disposition of rejected samples are maintained in the project record, and should be maintained in association with the sample receipt checklist. All data associated with samples that did not meet the sample acceptance criteria must be qualified with a Non-Conformance Memo (NCM) and/or noted in the project narrative that accompanies the final test report.

Sample receipt is considered deficient when the following conditions are observed:

- Shipping cooler and/or samples are received outside the temperature specification
- Sample bottles are received broken or leaking
- Samples are received beyond holding time
- Samples are received without the appropriate preservation
- Samples are not received in appropriate containers
- Chain of Custody does not match the samples received

- Chain of Custody was not received or is incomplete*
- Custody seals are broken
- Evidence of tampering with the cooler and/or samples
- Headspace in 40mL or 22 mL VOA vials
- Seepage of extraneous water or other material into the samples
- Inadequate sample volume
- Illegible, impermanent ink, or non-unique sample labeling
- One or more coolers missing from a multi parcel shipment
- Shipping container is damaged

**Complete documentation shall include sample identification, the location date/time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample.*

5.3.2 Sample Receipt & Login

Document the following activities on the checklist designated for this purpose.

Examine the cooler and record the presence/absence of custody seals. If custody seals are present, record their condition (intact, broken) and if custody seal numbers are present, record the number(s).

Open the cooler and inspect the sample containers. If any sample containers are broken or if an odor is detected, immediately close the cooler and transport the cooler to an available fume hood.

Check and record the temperature of the sample containers using a calibrated infrared thermometer (IR gun) to the nearest tenth of a degree. For samples that require thermal preservation, unless otherwise specified by client project plan or regulatory program, the arrival temperature is considered acceptable if it is within from 0.0-6.0°C. Samples that are hand delivered to the laboratory on the same day of collection may not meet the criteria. In these cases, the thermal preservation of the samples is considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

Remove the samples from the cooler. Check to ensure that all samples listed on the chain-of-custody (COC) were received. Verify that the sample labels match the description given on the COC. Record the presence/absence of sample tags and if present, record the sample tag number(s).

Dispose of the packing materials and dry the inside of the shipping cooler. If the cooler has an odor or otherwise appears dirty, wash the cooler with warm water and a suitable detergent such as Liqui-Nox diluted 1:100 with reagent water. Thoroughly rinse and dry the cooler. If washing does not seem effective, notify the department manager who will determine if the shipping cooler should be disposed of.

Check the information on the COC against the sample container labels. Check the test methods listed on the COC against the methods specified in the LIMS project assigned to the sample set. The test methods requested on the COC should agree with those assigned in the project. If the methods do not match, contact the PM for resolution. If a LIMS project has not been created, immediately notify the PM and request that he/she create the project.

Check and record the chemical preservation (pH) of inorganic samples. The chemical preservation of organic and volatile samples and the presence of residual chlorine are checked during sample preparation or prior to analysis.

To check the chemical preservation: Invert the sample to mix, and remove the sample container cap. Using a clean disposable pipette, transfer a drop of sample from the container to a narrow range pH indicator strip. Compare the color change on the indicator strip to the pH chart. If the pH is not within specification, notify the PM who must contact the client for further instruction. If instructed by the client to do so, add preservative to the samples with inadequate preservation. Record any pH adjustments along with the date of preservation, your initials and the lot number of the preservative added. *Samples designated for metals analysis should not be digested within 24 hours of preservation. If a pH adjustment is performed, flag the samples with the time of preservation and notify the metals department that a pH adjustment was performed.*

Document all instances of compromised sample receipt, notify the PM and wait for further instruction. Any decisions to proceed or cancel analysis must be documented by the PM.
Log the samples into TALS (LIMS database). The LIMS maintains a permanent record of all laboratory activities from sample receipt to disposition including (but not limited to): the client / project name, date of laboratory receipt and name of receipt personnel, the date of login and person that performing the log-in, laboratory ID codes assigned to the sample set and the link to associated client sample IDs as well as with the matrix of the samples, the date/time of sample collection, requested test methods, reporting specifications, billing information, and sample disposition requirements, deliverable due dates and all other information for the project samples. The checklists used to record the condition of samples on receipt are also maintained in the LIMS database.

For instructions on how to login samples refer to the instruction set prepared by the TALS IT staff. To view the current instruction set, open the company intranet website “Oasis”, select Information Technology from the Department List, select TALS LIMS from the Navigation column, then select User Documentation. Open the folder for Sample Management and select “Login”. Additionally each TALS module includes a Help radio button that should link to the user documentation but as of the publication date of this SOP, the radio buttons are not active in all modules. Try the help radio button first and if not active, go to the website.

Due to limitations with the LIMS the following characters should not be used in client sample IDs. : \ / ? * [or]. If you observe these characters in a client sample ID, immediately notify the PM so they can contact the client for resolution.

When login of the job is complete, assemble the original sample receipt paperwork and put the paperwork in the bin designated for this purpose. Laboratory PMs or their designee must review the sample receipt paperwork and the LIMS job for completeness and correctness and approve the login before the job will be at ready status for the various analytical sections. Log-in review should be completed within 24 hours of sample receipt so laboratory activities may begin in a timely manner.

Place the samples is the designated storage area.

If the samples are not logged in on the date of sample receipt, record the following project information in the Outstanding Log-In Spreadsheet.

- Date Received

- Client Name
- Project Manager
- Project Name
- Methods Requested
- Storage Location

Photocopy the chain of custody (COC) and record your initials, the storage location and the date the samples were placed in storage on the COC. Place the samples in the designated storage along with the original sample receipt paperwork and give the photocopied COC to the Department Supervisor. Each morning check the Outstanding Log-In Spreadsheet and complete the log-in. When log-in is complete enter the date of log-in into the spreadsheet then hide the row from view so that only outstanding log-ins appears on the screen. The Department Supervisor and/or Department Manager will check the spreadsheet against the photocopied COC to ensure that log-in of a sample shipment is not missed.

5.4 Sample Tracking, Storage & Security

5.4.1 Sample Tracking

Each sample is assigned a unique laboratory ID and a unique container ID to distinguish individual containers when multiple containers with the same sample ID are received. The laboratory ID is a sequential number that is printed on a durable bar coded laser label and affixed to each sample container. Output container codes appended to the LAB ID are further used to uniquely indentify each container for subsequent extracts, lecahates, digestates or other types of samples preparation. The IDs are bar coded. During laboratory activities the bar code is scanned into prep, acquisition and analytical batches stored in the LIMS. The status of the sample can be checked at any time in real time from any LIMS PC by reviewing the job to which the sample is assigned.

5.4.2 Sample Storage

Unless otherwise specified by client or regulatory program, samples are stored according to the conditions specified in the test method. Samples received for metals, air and geotechnical testing may stored at ambient temperature or under refrigeration. Samples received for wet chemistry and organic analyses are stored under refrigeration at a temperature of 4 ±2°C or kept frozen at < –10°C. The storage requirements for each method are specified in test method SOPs. All samples are stored away from standards, reagents, or other potentially contaminating sources. Samples designated for volatile organic analysis (VOA) are segregated from other samples. Storage blank are maintained with VOA samples and routinely analyzed to verify that no cross contamination has occurred. Subsequent digestates and extracts are stored under the same thermal conditions but are stored away from sample containers, standards, reagents, or other potentially contaminating sources.

Temperature measurements are recorded daily using NIST traceable thermometers. The accuracy of the thermometers is checked annually by the QA Department against an NIST traceable thermometer.

5.4.3 Sample Security & Internal Chain of Custody

For most projects a secure facility and monitored access to the laboratory is sufficient to ensure the integrity of samples. Access to the laboratory is controlled by measures that include locked doors, electronic access codes and a staffed reception area. All visitors must sign into the visitor registry and while in the facility, they are escorted by laboratory personnel.

Laboratory activities are recorded with a date and time stamp along with the person that performed the activity and this level of documentation is typically sufficient for most projects.

If needed for a project the laboratory will maintain a continuous record of sample and extract/digestate transfer within the laboratory (ICOC) on client request per the conditions agreed upon with the laboratory. To initiate ICOC protocol, the requirement for ICOC must be designated by the PM during job login to alert the sample management staff to initiate the ICOC record.

ICOC is recorded on client specific or laboratory created forms designated for this purpose. ICOC may also be recorded using the TALS ICOC module. Instructions for completion of the ICOC record are provided on the form. Gaps in the ICOC record must be documented using a TALS nonconformance memo (NCM) and reported to the client by the PM in the project narrative.

5.4.4 Sample Disposal

Unless otherwise specified by project or regulation, samples, extracts and digestates are retained for 30 days from the date of invoice and then disposed of following applicable regulations in accordance with laboratory SOP BR-EH-001 for hazardous waste management. The disposal of unused original sample containers is documented in the sample disposal.

6.0 RESPONSIBILITIES

All laboratory employees are responsible for following the procedures given in this SOP.

7.0 REFERENCES / CROSS-REFERENCES

- Constitution, Bylaws, and Standards National Environmental Laboratory Accreditation Conference, Current NELAC Voted Revision. USEPA Office of Research and Development.

- Department of Defense Quality System Manual for Environmental Laboratories, Final Version 4.1. DOD Environmental Data Quality Workgroup.

8.0 ATTACHMENTS

Appendix A: List of Terms & Definitions

9.0 REVISION HISTORY

BR-SM-001, Rev. 9:

Title Page: Updated approval signatures
All Sections: Revised to current practice with new LIMS system.

BR-SM-001, Rev. 8:

Title Page: Updated Approval Signatures
Entire SOP: Converted into new template.
Section 5.0: Changed hours of operation.

Appendix A: Terms & Definitions

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in required documents. (DOD / ASQC)

Chain of Custody Form: a record that documents the possession of samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection, collector, time of collection and requested analyses (NELAC).

Client: The party that has agreed to pay the bill for services rendered by the laboratory and with whom the laboratory has a contractual relationship for the project (DOD).

Compromised Sample: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers or exceeding holding times when delivered to the laboratory. (NELAC)

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

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Title: Chlorinated Pesticides [Method No. 8081A & 8081B]

Approvals (Signature/Date):

Dennis Jonsrud — 10-10-12 — Date Technical Specialist	Adam Alban — 10 Oct 12 — Date Health & Safety Manager / Coordinator
John Morris — 10/10/12 — Date Quality Assurance Manager	Robert C. Hanisch — 10/12/12 — Date Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the determination of chlorinated pesticides using the methodology described in EPA SW-846 Method 8081A and 8081B with 8000B or 8000C as specified by project requirements.

1.2 This SOP is applicable to the gas chromatographic (GC) analysis of extracts of soil and water samples. Table 1 lists the compounds that can be determined by this method and their associated routine reporting limits (RLs).

1.3 This SOP does not include the procedures for extracting soil and water samples. Refer to the following SOPs for sample extraction procedures:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW-846 3510C
DV-OP-0007	Concentration of Organic Extracts, SW-846 3510C, 3520C, 3540C, and 3550C
DV-OP-0016	Ultrasonic Extraction of Solid Samples by SW-846 3550C
DV-OP-0015	Microwave Extraction of Solid Samples by SW-846 3546
DV-OP-0023	Extraction of Aqueous Samples by Microextraction (LVI)

1.4 Analytes, Matrix(s), and Reporting Limits

See Table 1 for analytes and reporting limits by matrix.

1.5 This SOP contains a Large Volume Injection (LVI) procedure. This procedure has not been approved by the State of South Carolina and therefore no samples from South Carolina may be analyzed using LVI.

2.0 Summary of Method

2.1 Sample Preparation

2.1.1 Chlorinated pesticides are extracted from a one-liter water sample with methylene chloride using a separatory funnel (Method 3510C). Detailed instructions are given in SOP DV-OP-0006. The methylene chloride extract is exchanged to hexane as described in SOP DV-OP-0007. An alternate procedure has been developed using a lower volume of sample (35 ml) and a larger injection volume in order to minimize shipping requirements and conserve the reagents needed for extraction.

2.1.2 Chlorinated pesticides are extracted from a 30-gram soil subsample into a 50:50 acetone-methylene chloride solution by sonication (Method 3550C) or by microwave extraction (Method 3546). The extract is dried and exchanged to hexane. Detailed instructions are given in SOPs DV-OP-0016 and DV-OP-0015.

2.1.3 SOP DV-OP-0007 provides instructions for the concentration and cleanup of sample extracts. Florisil is used to clean extracts that show color. Sulfur is removed if observed. All extracts are in hexane and the final extract volume is 10 mL.

2.2 Analysis

2.2.1 Samples are analyzed using a gas chromatograph equipped with dual columns and dual electron capture detectors (ECDs).

2.2.2 The instrument is calibrated using external standards. Compounds are identified by their retention time on the columns.

2.2.3 Positive results from the primary column are confirmed with a second, dissimilar column. The laboratory maintains a total of four dissimilar columns for additional confirmation capability.

3.0 Definitions

3.1 Single-Component Pesticides: A pesticide formulation that consists of a single chemical compound. Most of the analytes determined by this procedure are single-compound pesticides.

3.2 Multi-Component Pesticides: A pesticide formulation that consists of more than one chemical compound. Toxaphene and Technical Chlordane are production mixtures of multiple compounds. Toxaphene is manufactured by the chlorination of camphenes, which produces a variety of compounds, not all of which are chromatographically resolved. Technical Chlordane is produced by the chlorination of a mixture of camphenes and pinenes.

3.3 Chlordane: As just described, Technical Chlordane (CAS# 12789-03-6) is a mixture of compounds. Method 8081A, Section 7.6.2 and Method 8081B, Section 11.6.2 note that Technical Chlordane includes at least 11 major components and 30 minor components, and adds "the exact percentage of each [*cis*-chlordane and *trans*-chlordane] in the technical material is not completely defined, and is not consistent from batch to batch." The laboratory has found that manufacturing lots of Technical Chlordane produced at different times or at different production facilities have different ratios of the key components. For this reason, it is more common to analyze for the major components of technical Chlordane (α-Chlordane, γ-Chlordane, and heptachlor) instead of analyzing for the total mixture. For the purpose of reporting results under this SOP, the following compounds are reported. Alpha-chlordane (*cis*-chlordane) CAS # 5103-71-9 and gamma-chlordane (*trans*-chlordane) CAS # 5103-74-2. The laboratory may also report chlordane (not otherwise specified) or, n.o.s under CAS# 57-74-9.

3.4 The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and SOP DV-QA-003P.

4.0 Interferences

4.1 Contamination by carryover can occur when a low concentration sample is analyzed immediately following a high concentration sample. It is the laboratory's policy to reanalyze any samples that follow an unusually concentrated sample and that show detectable levels of the same compounds that appeared in the preceding concentrated sample.

4.2 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3 Sulfur will interfere, and, when observed, is removed using cleanup procedures described in SOP DV-OP-0007.

4.4 Soil and water sample extracts are subject to Florisil cleanup when the extracts have noticeable color or whenever there is clear evidence of interferences in the initial sample chromatograms. Florisil removes low- to medium-molecular weight polar organic interferences from sample extracts. One limitation for this cleanup method is that recoveries for the most polar compounds, endosulfan sulfate and endrin aldehyde in particular, will be lower. Florisil has been observed to remove the compound kepone and is not used where the determination of kepone is required. Instructions for performing Florisil cleanups can be found in SOP DV-OP-0007.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.1.4 The ECD contains a ^{63}Ni radioactive source. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the facility's radioactive material license. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Radiation Safety Officer shall be immediately notified and a letter sent to the Colorado Department of Public Health and Environment. Follow the proper procedures and precautions for the safe handling of radioactive materials when handling the ECDs in the event that leakage may have occurred.

5.1.5 As a safety precaution, all standards, samples, and extracts are handled in an approved fume hood.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects are exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 An analytical system complete with a gas chromatograph and dual ECD (Ni-63) detectors is required. A data system capable of measuring peak area and/or height is required.

6.2 An analytical balance capable of weighing to 0.01 g.

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls for the current software and hardware to be used for data processing.

6.4 Columns

6.4.1 Primary Column: CLPI, 30 m X 0.32 mm id

6.4.2 Secondary Column: CLPII, 30 m X 0.32 mm id

6.4.3 Additional columns that can be used for confirmation include 30 m X 0.32 mm id RxiSil 35-MS or Rxi-XLB.

6.5 Autosampler vials, crimp-top cap with PTFE-faced septa

6.6 Y-splitter, septa, guard columns, ferrules, Siltek injection port liners, Siltek glass wool.

6.7 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.8 Class A volumetric flasks various sizes.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagents

7.1.1 Hexane, pesticide grade; each lot tested for purity prior to use per SOP CA-Q-S-001.

7.1.2 Carrier gas, ≥ 99.99999% pure hydrogen or helium

7.1.3 Make-up gas, ≥ 99.99980% pure nitrogen

7.2 Standards Verification

All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

7.3 Storage of Stock Standards

7.3.1 Standards are purchased from commercial vendors and are received as certified solutions in flame sealed ampoules. Neat stocks with applicable certification may also be used. Stock standards are stored refrigerated at ≤ 6 ºC. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

7.3.2 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem. Kepone in particular may demonstrate signs of degradation faster than the other compounds, and/or the expiration date. Endosulfan I and II appear to degrade in the presence of methanol. gamma-BHC appears to degrade in the presence of acetone.

7.4 Calibration Stock Standards

NOTE: The availability of the specific commercial standard solutions upon which the following sections are based may change at any time. As a result, it may be necessary to alter the dilution scheme presented herein to accommodate changes in stock standard concentrations. All such changes are documented in the standards preparation records.

7.4.1 Routine Pesticide AB Mix Stock Standard, 1,000 µg/mL

The routine pesticide AB mix stock standard, Accustandard M-8081-C, contains

all of the "routine" single-component pesticides, as identified in Table 1 with the addition of Hexachlorobenzene at 100 µg/mL (Accustandard APP-9-112), Mirex at 100 µg/mL (Accustandard P-066S) and Isodrin at 1000 ug/mL (Accustandard P471S-10x).

7.4.2 Surrogate B Mix Stock Standard, 200 µg/mL

The surrogate B mix stock standard contains decachlorobiphenyl (DCB) and tetrachloro-*m*-xylene (TCMX). Accustandard CLP-032-R.

7.4.3 Toxaphene Stock, 100 µg/mL

The Toxaphene stock standard contains a specific production mixture of Toxaphene. This mixture does not necessarily match all possible production mixtures that could be found in the environment. This can present problems for Toxaphene quantitation (see Section 12). Ultra PP-271.

7.4.4 Chlordane Stock, 100 µg/mL

The Chlordane stock contains Technical Chlordane (CAS# 12789-03-6). Ultra PP-151.

7.4.5 Appendix IX Calibration Stock

The Appendix IX stock calibration mixture contains the compounds at the concentrations listed in the following table. Accustandard S-6880 custom.

Appendix IX Calibration Stock Standard

Compound	Concentration (µg/mL)
2,4'-DDD	100
2,4'-DDE	100
2,4'-DDT	100
Chlorobenzilate	1,000
Chlorpyrifos	500
Diallate	10,000
Dicofol	1,000
Isodrin	500
Kepone	1,000
DBPP	5,000
Mirex	500

7.4.6 Non-Routine Compounds

Other, non-routine compounds not listed in this section may be requested by a client and may be added to this procedure.

7.4.6.1 In these cases, all stock solutions will be obtained from commercial sources and will be verified with a second-source standard as described in Section 7.2 above.

7.4.6.2 Non-routine standards will be stored and treated as described in Section 7.3 above or as specified by the manufacturer.

7.4.6.3 Subsequent dilutions of specially requested compounds will be determined in a manner consistent with the client's recommendations for number of calibration points, inclusion of reporting limit, and concentration range adequate to represent the linearity of the instrument.

7.4.6.4 These specially requested, non-routine compounds either may be added to the dilution scheme used for routine compounds or may be prepared as a separate calibration.

7.4.6.5 All standards preparation for non-routine compounds shall be documented using the same method that is used for routine compounds.

7.5 Intermediate Level Calibration Standards

7.5.1 Routine Pesticide Mix C Intermediate Calibration Standard, 1.0 µg/mL. The intermediate level calibration standard for routine pesticide compounds including Hexachlorobenzene and Mirex is prepared by diluting the AB (Section 7.4.1) and B (Section 7.4.2) mix stock standards in hexane as follows (all compounds are the same final concentration):

Mix C Intermediate Calibration Standard

Stock AB & Isodrin (mL)	Stock B (mL)	Mirex & HCB (mL)	Hexane (mL)	Final Concentration of Each Pesticide (µg/mL)
0.1	0.5	1.0	98.4	1.0

7.5.2 Appendix IX Intermediate Calibration Standard

The Appendix IX intermediate level calibration standard is prepared by diluting 0.5 mL of the Appendix IX stock standard (Section 7.4.5) with hexane to a final volume of 50 mL, which results in the following concentrations:

Appendix IX Intermediate Calibration Standard

Compound	Concentration (µg/mL)
2,4'-DDD	1.0
2,4'-DDE	1.0
2,4'-DDT	1.0
Chlorobenzilate	10.
Chlorpyrifos	5.0
DBPP	50.
Diallate	100.
Dicofol	10.
Isodrin	5.0
Kepone	10.
Mirex	5.

7.6 Working Level Calibration Standards

7.6.1 Routine Pesticide AB Mix Working Level Calibration Standards

The following volumes of the 1.0 µg/mL Mix C intermediate standard (Section 7.5.1) are diluted to 100 mL with hexane to produce calibration standards at 6 concentration levels, as summarized in the following table:

AB Mix Working Level Calibration Standards

Level	Volume of Mix C Intermediate Std (mL)	Final Concentration (µg/mL)
1	0.4	0.0040
2	1.0	0.010
3	2.5	0.025
4*	5.0	0.050
5	7.5	0.075
6	10	0.10

* *This level is used as the Continuing Calibration Verification (CCV) standard. As a result, it may be convenient to make a larger volume of this calibration level, by diluting 12.5 mL of the intermediate standard with hexane to a final volume of 250 mL.*

7.6.2 Toxaphene Working Level Calibration Standards

The following volumes of the 100 µg/mL Toxaphene stock standard (Section 7.4.3) are diluted with hexane to the final volumes indicated in the following table:

Toxaphene Working Level Calibration Standards

Level	Volume of Stock Std (mL)	Final Volume (mL)	Final Concentration (µg/mL)
1	0.010	5.0	0.20
2	0.025	5.0	0.50
3	0.05	5.0	1.0
4	0.2	10.0	2.0
5	0.25	5.0	5.0
6	0.5	5.0	10.0

- *Level 4 is used as the CCV standard when running a 5 pt curve.*
- *To make additional volume of the level 1 standard for the single pt. CCV, dilute 0.5 mL of the stock with hexane to a final volume of 250 mL.*

7.6.3 Chlordane Working Level Calibration Standards

The following volumes of the 100 µg/mL Chlordane stock standard (Section 7.4.4) are diluted with hexane to the final volume indicated in the following table:

Chlordane Working Level Calibration Standards

Level	Volume of Stock Std (mL)	Final Volume (mL)	Final Concentration (µg/mL)
1	0.005	10.0	0.05
2	0.02	10.0	0.20
3	0.05	10.0	0.50
4*	0.10	10.0	1.0
5	0.20	10.0	2.0
* *This level is used as the CCV standard.*			

7.6.4 Appendix IX Working Level Calibration Standards

The following volumes of the Appendix IX intermediate calibration standard (Section 7.5.2) are diluted with hexane to a final volume of 1.0 mL. The following table summarizes the final compound concentration ranges for each calibration level. The concentration for each compound at each level is given in Table 3.

Appendix IX Working Level Calibration Standards

Level	Volume of Intermediate Std (mL)	Final Compound Concentration Range (µg/mL)
1	0.005	0.005 - 0.50
2	0.010	0.01 - 1.0
3	0.025	0.025 - 2.5
4 *	0.035	0.035 - 3.5
5	0.050	0.05 - 5.0
6	0.100	0.1 - 10
* *This level is used as the CCV. Because some compounds in this standard are not stable, it is not recommended to make extra volume of the level 4 standard.*		

7.7 Working Level Calibration Standards for the large volume injection (LVI) procedure

7.7.1 Routine Pesticide AB Mix Working Level Calibration Standards

An alternate intermediate standard is prepared by diluting 2mls of the 1.0 ug/ml standard to 10mls with hexane for a solution with a final concentration of 0.2 ug/mL. The following volumes of 0.2 µg/mL LVI (large volume injection) AB mix intermediate standard are diluted to 10 mL with hexane to produce calibration standards at 6 concentration levels, as summarized in the following table:

AB Mix Working Level Calibration Standards

Level	Volume of Mix C Intermediate Std (mL)	Final Concentration (µg/mL)
1	0.04	0.0008
2	0.1	0.002
3	0.25	0.005
4*	0.5	0.010
5	0.75	0.015
6	1.0	0.02

* *This level is used as the Continuing Calibration Verification (CCV) standard. As a result, it may be convenient to make a larger volume of this calibration level, by diluting 2.5 mL of the intermediate standard with hexane to a final volume of 50 mL.*

7.7.2 Toxaphene Working Level Calibration Standards

An alternate intermediate standard is prepared by diluting 2 mls of the 100 ug/ml Toxaphene stock to a final volume of 10 mls with hexane to make a 20 ug/ml standard. The following volumes of the 20 µg/mL LVI Toxaphene stock standard are diluted with hexane to the final volumes indicated in the following table:

Toxaphene Working Level Calibration Standards

Level	Volume of Stock Std (mL)	Final Volume (mL)	Final Concentration (µg/mL)
1	0.010	10.0	0.02
2	0.025	5.0	0.1
3	0.05	5.0	0.2
4	0.2	10.0	0.4
5	0.25	5.0	1.0
6	0.5	5.0	2.0

- *Level 4 is used as the CCV standard when running a 5 pt curve.*
- *To make additional volume of the level 1 standard for the single pt. CCV, dilute 0.05 mL of the stock with hexane to a final volume of 50 mL.*

7.7.3 Chlordane Working Level Calibration Standards

An alternate intermediate standard is prepared by diluting 2.0 mls of the 100 ug/mL Chlordane stock to 10.0 ml final volume with hexane to make a 20 ug/mL standard. The following volumes of the 20 µg/mL LVI Chlordane stock standard are diluted with hexane to the final volume indicated in the following table:

Chlordane Working Level Calibration Standards

Level	Volume of Stock Std (mL)	Final Volume (mL)	Final Concentration (µg/mL)
1	0.01	50.0	0.004
2	0.005	10.0	0.01
3	0.02	10.0	0.04
4*	0.05	10.0	0.10
5	0.10	10.0	0.20
6	0.20	10.0	0.40
* *This level is used as the CCV standard.*			

7.7.4 Appendix IX Working Level Calibration Standards

An alternate intermediate APIX standard is prepared by diluting 2 mL of the stock in section 7.5.2 to 10 mL final volume with hexane. The following volumes of the Appendix IX LVI intermediate calibration standard prepared here are diluted with hexane to a final volume of 1.0 mL. The following table summarizes the final compound concentration ranges for each calibration level. The concentration for each compound at each level is given in Table 3.

Appendix IX Working Level Calibration Standards

Level	Volume of Intermediate Std (mL)	Final Compound Concentration Range (µg/mL)
1	0.004	0.0008 - 0.08
2	0.010	0.002 – 0.2
3	0.025	0.005 - 0.5
4 *	0.035	0.007 – 0.7
5	0.050	0.01 - 1.0
6	0.100	0.02 - 2
* *This level is used as the CCV. Because some compounds in this standard are not stable, it is not recommended to make extra volume of the level 4 standard.*		

7.8 Second-Source Standards for Initial Calibration Verification (ICV)

The second-source stock standards are purchased from a vendor different from the one that supplied the stock calibration standards

7.8.1 Routine Pesticide AB Mix ICV Stock Standard, 1,000 µg/mL, (with Mirex at 100 µg/mL, Isodrin at 5000 ug/mL, HCB at 1000 ug/ml)

Commercial standards containing all single-component pesticide compounds are obtained from a vendor different from the one that supplied the calibration stock standard. Typically, the standards are obtained from Ultra Scientific (standard PPM-808C for the AB mix, standard EPA-1125 for Hexachlorobenzene, standard PST-720S for Mirex, and standard EPA-1131 for Isodrin).

The current toxaphene second source is AccuStandard P-093S-H-10X and it is prepared by diluting 5ul of the stock standard to 5 mL with hexane.

The current chlordane second source is AccuStandard P-017x-10x and it is prepared by diluting 5ul of the stock standard to 10mL with hexane.

7.8.2 Appendix IX ICV Stock Standard

Commercial standards are obtained at the same concentrations as shown for the calibration stock standards in Section 7.4.5, but from a different vendor (typically Ultra Scientific standard CUS-7007).

Compound	Concentration (µg/mL)
2,4'-DDD	10
2,4'-DDE	10
2,4'-DDT	10
Chlorobenzilate	100
Chlorpyrifos	50
DBPP	5,000
Diallate	1,000
Dicofol	100
Isodrin	50
Kepone	100
Mirex	50

7.8.3 Surrogate ICV Stock Standards, 200 µg/mL

Commercial standards (typically Ultra Scientific standard ISM-320) are obtained containing decachlorobiphenyl (DCBP) and tetrachloro-*m*-xylene (TCMX).

7.8.4 ICV Intermediate Level Standards, 1.0 µg/mL

The ICV intermediate level calibration standard for routine pesticide compounds is prepared by diluting the AB, Hexachlorobenzene, and Mirex, and surrogate stock standards (Sections 7.8.1 and 0) with hexane to a final volume of 25 mL as summarized in the table below. All compounds in the intermediate standard are at the same final concentration, i.e., 1.0 µg/mL.

Second-Source ICV Intermediate Standard

Vol of AB & HCB Stock (mL)	Vol of Mirex Stock (mL)	Vol of Isodrin (mL)	Vol of Surrogate Stock (mL)	Final Volume (mL)	Final Conc (µg/mL)
0.025	0.25	0.005	0.125	25.0	1.0

7.8.5 Routine Pesticide ICV Working Level Standard, 0.025 µg/mL

The working level ICV standard for the routine pesticide compounds is prepared by diluting the ICV intermediate standard (Section 7.8.4) in hexane follows:

Routine Pesticide Second-Source ICV Working Level Standard

Volume of Intermediate Standard (mL)	Final Volume (mL)	Final Concentration (µg/mL)
2.5	100	0.025

7.8.6 Appendix IX ICV Working Level Standard

The working level ICV standard for the Appendix IX compounds is prepared by diluting 0.0025 mL of the second-source Appendix IX stock standard (Section 7.8.2) with hexane to a final volume of 1 mL. The following table lists the final concentration of each pesticide:

Appendix IX ICV Working Level Standard

Pesticide	Final Concentration (µg/mL)
2,4'-DDD	0.025
2,4'-DDE	0.025
2,4'-DDT	0.025
Chlorobenzilate	0.25
Chlorpyrifos	0.125
Diallate	2.5
Dicofol	0.25
Isodrin	0.125
Kepone	0..25
Mirex	0.125

Note: The ICVs prepared above may be diluted 5x (200 ul to a volume of 1ml) for use with the large volume injection ICALs.

7.9 Continuing Calibration Verification (CCV) Standards

The level 4 AB mix working calibration standard (Section 7.6.1) and the level 4 Appendix IX working calibration standard (Section 0) are used as the CCV standards.

7.10 RL Standard

The lowest concentration calibration standard (i.e., Level 1) is used as the RL standard.

7.11 Laboratory Control Standard (LCS) Spike Solution, 0.5 µg/mL

The LCS working spike stock solution is prepared by diluting 0.50 mL of the AB mix stock standard Ultra PPM-808C (1000 ug/mL) in acetone (see Section 7.4.1) to a final volume of 10 mL in a volumetric flask. The LCS spike solution is prepared fresh each week by diluting 0.5 mL of the LCS working spike stock to a final volume of 50 mL as summarized in the table below.

The LCS for batches of aqueous samples is prepared by adding 1.0 mL of the LCS spike solution to one liter of reagent water. The LCS for batches of soil samples is prepared by adding 1.0 mL of the LCS spiking solution to 30 g of Ottawa sand.

LCS Spiking Solution

Volume of AB Mix Stock (mL)	Conc of AB Mix Stock (µg/mL)	Final Volume (mL)	Final Concentration (µg/mL)
0.5	50	50	0.5

7.12 Matrix Spike (MS) Spike Solution, 0.5 µg/mL

The working matrix spike solution is the same as the LCS spike solution (Section 7.11). Matrix spikes (MS and MSD) are prepared by adding 1.0 mL of the working spike solution to one liter of an aqueous sample or to a 30-gram soil subsample.

7.13 Toxaphene Spike Solution, 2.0 µg/mL

7.13.1 A Toxaphene stock standard solution at a concentration of 1,000 µg/mL is purchased from commercial sources. This must be from a different source than is used for the initial calibration.

7.13.2 The working Toxaphene spike solution is prepared in a 500 mL volumetric flask by adding 1.0 mL of the stock solution (Section 7.13.1) and diluting to volume with acetone.

7.13.3 Aqueous LCSs are prepared by adding 1.0 mL of the Toxaphene spike solution (Section 7.13.2) to 1.0 liter of reagent water. Soil LCSs are prepared by adding 1.0 mL of the Toxaphene spike solution (Section 7.13.2) to 30 grams of Ottawa sand.

7.13.4 Aqueous MS/MSDs are prepared by adding 1.0 mL of the Toxaphene spike solution (Section 7.13.2) to 1.0 liter of the selected aqueous sample. Soil sample MS/MSDs are prepared by adding 1.0 mL of the Toxaphene spike solution (Section 7.13.2) to 30 grams of the selected soil subsample.

7.14 Surrogate Spike Solution, 0.2 µg/mL

7.14.1 The surrogate stock solution, containing 200 µg/mL each of decachlorobiphenyl and tetrachloro-*m*-xylene (TCMX), is purchased form commercial sources.

7.14.2 The working surrogate spike solution is prepared in a 500 mL volumetric flask by adding 0.5 mL of the stock solution (Section 7.14.1) and diluting to volume with acetone.

7.14.3 For aqueous sample batches, 1.0 mL of the surrogate spike solution (Section 7.14.2) is added to each one-liter sample and QC sample. For soil sample batches, 1.0 mL of the surrogate spike solution (Section 7.14.2) is added to each 30-gram soil subsample and QC sample matrix.

7.15 Column Degradation Mix (EVAL B)

7.15.1 The DDT/endrin breakdown stock standard solution is obtained from commercial sources, with endrin at a concentration of 1.0 µg/mL, and 4,4'-DDT at 2.0 µg/mL.

7.15.2 The working EVAL B solution is prepared in a 50 mL volumetric flask, by diluting 1.0 mL of the stock solution (Section 7.15.1) in hexane, as summarized in the following table:

Column Degradation Mix (Eval B Std) Spike Solution

Compound	Volume of Stock (mL)	Final Volume (mL)	Final Concentration (µg/mL)
Endrin	1.0	50	0.02
4,4'-DDT			0.04

7.16 Primer Mix

The concentration of the column primer mix is not critical. It generally consists of a mixture of CCV, old ICAL standards, and /or old soil LCS extracts. The primer mix is used to initialize the column and does not affect calibration or quantitation.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples are collected in pre-cleaned, amber glass bottles fitted with a Teflon-lined cap. To achieve routine reporting limits, a full one liter of sample is required. Additional one-liter portions are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes.

8.2 Soil samples are collected in 8-ounce, pre-cleaned, wide-mouth jars with a Teflon-lined lid.

8.3 Samples are stored at $\leq$ 6 °C and not frozen.

8.4 Extracts are refrigerated at ≤ 6 °C.

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	Amber glass	1 Liter 40 mL VOA (for LVI)	Cool, <6°C, not frozen	7 Days	40 Days from extraction	SW-846
Soils	Glass	30 grams	Cool, <6°C, not frozen	14 Days	40 Days from extraction	SW-846

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on an instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13.0 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together

through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for batches of aqueous samples consists of 1.0 liter of reagent water (the LVI method will require a 35 mL volume of reagent water), and for batches of soil samples, consists of 30 grams of Ottawa sand, both of which are free of any of the analyte(s) of interest. The method blank is processed and analyzed just as if it were a field sample.

Acceptance Criteria: The result for the method blank must be less than one-half the reporting limit for the analyte(s) of interest.

Corrective Action: If target analytes in the blank exceed the acceptance limits, the source of the contamination must be investigated. All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If the analyte was not detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

Some programs (e.g., DOD) require corrective action if the concentration of an analyte in the blank is at or above the reporting limit AND is greater than 1/10 the amount measured in any sample. See Policy DV-QA-003P and Policy DV-QA-024P for further details.

9.5 Laboratory Control Sample (LCS)

At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of reagent water to which the analyte(s) of interest are added at a known concentration. For soil sample batches, the LCS consists of reagent sand to which the analyte(s) of interest are added at a known concentration. See Section 7.11 for the preparation of LCSs. The LCS is carried through the entire analytical procedure just as if it were a sample.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at $\pm$ 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at $\pm$ 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Note: Some programs (e.g., South Carolina) do not allow marginal exceedances. Please see the QAS's in the public folders for the current requirements.

Corrective Action: If LCS recoveries are outside of the established control limits, and the MS/MSD recoveries are also out of control limits then the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed. If instrument maintenance and recalibration is performed and the LCS is reanalyzed as a corrective action for out of control LCS then all of the associated samples in the batch must also be reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair should be processed with each preparation batch. If sufficient sample is not available for an MS/MSD then a duplicate LCS should be prepared to establish precision. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Refer to Section 7.12 for preparation of matrix spikes. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

Acceptance Criteria: The recovery results for the MS and MSD must fall within the established control limits, which are set at $\pm$ 3 standard deviations around the historical mean. The relative percent difference (RPD) between the MS and MSD must be less than the established RPD limit, which is set at 3 standard deviations above the historical mean. Current control limits are maintained in the LIMS.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e., method blank, LCS, LCSD, MS, and MSD) is spiked with DCB and TCMX surrogate compounds. Refer to Section 7.14 for preparation of the surrogate spike solution.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at $\pm$ 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

For field samples, surrogate recoveries are usually calculated and reported for DCB only. TCMX may also be added, however, if two surrogate compounds are analyzed and recoveries calculated, and either surrogate fails to meet acceptance criteria, corrective actions are required. (This also applies to programs that require the use of only one surrogate.)

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram, or due to the sample concentrating due to evaporation or improper adjustment of the final extract volume. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control. All out of control surrogates and associated corrective actions must be documented in an NCM.

10.0 Calibration and Standardization

10.1 TestAmerica Denver gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

10.1.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and under the public folder, *Arizona Calibration Training.*

10.1.2 Use the ChemStation chromatography data system to set up GC conditions for calibration. See Table 2 for typical operating conditions.

10.1.3 Transfer calibration standard solutions into autosampler vials and load into the GC autosampler. Use the ChemStation software to set up the analytical sequence.

10.1.4 Unprocessed calibration data are transferred to the TARGET DB database for processing. After processing the calibration data, print the calibration report and review it using the calibration review checklist, GC and HPLC ICAL TALS Review Checklist. (See SOP DV-QA-0020.) Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration reports are scanned and stored as Adobe Acrobat files on the Public Drive.

10.2 Column Degradation Evaluation

10.2.1 Each day of operation before any calibration or calibration verification standards are analyzed and at the beginning of each 12-hour shift, the column degradation evaluation mix (EVAL B) must be analyzed. The degradation check must be performed whether or not DDT, endrin, or degradation compounds are designated as target analytes. The purpose of the evaluation is to determine whether instrument/column maintenance is needed. The preparation of this standard is described in Section 7.15.

10.2.2 The results of the analysis of the EVAL B standard solution are used to calculate column degradation in terms of DDT percent breakdown (%B) and Endrin %B as follows:

$$\text{DDT } \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100\% \qquad \textit{Equation 1}$$

Where A_{DDD}, A_{DDE}, and A_{DDT} are the peak responses for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT, respectively, in the EVAL B chromatogram.

$$\text{Endrin } \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100\% \qquad \textit{Equation 2}$$

Where A_{EK}, A_{EA}, and A_{E} are the peak responses for endrin ketone, endrin aldehyde, and endrin, respectively, in the EVAL B chromatogram.

10.2.3 Acceptance Criteria

The %B for each of these two compounds, DDT and endrin, must not be greater than 15%.

10.2.4 Corrective Action

If the breakdown of DDT and/or endrin exceeds the 15% limit, corrective action must be taken. This action may include any or all of the following:

- Replacing the injection port liner or the glass wool.
- Cutting off a portion of the injection end of the column or guard column.
- Replacing the GC column or guard column
- Replacing the y-splitter.

After taking the appropriate corrective action, the degradation evaluation standard must be reanalyzed and must pass acceptance criteria before conducting any calibration events.

10.3 The laboratory uses six calibration levels (as shown in Table 3) for the single-component pesticides. The lowest point on the calibration curve is at or below the reporting limit (RL). The highest standard defines the highest sample extract concentration that may be reported without dilution. The preparation of the calibration standards is described in Section 7.6.

10.4 All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.5 Calibration for the multi-peak component analytes, Toxaphene and Technical Chlordane, begins with a single-point calibration at or near the RL. If any multi-peak components are found to be present in the samples, a calibration for the multi-component analyte(s) is conducted with a minimum of five calibration levels. The samples are then reanalyzed using the full calibration curve that brackets the quantitation range.

10.6 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

10.7 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious mis-injection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

10.7.1 The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and

10.7.2 The lowest remaining calibration point is still at or below the project reporting limit; and

10.7.3 The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and

10.7.4 The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average calibration factors or linear regressions, or six levels for second-order curve fits.

10.8 If a data point is rejected, it must be documented in the sequence log and on an NCM which is filed with the project.

NOTE: Second order curves are not allowed for South Carolina work.

10.9 External Standard Calibration

External standard calibration involves the comparison of instrument responses (e.g., peak area or peak height) from the target compounds in the sample to the responses of the target compounds in the calibration standards. The ratio of the detector response to the amount or concentration of target analyte in the calibration standard is defined as the calibration factor (CF), as follows:

$$CF = \frac{A_S}{C_S}$$ *Equation 3*

Where:

A_S = Peak area (or height) of the analyte or surrogate in the calibration standard.

C_S = Concentration of the analyte or surrogate, in ng/mL, in the injected calibration standard.

10.10 Establishing the Calibration Function

Calibrations are modeled either as average calibration factors or as calibration curves, using a systematic approach to selecting the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until the calibration acceptance criteria are met.

10.10.1 Linear Calibration Using Average Calibration Factor

Tabulate the peak area response for each target analyte in each calibration level against the concentration injected. For each analyte in each calibration standard, calculate the calibration factor (CF) as shown in Equation 3 above. The calibration factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (i.e., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

For each target analyte, calculate the average calibration factor as follows:

Equation 4

$$Average\,Calibration\,Factor = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where:

CF_i = Calibration factor for the i^{th} calibration level.

n = The number of calibration levels.

The relative standard deviation (RSD) is calculated as follows:

Equation 5

$$RSD = \frac{SD}{\overline{CF}} \times 100\%$$

Where SD is the standard deviation of the average CF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)^2}{n-1}}$$

Equation 6

10.10.2 Evaluation of the Average Calibration Factor

The calibration relationship can be graphically represented as a line through the origin with a slope equal to the average calibration factor. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Note: The use of grand average (evaluation of the average response over all the compounds) is no longer allowed in Method 8000C. Each compound must meet the RSD criteria.

Note: Arizona requires the use of Method 8000C for GC methods.

Acceptance Criteria: The RSD must be ≤ 20%.

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.10.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not necessarily pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

Equation 7

$$y = ax + b$$

Where:

y = Instrument response (peak area or height).

x = Concentration of the target analyte in the calibration standard.

a = Slope of the line.

b = The y-intercept of the line.

For an external standard calibration, the above equation takes the following form:

$$A_s = aC_s + b$$

Equation 8

To calculate the concentration in an unknown sample extract, the regression equation (Equation 8) is solved for concentration, resulting in the following equation, where C_s is now C_e, the concentration of the target analyte in the unknown sample extract.

$$C_e = \frac{A_e - b}{a}$$

Equation 9

Where:

A_s = Area of the chromatographic peak for the target analyte in the calibration standard.

A_e = Area of the chromatographic peak for the target analyte in the sample extract.

a = Slope of the line as determined by the least-squares regression.

C_s = Concentration of the target analyte in the calibration standard.

C_e = Concentration of the target analyte in the sample extract.

b = Intercept of the line as determined by the least-squares regression.

10.10.4 Linear Regression Evaluation

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum “Clarification Regarding Use of SW-846 Methods”, Attachment 2, Page 9, includes the statement “The Agency further recommends the use of a weighted regression over the use of an unweighted regression.”

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit, and preferably less than the MDL.

Also examine the residuals, paying particular attention to the residuals at the low end of the curve. If the intercept or the residuals are large, a second-order regression should be considered.

The linear regression must have a correlation coefficient (r) ≥ 0.990. Some programs (e.g., AFCEE, DoD) require a correlation coefficient ≥ 0.995.

Corrective Action: If the correlation coefficient falls below the acceptance limit, linear regression cannot be used and a second-order regression should be attempted.

10.10.5 Non-Linear Calibration

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$y = ax^2 + bx + c$$

Equation 10

Where a, b, and c are coefficients determined using a statistical regression technique; y is the instrument response; and x is the concentration of the target analyte in the calibration standard.

NOTE: Some programs (e.g., South Carolina) do not allow the use of second-order regressions.

10.10.6 Non-Linear Calibration Evaluation

A minimum of six points must be used for a second-order regression fit. Second-order regressions should be the last option. They should not be used to avoid instrument maintenance.

Acceptance Criteria: The coefficient of determination must be ≥ 0.990.

The absolute value of the intercept must not be large relative to the lowest concentrations being reported.

The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).

The distribution of concentrations is adequate to characterize the curvature.

Corrective Action: If the coefficient of determination falls below the acceptance limit and the other calibration models are unacceptable, the source of the problem must be investigated and the instrument recalibrated. Third-order regressions are <u>not</u> allowed at TestAmerica Denver.

10.11 Initial Calibration Verification (ICV), 0.025 µg/mL for most compounds

A mid-level standard that is obtained from a source different from that of the calibration standards (second-source standard) is used to verify the initial calibration (see Section 7.8). The ICV standard is analyzed immediately following the initial calibration (ICAL).

Acceptance Criteria: The result for the target analyte(s) in the ICV standard must be within ± 15% for Method 8081A and ± 20% of the expected value(s) for Method 8081B.

Corrective Action: If the applicable criteria is not achieved, the ICV standard, calibration standards, and instrument operating conditions should be checked. Correct any problems and rerun the ICV standard. If the ICV still fails to meet acceptance criteria, then repeat the ICAL.

10.12 Calibration Verification

10.12.1 12-Hour Calibration Verification

The 12-hour calibration verification sequence consists of, at a minimum, an instrument blank and the mid-level calibration standard. The 12-hour calibration verification sequence must be analyzed within 12 hours of the initial calibration and at least once every 12 hours thereafter when samples are being analyzed.

NOTE: It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.12.2 Continuing Calibration Verification (CCV), 0.05 µg/mL for most compounds.

Note that some programs including the State of Arizona and Wisconsin require that the CCV concentration be varied throughout the sequence when calibration fits other than average response are used.

It may be appropriate to analyze a mid-level standard more frequently than every 12 hours. The mid-level calibration standard is analyzed as the continuing calibration verification (CCV) standard (see Section 7.9). At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCSs, and method blanks. Some programs, specifically drinking water programs, require a CCV after every 10 samples to minimize the number of samples requiring re-injection when QC limits are exceeded. If 12 hours elapse, analyze the 12-hour standard sequence instead.

10.12.3 RL Standard

It may also be appropriate to analyze a standard prepared at or very near the reporting limit (RL) for the method at the end of the analytical sequence, as a minimum (see Section 7.10). This standard can be used to rule out false negatives in client samples in cases where the %D for one or more of the analytes in a bracketing CCV falls below the lower acceptance limit. The results for the RL standard are not evaluated unless the previous CCV fails acceptance criteria.

10.12.4 Acceptance Criteria for Continuing Calibration Verification (CCV)

10.12.4.1 Detected Analytes (≥ RL)

For any analyte detected at or above the reporting limit (RL) in client samples, the percent difference (%D) for that analyte in the

preceding and following CCVs (i.e., bracketing CCVs) or 12-hour calibration must be within ± 15% for Method 8081A and ± 20% for Method 8081B if an average curve fit is used. For other curve fits (i.e., linear or 2nd order) see DV-QA-027P which requires the allowed %D be 15% for both methods (on both columns).

In some cases, the nature of the samples being analyzed may be the cause of the failing %D. When the %D for an analyte falls outside of the CCV criteria stated above, and that analyte is detected in any or all of the associated samples, then those samples must be reanalyzed (at a dilution if column damage is imminent) to prove a matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect if so desired by the client.

Refer to Section 12 for which result to report.

In cases where additional compounds are to be analyzed in conjunction with compounds defined by this method and that are not defined in the scope and application of method 8081B different CCV acceptance criteria may apply. Kepone is not recommended by method 8081B and the CCV acceptance criteria is defined as +/- 53%. Further these additional compounds will not be used in grand mean calculations (when applicable) as discussed below.

The %D is calculated as follows:

$$\%D = \frac{\text{Measured Conc} - \text{Theoretical Conc}}{\text{Theoretical Conc}} \times 100 \qquad \textit{Equation 11}$$

10.12.4.2 Analytes Not Detected (< RL)

For any analyte not detected in client samples, the %D for that analyte in the bracketing CCVs should also be within ± 20% for an average curve fit for Method 8081B or within 15% for an average curve fit for Method 8081A or for other curve fits by either method. Method 8081B references method 8000 for compounds with curve fits other than an average curve fit and the criteria in Table 6 applies to those compounds. See also DV-QA-027P for further evaluation criteria. Any deviation for the calibration criteria outlined in this procedure must be documented in an NCM.

When applying Method 8000B to the analysis, the analysis is acceptable if the average of the %D values for all the analytes is within ± 20% and the %D for any individual analyte is within ± 30%. The average %D is calculated by summing all the %D results in the calibration and dividing by the number of analytes. If an average %D is used and the %D for any individual analyte falls outside of ± 30%, then additional evaluation is needed as summarized in Table 6.

NOTE: The grand mean must <u>not</u> be applied when Method 8000C is applicable (e.g., Arizona)

10.13 Retention Time Windows

Retention time (RT) windows must be determined for all analytes.

10.13.1 Determine new RT windows each time a new column is installed or annually, whichever is most frequent.

10.13.2 Make an injection of all analytes of interest each day over a 72-hour period.

10.13.3 Calculate the mean and standard deviation for the three RTs for each analyte as follows:

$$\text{Mean RT} = \overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RT_i - \overline{RT}\right)^2}{n-1}}$$

Equations 12 & 13

Where:

RT_i = Retention time for the i^{th} injection.

n = Number of injections (typically 3).

SD = Standard deviation.

NOTE: For the multi-component analytes, Toxaphene and Technical Chlordane, the mean and standard deviation must be calculated for each of the 3 to 6 major peaks used for sample calculations.

10.13.4 Set the width of the RT window for each analyte at ± 3 standard deviations of the mean RT for that analyte.

10.13.5 The center of the RT window for an analyte is the RT for that analyte from the last of the three standards measured for the 72-hour study.

10.13.6 The center of the window for each analyte is updated with the RT from the level 4 standard of the ICAL, or the CCV at the beginning of the analytical sequence. The width of each window remains the same until new windows are generated following the installation of a new column, or in response to an RT failure. The RT window width may be expanded if the RT drift observed in the ICAL is greater than the established window. The expanded window is noted on the ICAL checklist.

10.13.7 If the RT window as calculated above is less than ± 0.01 minute, use ± 0.01 minute as the RT window. This allows for slight variations in retention times caused by sample matrix.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Preparation

11.3.1 Sample preparation for aqueous samples is described in SOP DV-OP-0006 (and SOP DV-OP-0023 for the LVI method).

11.3.2 Sample preparation for solid samples is described in SOPs DV-OP-0016 and DV-OP-0015.

11.3.3 Cleanup and concentration of sample extracts are described in SOP DV-OP-0007. Note that it is highly recommended that all samples be check for sulfur and cleaned up if necessary before the samples are analyzed on the instrument. Sulfur can contaminate the column and hinder the quantification of certain compounds.

11.3.4 The final extract volume in hexane is 10 mL. The LVI method final volume is 2 mL.

11.3.5 Use hexane to dilute sample extracts, if necessary.

11.4 Instrument Maintenance

Before the start of any daily sequence the instrument system should be evaluated for possible maintenance. Typically for the 8081 analysis the injection port liner must be changed daily in order to facilitate a passing DDT/Endrin breakdown standard. If the previous run ended with a failing continuing calibration then the system should be maintained to bring it back into control. The injector septum should be changed after about 200 injections have been completed. If the last CCV that was analyzed indicated a high response then a simple liner change is typically sufficient to bring the system back into control. Analysis of a few solvent blanks or a system bake out may be necessary to drive out any residual contamination on the column. A reduced response may indicate that the system needs to be evaluated for leaks. Poor peak shape may necessitate clipping a loop out of the analytical column. If this fails to solve the peak shape problem then replacement of the columns may be indicated. The goal is to maintain the system as close to top condition as possible as was observed when new columns and injector parts were installed. Re-calibration should not be used to correct for maintenance related issues. Always document any maintenance procedure in the maintenance logbook.

11.5 Gas Chromatography

Chromatographic conditions for this method are presented in Table 2. Use the ChemStation interface to establish instrument operating conditions for the GC. Raw data obtained by the ChemStation software is transferred to the TARGET DB database for further processing. The data analysis method, including peak processing and integration parameters, calibration, RT windows, and compound identification parameters, is set up in the TARGET DB software.

11.6 Sample Introduction

All extracts and standards are allowed to warm to room temperature before injection. An autosampler is used to introduce samples into the chromatographic system by direct injection of 1 or 2 µL of the sample extract. Samples, standards, and QC samples must be

introduced using the same procedure. Use the ChemStation interface to set up and run the analytical sequence. Sample injection and analysis are automated and may proceed unattended.

11.7 Analytical Sequence

An analytical sequence starts with a minimum five-level initial calibration (ICAL) or a daily calibration verification. Refer to Table 3 for the calibration levels used.

11.7.1 Prior to analyzing any calibration or calibration verification standards, the column degradation evaluation standard is injected and the results are evaluated as described in Section 10.2.

11.7.2 The daily calibration verification includes analysis of the 12-hour calibration sequence (Section 10.12.1) and updating the retention time windows (see Section 10.13).

11.7.3 If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration verification.

11.7.4 The following is a typical analytical sequence:

- Primer
- Hexane blank
- Eval B Std (column degradation evaluation)
- Daily initial CCVs
- LCS
- Method Blank
- 10 samples
- CCVs
- Followed by cycles of 10 samples and CCVs as needed
- Closing CCV

11.8 Daily Retention Time Windows

The centers of the retention time (RT) windows determined in Section 10.13 are adjusted to the RT of each analyte as determined in the 12-hour calibration verification. The centers of the RT windows must be updated at the beginning of each analytical sequence.

11.9 Manual Integration and Data Review

Upon completion of the analytical sequence, transfer the raw chromatography data to the TARGET DB or CHROM database for further processing.

11.9.1 Review chromatograms online and determine whether manual data manipulations are necessary.

11.9.2 All manual integrations must be justified and documented. See DV-QA-011P requirements for manual integration.

11.9.3 Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature.

11.9.4 Alternatively, the manual integration may be processed manually. In the latter case, print both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration. The manually processed chromatograms must be scanned and attached to the project in TALS.

11.10 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD.

11.10.1 The data package should consist of the checklist, sequence(s), ICAL cover, ICAL summary and history used for data quantitation and the prep batch paperwork.

11.10.2 Perform a level 1 data review and document the review on the data review checklist, GC Data Review Checklist/Batch Summary (See SOP DV-QA-0020.)

11.10.3 Submit the data package and review checklist to the Data Review Group for the level 2 review. All manual integrations must be evaluated by the peer reviewer and this review must be documented by date and initial on the manual integration summary report and/or the level 2 review checklist. For Federal projects and certain client specified projects, the documentation of the manual integration review must be scanned and attached to the project tin the LIMS to be included with the Level 4 data package. The level 2 review is documented on the review checklist initiated at the level 1 review. The data review process is explained in SOP DV-QA-0020.

12.0 Calculations / Data Reduction

12.1 Qualitative Identification

12.1.1 Tentative identification of an analyte occurs when a peak is found on the primary column within the RT window for that analyte, at a concentration above the reporting limit, or above the MDL if qualified data (J flags) are to be reported. Identification is confirmed if a peak is also present in the RT window for that analyte on the second (confirmatory) column and if the analyte concentration is greater than the MDL. When confirmation is made using a second column, the analysis on the second column must meet all of the QC criteria for continuing calibration verification and RTs.

12.1.2 The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times. If a RT shift greater than the RT window occurs for a reported compound the situation must be explained in an NCM.

12.2 Dual-Column Quantitation and Reporting

12.2.1 A primary column is designated. The result from the primary column is normally reported. If the continuing calibration verification fails on one of the columns,

the appropriate corrective action must be taken. The result from the secondary (confirmation) column may be reported if either of the following possibilities are true:

12.2.1.1 There is obvious chromatographic interference on the primary column.

12.2.1.2 The result on the primary column is > 40% greater than the result on the secondary column.

12.2.2 Dual Column Results With >40% RPD

12.2.2.1 If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, the higher of the two results is reported unless there is obvious interference documented on the chromatogram.

12.2.2.2 If there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

12.2.2.3 If there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.

12.2.2.4 The RPD between two results is calculated using the following equation:

$$RPD = \frac{\left|R_1 - R_2\right|}{\frac{1}{2}\left(R_1 + R_2\right)} \times 100\%$$

Where R_1 is the result for the primary column and R_2 is the result for the confirmation column.

12.3 Multi-Component Analytes (Toxaphene and Technical Chlordane)

12.3.1 Qualitative Identification

Retention time windows are also used for identification of multi-component analytes, but the “fingerprint” produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3.2 Quantitation of Toxaphene

12.3.2.1 While Toxaphene contains a large number of compounds that produce well resolved peaks in a GC/ECD chromatogram, it also contains many other components that are not chromatographically resolved. The unresolved complex mixture results in a "hump" in the chromatogram that is characteristic of the Toxaphene mixture of compounds. The resolved peaks are important for the identification

of the mixture, and the area of the unresolved complex mixture contributes a significant portion of the area of the total response.

12.3.2.2 To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the RTs of the first and last eluting Toxaphene components in the standard. In order to use the total area approach, the pattern in the sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present in the sample. Otherwise, the sample concentration may be significantly underestimated.

12.3.2.3 Toxaphene may also be quantitated on the basis of 4 to 6 major peaks. Using a subset of 4 to 6 peaks for quantitation provides results that agree well with the total peak approach and may avoid difficulties when interferences with Toxaphene peaks are present in the early portion of the chromatogram from compounds such as DDT. Construct the baseline as outlined in 12.3.2.2.

12.3.2.4 When Toxaphene is determined using the 4 to 6 peaks approach, care must be taken to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms.

12.3.2.5 The chosen peaks must be within the established retention time. If there is an interference that affects the accuracy of results, the analyst may use as few as 4 major peaks. The same peaks that are used for sample quantitation must be used for calibration.

12.3.2.6 The heights or areas of the chosen peaks should be summed together and averaged to determine the Toxaphene concentration.

12.3.2.7 Second column confirmation of multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

NOTE: USACE projects require the use of second-column confirmation of multi-component analytes unless the project work plans (SOW, SAP, QAPP, etc.) specify single-column analysis.

12.3.3 Quantitation of Technical Chlordane

12.3.3.1 Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. *cis*-Chlordane (or α-Chlordane) and *trans*-Chlordane (or γ-Chlordane) are the two most prevalent major components of Technical Chlordane. However, the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch.

12.3.3.2 When the GC pattern of the sample resembles that of Technical Chlordane, Chlordane may be quantitated by comparing the total area of the Chlordane chromatogram using 3 to 5 major peaks or the total area. If the Heptachlor epoxide peak is relatively small, include it as part of the total Chlordane area for calculation. If Heptachlor and/or Heptachlor epoxide are much out of proportion, calculate

these separately and subtract their areas from the total area to give a corrected Chlordane area.

NOTE: Octachlor epoxide, a metabolite of Chlordane, can easily be mistaken for Heptachlor epoxide on a nonpolar GC column.

12.3.3.3 To measure the total area of the Chlordane chromatogram, construct the baseline of Technical Chlordane in each calibration chromatogram between the RTs of the first and last eluting Technical Chlordane components. Use this area and the mass or concentration of Technical Chlordane in each calibration standard to establish the calibration function (Section 10.10). Construct a similar baseline in the sample chromatogram, measure the area, and use the calibration function to calculate the concentration in the sample extract.

12.3.3.4 When the GC pattern of Chlordane in a sample differs considerably from that of the Technical Chlordane standard, it may not be practical to relate a sample chromatogram back to the Technical Chlordane standard chromatogram. In these cases, all identifiable Chlordane components may be summed and reported as "Chlordane (not otherwise specified, CAS number 57-74-9)."

12.3.3.5 A third option for quantitating Technical Chlordane is to quantitate the peaks for α-Chlordane, γ-Chlordane, and Heptachlor separately against the appropriate reference materials, and report these individual components under their respective CAS numbers.

NOTE: See Section 12.6.2 for use of CLD Flag when only the isomers are reported and Technical Chlordane is the requested analyte.

12.3.3.6 Second column confirmation of multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

NOTE: USACE projects require the use of second-column confirmation of multi-component analytes unless the project work plans (SOW, SAP, QAPP, etc.) specify single-column analysis.

12.4 Surrogate recovery results are calculated and reported for DCB. TCMX may also be added, however if the two surrogate compounds are analyzed, and recoveries are calculated, and either surrogate fails to meet control limits, corrective actions are required (this also applies to programs that require the use of only one surrogate). See section 9.7 for further details.

12.5 Calibration Range and Sample Dilutions

12.5.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted with hexane (record the hexane lot number in the run sequence) and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for the analyte(s) that were found to be over the

calibration range in the high sample, they must be reanalyzed to rule out carryover, unless other objective evidence indicates that the detection is not the result of carryover. Such evidence may include an observation where carryover was not observed when samples or blanks were analyzed after another sample with similar high compound recovery or when the detection in the sample with suspected carryover is much higher than the expected amount of carryover (i.e. the sample's concentration may be similar to or higher than the concentration found in the previous sample). It may also be necessary to dilute samples because of matrix interferences.

12.5.2 If the initial diluted run has no hits or hits below 20% of the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.5.3 Guidance for Dilutions Due to Matrix Interference

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

12.5.4 Reporting Dilutions

Some programs (e.g., South Carolina and AFCEE) and some projects require reporting of multiple dilutions (check special requirements in LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported.

12.6 Interferences Observed in Samples

12.6.1 Dual column analysis does not entirely eliminate interfering compounds. Complex samples with high background levels of interfering organic compounds can produce false positive and/or false negative results. The analyst must use appropriate judgment to take action as the situation warrants.

12.6.2 Suspected Negative Interferences

If peak detection is prevented by interferences, further cleanup should be attempted (see SOP DV-OP-0007). Elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

If the individual isomers of chlordane are identified, but there is no pattern for the confirmation of "Technical Chlordane", and the project has ONLY technical chlordane requested, the results for technical chlordane should be qualified ("CLD") by the analyst to indicate the presence of the chlordane isomers.

12.6.3 Suspected Positive Interferences

If no further cleanup is reasonable and interferences are evident that are suspected of causing false positive results, consult with the laboratory Project Manager to determine if analysis using additional confirmation techniques is appropriate for the project. Use of additional confirmation columns is another possible option, however caution is warranted in order to rule out false

negatives. At a minimum, an NCM should be prepared by the analyst and should include the following comment for inclusion in the case narrative:

"*Based on review of the chromatograms for samples _____, it is my opinion that the evident interferences may be causing false results.*

Date _____ Analyst _____"

Sample dilution may be the only acceptable recourse to resolve detections when large amounts of non-target matrix are observed.

12.7 Calculations

12.7.1 LCS and Surrogate Spike Recovery Calculation

LCS and surrogate spike recoveries are calculated using the following equation:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\% \qquad \textit{Equation 15}$$

12.7.2 MS and MSD Recovery Calculation

Matrix spike recoveries are calculated as follows:

$$\text{MS or MSD } \%\text{Recovery} = \left(\frac{SSR - SR}{SA} \right) \times 100\% \qquad \textit{Equation 16}$$

Where:

SSR = Measured concentration in spiked sample.

SR = Measured concentration in unspiked sample.

SA = Concentration of spike added to sample.

12.7.3 MS/MSD RPD Calculation

The relative percent difference between the MS and MSD is calculated as follows:

$$\%RPD = \frac{\left|R_1 - R_2\right|}{\frac{1}{2}\left(R_1 + R_2\right)} \times 100\% \qquad \textit{Equation 17}$$

Where R_1 is the result for the MS and R_2 is the result for the MSD.

12.7.4 Concentration of Analyte in the Sample Extract

Depending on the calibration function used, the concentration of the analyte in the sample extract is calculated as follows (see Section 10.10 for details on establishing the calibration function):

Average Calibration Factor: $C_e = \frac{A_s}{CF}$ *Equation 18*

Linear Regression: $C_e = \frac{\left[A_s - b\right]}{a}$ *Equation 19*

Non-Linear Regression: $C_e = f(A_s)$ *Equation 20*

Where:

C_e = Concentration of the analyte in the sample extract (ng/mL).

A_s = Peak area for the analyte in the sample extract injection.

b = y-intercept of the calibration fit.

a = Slope of the calibration fit.

$f(A_s)$ = Mathematical function established by the non-linear regression.

12.7.5 Concentration of Analyte in Original Sample (for 1 uL injection)

$$C_{sample} = \frac{C_e}{1000\frac{ng}{\mu g}} \times \frac{V_e}{V_s} \times DF \qquad \textit{Equation 21}$$

Where:

C_{sample} = Concentration of analyte in original sample (µg/L or µg/kg).

C_e = Concentration of analyte in sample extract injected in GC (ng/mL).

$1000\frac{ng}{\mu g}$ = Factor to convert ng/mL to µg/mL.

V_e = Volume of sample extract (mL).

V_s = Volume (or weight) of original sample (L or kg).

DF = Dilution Factor (post extraction dilutions)

12.8 All data are subject to two levels of review, which is documented on a checklist, as described in SOP DV-QA-0020.

13.0 Method Performance

13.1 Initial Demonstration of Capability

An initial demonstration of capability for each method must be performed prior to analyzing samples.

13.1.1 For the standard analyte list, the initial demonstration consists of the preparation and analysis of a QC check sample (e.g., LCS) containing all of the standard analytes for the method, as well as a method detection limit (MDL) study (described in Section 13.2 below).

13.1.2 Four aliquots of the QC check sample are analyzed with the same procedures used to analyze samples, including sample preparation.

13.1.3 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific

acceptance criteria (e.g., LCS control limits). All four results must meet acceptance criteria before the method can be used to analyze samples.

13.1.4 For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is successful analysis of an extracted standard at the reporting limit and a single point calibration.

13.2 Method Detection Limit (MDL)

An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy DV-QA-005P. An MDL verification is performed once a year to satisfy state accreditation requirements. For DoD, AFCEE, and DOE projects, an MDL verification is performed quarterly. MDLs are stored in the LIMS.

13.3 Analyst Training and Qualification

13.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, ICVs, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

14.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, “Waste Management and Pollution Prevention”, of the Environmental Health and Safety Manual, and DV-HS-001P, “Waste Management Program.”

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Expired extract vial waste - Waste Stream A

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.1.1 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

16.1.2 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

16.1.3 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

16.1.4 Method 3546, Microwave Extraction, Revision 0, February 2006.

16.1.5 Method 3620C, Florisil Cleanup, Revision 3, February 2007.

16.1.6 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

16.1.7 Method 3665A, Sulfuric Acid/Permanganate Cleanup, Revision 1, December 1996.

16.1.8 Method 8081A, Organochlorine Pesticides by Gas Chromatography, Revision 1, December 1996.

16.1.9 Method 8081B, Organochlorine Pesticides by Gas Chromatography, Revision 2, February, 2007.

16.1.10 Method 8000B, Determinative Chromatographic Separations, Revision 2, December, 1996.

16.1.11 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

17.0 Method Modifications:

Item	Method	Modification
1	8081A 8081B	Method 8081B includes an internal standardization option. Because of the high probability of interferences affecting internal standards, this SOP allows only external standards.
2	8081A 8081B	Section 11.4.1.1, allows the use of a single-point calibration for the multi-component pesticides. In this SOP an initial single-point calibration is used, but a five-point calibration followed by reanalysis of associated samples is required when one of the multi-component pesticides is detected.
3	8081A 8081B	Method 8081 references 8000, which allows the use of third-order calibration curves. TestAmerica Denver does not allow third-order curves.
4	8081A 8081B 8000B	Section 10.7.2 excludes the use of the grand average of % RSD and requires each compound meet % RSD criteria for the initial calibration while Method 8000 B allows acceptance using the mean of % RSD for all compounds in the calibration.
5	8081A 8081B 8000B 8000C	Minimum retention time window (± 0.01 minute) is more stringent than the Method 8000B window of ± 0.03 minute. The established window may be adjusted based on RT drift observed in the ICAL.

18.0 Tables and Attachments

Table 1: Analyte List and Standard Reporting Limits
Table 2: Typical Instrument Conditions
Table 3: Calibration Levels (μg/mL)
Table 4: LVI Method Calibration Levels (μg/mL)
Table 5: Column Degradation Evaluation Mix
Table 6: LCS/Matrix Spike and Surrogate Spike Levels
Table 7: Evaluation Criteria and Corrective Actions for Continuing Calibration Verification

Attachment 1: Example Chromatogram – RTx CLP-I – AB Standard
Attachment 2: Example Chromatogram – RTx CLP-I – AP9 Standard
Attachment 3: Example Chromatogram – RTx CLP-II – AB Standard
Attachment 4: Example Chromatogram – RTx CLP-II – AP9 Standard
Attachment 5: Example Chromatogram – Rxi-35SIL MS – AB Standard
Attachment 6: Example Chromatogram – Rxi-35SIL MS – AP9 Standard
Attachment 7: Example Chromatogram – Rxi-XLB – AB Standard
Attachment 8: Example Chromatogram – Rxi-XLB – AP9 Standard

19.0 Revision History

- Revision 7.0, dated 12 October 2012
  - Added section 1.5 to state that the LVI procedure is not approved by South Carolina
- Revision 6.0, dated 16 July 2012
  - Corrected grammatical and formatting errors.
  - Added the information on the LVI procedure throughout the SOP
  - Added paragraph on “reagent grade” materials to Section 7
  - Added Section 11.4 – Instrument Maintenance
  - Updated Table 1 to include LVI information
  - Added Table 3
- Revision 5.0, dated 30 June 2011
  - Combines SOP No. DV-GC-0020 and SOP No. DV-GC-0026, superseding the latter, implemented 28 February 2011.
  - Updated equipment and supplies section
  - Aligned language with other GC SOPs for clarity and consistency in calibration and data review sections
  - Updated standards and reporting limits table.
  - Revised reporting criteria in Section 12.2

Revision History for SOP No. DV-GC-0020

- Revision 4.1, dated 11 June 2010
  - Annual Technical Review
  - Made minor grammatical and typographical corrections.
  - Added section 12.1 to reference corporate SOP CA-Q-S-005 “Calibration Curves”
- Revision 4, dated 23 February 2009
  - Integration for TestAmerica and STL operations.

- Updated the definition of Chlordane as defined in Method 8081B.
- Updated Section 12.3.3 Concerning the explanation of Chlordane as defined in Method 8081B.

- Revision 3, dated 19 October 2007
  - Integration for TestAmerica and STL operations.
  - Added additional observations of standards degradation issues in section 7.5.2.
  - Added Hexachlorobenzene and Mirex to the AB Mix Standards in section 7.
  - Removed Mirex from the Appendix IX Standards in section 7.
  - Added a sixth calibration level in section 7.8.2.
  - Updated Chlordane Working Level Calibration Standard concentrations in section 7.8.3.
  - Updated the Appendix IX Working Level Calibration Standards dilution instructions in section 7.8.4.
  - Changed the detector temperature from 300°C to 325°C and the pressure from 30 psi to 20 psi in Table 2. Typical Instrument Conditions.
  - Updated the Calibration Levels Table to reflect instructions in Section 7.
  - Updated Table 5. LCS/Matrix Spike and Surrogate Spike Levels to the current practice.

- Revision 2, dated 12 October 2006
  - The formatting was updated to comply with Policy QA-001.
  - Removed all instructions, equipment, and reagents from this SOP that are used for sample preparation only. Sample preparation is performed by a different analytical group and according to separate sample preparation SOPs.
  - Revised Section 9.1 to include a reference to Policy QA-024, *Requirements for Federal Programs*.
  - Revised Section 10.12 to describe current practice for CCV evaluations and added Table 6.
  - Move instructions for establishing retention time windows from Section 13, Method Performance, to Section 10, Standardization and Calibration.
  - Added Sections 10.1, 11.5, 11.6, and 11.10 to better explain how the gas chromatograph systems are operated, data are analyzed and transferred to the LIMS, and data reviews are performed.
  - In Section 12.2.1, expanded instructions for quantitating multi-component analytes.
- Revision 1, dated 9 September 2004
  - Interim changes since the last full revision are incorporated.
  - Section 4.4 is changed to make it clear when Florisil cleanup is performed.
  - The Safety Section 5 is updated per current STL requirements.
  - The ID of the additional columns discussed in 6.2.3 is corrected.
  - The autosampler vials are changed to crimp-top, rather than screw top.
  - The lowest calibration point for the AB mix is changed from 0.005 ug/mL to 0.004 ug/mL to support lower reporting limits. Section 7.4.3.1 and Table 3 were changed accordingly.
  - Section 7.9.2 is corrected to state that the working spike solution is made up to 10 mL, rather than 100 mL.
  - The amount of 4,4'-DDT stock standard added to make the column degradation mix (Eval B Std) is changed from 2.0 mL to 1.0 mL.
  - The requirement to adjust retention time windows every 12 hours was changed to require the adjustment with every daily CCV, per method.
  - The injection volume on the GC is 1 uL, rather than 2 uL, in Section 11.3.

- The discussion about dual column reporting in 12.2.1 is clarified.
- Section 12.3.2 is changed to require quantitation with a minimum of 5 major peaks, rather than 6. This is in accordance with the method.
- The minimum retention time windows for the faster HP 6890 GCs is changed to ± 0.01 minutes in Section 13.2.3, and ± 0.03 minutes is still the minimum for the older HP 5890 GCs.

- Revision 0, dated 16 September 2002
  - The previous SOP (CORP-GC-0001) combined several 600 series and 8000 series GC procedures. This SOP is just for Method 8081A.
  - Program specific requirements for AFCEE, North Carolina, South Carolina, and USACE were added.
  - Cross-references to other STL Denver SOPs were added as appropriate.
  - The discussion about chlordane was added to the Definitions section.
  - Section 7 was expanded to include complete details about preparation of standards.
  - More details about selecting initial calibration functions were added to provide guidance to analysts for a more systematic approach.
  - The minimum retention time windows were changed from ± 0.05 minutes to ± 0.03 minutes.
  - Section 12.2.2 includes further clarification about the reporting of results when dual column values differ by more than 40%.
  - A minimum of 4 peaks will be used to quantitate technical chlordane, instead of 3.
  - The example formula for calculating sample results was changed to match the form of the equation presented on the Target system quantitation reports.
  - A qualifying statement to be used in final report case narratives when false positive results are suspected was added in Section 12.6.3.
  - Calibration levels for the Appendix IX compounds added to Table 3.
  - Spike levels for the Appendix IX compounds were added to Table 5.

Revision History for SOP No. DV-GC-0026

- Revision 0.3, dated 28 February 2011
  - Updated column types in section 6.4.3
  - Added volumetric glassware to section 6.7
  - Added details to standards preparation section 7.
  - Changed section 7.1. LCS is prepared from a different source than ICAL.
  - Added detail to section 9.6 about LCS/LCD.
  - Added Arizona criteria to section 10.9.2. Need to run the CCV at various concentrations.
  - Source method review
  - Updated Method Modifications table
  - Updated references to include method references for sample preparation.
  - Spell check and corrections throughout.

- Revision 0.2, dated 26 January 2010
  - Updated SOP references – DV-OP-0009 to DV-OP-0016, S-T-001 to CA-Q-S- 001 DV-1, DV-QA-003 to DV-QA-003P, DV-QA-024 to DV-QA-024P, DV-QA-0033 to DV-QA-011P.
  - Removed DBPP references.
  - Changed 10.2.3 to read greater than instead of less than.
  - Changed section reference in 10.9.2 from section 0 to section 7.
  - Revised section 10.9.4.1 and 10.9.4.2

- Removed section 12.4
- Revised section 12.5.1

- Revision 0.1, dated 31 December 2009
  - Added criteria to section 9.5 stating that marginal exceedances are not allowed in all programs.
  - Added a not to Table 6 stating that average percent differences are not allowed in all programs when evaluating calibration verification standards.

- Revision 0, dated 01 November 2008
  - Initial Release

Table 1. Analyte List and Standard Reporting Limits (LVI also)

Compound	Water Reporting Limit (µg/L)	Soil Reporting Limit (µg/kg)
Aldrin	0.05	1.7
α-BHC	0.05	1.7
β-BHC	0.05	1.7
δ-BHC	0.05	1.7
γ-BHC (Lindane)	0.05	1.7
α-Chlordane	0.05	1.7
γ-Chlordane	0.05	1.7
Chlordane (technical)	0.5	17
Chlorobenzilate*	0.10	3.3
Chlorpyrifos*	0.05	1.7
DBPP***	2.50	
2,4'-DDD*	0.05	0.33
4,4'-DDD	0.05	1.7
2,4'-DDE*	0.05	0.33
4,4'-DDE	0.05	1.7
2,4'-DDT*	0.05	0.33
4,4'-DDT	0.05	1.7
Diallate*	1.0	33.
Dicofol*	1.0	33.
Dieldrin	0.05	1.7
Endosulfan I	0.05	1.7
Endosulfan II	0.05	1.7
Endosulfan Sulfate	0.05	1.7
Endrin	0.05	1.7
Endrin Aldehyde	0.05	1.7
Endrin Ketone	0.05	1.7
Heptachlor	0.05	1.7
Heptachlor Epoxide	0.05	6.7
Hexachlorobenzene	0.05	1.7
Isodrin*	0.10	1.7
Kepone**	1.0	33.
Methoxychlor	0.10	3.3
Mirex	0.05	1.7
Toxaphene	2.0	10.

* These are non-routine compounds that require a separate calibration, and are analyzed only upon request.

** The laboratory has some clients with permits requiring kepone by method 8081A and 8081B. However, the method warns that kepone may change form during extraction and shift out of the expected retention time window. Kepone is <u>not</u> recommended by 8081A and 8081B.

***Available for analysis by method 8081A only.

Table 2. Typical Instrument Conditions

Parameter	Recommended Conditions*
Injection port temperature	200 °C
Detector temperature	325 °C
Column 1 (HP6890 GC)	Rtx® CLPI: 30 m X 0.32 mm id, 0.5 µm
Column 2 (HP6890 GC)	Rtx®CLPII: 30 m X 0.32 mm id, 0.25 µm
HP6890 GC Temperature program and inlet pressure Columns 1 and 2	110 °C for 1 minute 35 °C/min to 180 °C 20 °C/min to 200 °C 35 °C/min to 235 °C and hold for 1 minute 25 °C/min to 300 °C and hold for 4 minutes 40 °C/min to 310 °C Pressure 20 psi, pulse to 40 psi for 1 minute
Column 3 (HP6890 GC)	DB-35MS: 30 m X 0.32 mm id, 0.5 µm
Column 4 (HP6890 GC)	DB-XLB: 30 m X 0.32 mm id, 0.5 µm
HP6890 GC Temperature program Columns 3 and 4	110 °C for 1 minute 35 °C/min to 245 °C and hold for 1.5 minutes 25 °C/min to 300 °C and hold for 4 minutes 40 °C/min to 310 °C
Injection	1 or 2 µL (5 µL)
Carrier gas	Hydrogen
Make up gas	Nitrogen, 60 mL/min
Y splitter	Restek or J&W or Supelco glass tee (Siltek)

* Variations in instrument conditions may exist in order to facilitate compound separation or to accommodate matrix effects from sample analysis.

NOTE: 4,4'-DDE and dieldrin are closely eluting pairs on the HP-5 column . Endosulfan II and 4,4'-DDD are closely eluting pairs on the 1701 column. For these reasons, these columns are no longer in use in the laboratory.

Table 3. Calibration Levels (μg/mL)

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Individual Mix AB						
Aldrin	0.004	0.01	0.025	0.05	0.075	0.10
α-BHC	0.004	0.01	0.025	0.05	0.075	0.10
β-BHC	0.004	0.01	0.025	0.05	0.075	0.10
δ-BHC	0.004	0.01	0.025	0.05	0.075	0.10
γ-BHC (Lindane)	0.004	0.01	0.025	0.05	0.075	0.10
α-Chlordane	0.004	0.01	0.025	0.05	0.075	0.10
γ-Chlordane	0.004	0.01	0.025	0.05	0.075	0.10
4,4'-DDD	0.004	0.01	0.025	0.05	0.075	0.10
4,4'-DDE	0.004	0.01	0.025	0.05	0.075	0.10
4,4'-DDT	0.004	0.01	0.025	0.05	0.075	0.10
Dieldrin	0.004	0.01	0.025	0.05	0.075	0.10
Endosulfan I	0.004	0.01	0.025	0.05	0.075	0.10
Endosulfan II	0.004	0.01	0.025	0.05	0.075	0.10
Isodrin	0.004	0.01	0.025	0.05	0.075	0.10
Endrin	0.004	0.01	0.025	0.05	0.075	0.10
Endrin Aldehyde	0.004	0.01	0.025	0.05	0.075	0.10
Endrin Ketone	0.004	0.01	0.025	0.05	0.075	0.10
Heptachlor	0.004	0.01	0.025	0.05	0.075	0.10
Heptachlor Epoxide	0.004	0.01	0.025	0.05	0.075	0.10
Hexachlorobenzene	0.004	0.01	0.025	0.05	0.075	0.10
Methoxychlor	0.004	0.01	0.025	0.05	0.075	0.10
Endosulfan Sulfate	0.004	0.01	0.025	0.05	0.075	0.10
Mirex	0.004	0.01	0.025	0.05	0.075	0.10
Appendix IX Standards						
2,4'-DDD	0.005	0.010	0.025	0.035	0.05	0.10
2,4'-DDE	0.005	0.010	0.025	0.035	0.05	0.10
2,4'-DDT	0.005	0.010	0.025	0.035	0.05	0.10
Chlorobenzilate	0.050	0.10	0.25	0.35	0.5	1.0
Chlorpyrifos	0.025	0.050	0.125	0.175	0.25	0.5
DBPP	0.250	0.5	1.25	1.75	2.5	5.0
Diallate	0.50	1.0	2.5	3.5	5	10.
Dicofol	0.050	0.10	0.25	0.35	0.5	1.0
Isodrin	0.025	0.050	0.125	0.175	0.25	0.5
Kepone	0.050	0.10	0.25	0.35	0.5	1.0
Multicomponent Standards						
Chlordane (Technical)	0.10	0.20	0.50	1.0	2.0	N/A
Toxaphene	0.20	0.50	1.0	2.0	5.0	10.0
Surrogates are included the AB Mix calibration mix at the following levels:						
Tetrachloro-*m*-xylene	0.005	0.10	0.025	0.05	0.075	0.10
Decachlorobiphenyl	0.005	0.10	0.025	0.05	0.075	0.10

Table 4. LVI method. Calibration Levels (μg/mL)

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Individual Mix AB						
Aldrin	0.0008	0.002	0.005	0.01	0.015	0.02
α-BHC	0.0008	0.002	0.005	0.01	0.015	0.02
β-BHC	0.0008	0.002	0.005	0.01	0.015	0.02
δ-BHC	0.0008	0.002	0.005	0.01	0.015	0.02
γ-BHC (Lindane)	0.0008	0.002	0.005	0.01	0.015	0.02
α-Chlordane	0.0008	0.002	0.005	0.01	0.015	0.02
γ-Chlordane	0.0008	0.002	0.005	0.01	0.015	0.02
4,4'-DDD	0.0008	0.002	0.005	0.01	0.015	0.02
4,4'-DDE	0.0008	0.002	0.005	0.01	0.015	0.02
4,4'-DDT	0.0008	0.002	0.005	0.01	0.015	0.02
Dieldrin	0.0008	0.002	0.005	0.01	0.015	0.02
Endosulfan I	0.0008	0.002	0.005	0.01	0.015	0.02
Endosulfan II	0.0008	0.002	0.005	0.01	0.015	0.02
Isodrin	0.0008	0.002	0.005	0.01	0.015	0.02
Endrin	0.0008	0.002	0.005	0.01	0.015	0.02
Endrin Aldehyde	0.0008	0.002	0.005	0.01	0.015	0.02
Endrin Ketone	0.0008	0.002	0.005	0.01	0.015	0.02
Heptachlor	0.0008	0.002	0.005	0.01	0.015	0.02
Heptachlor Epoxide	0.0008	0.002	0.005	0.01	0.015	0.02
Hexachlorobenzene	0.0008	0.002	0.005	0.01	0.015	0.02
Methoxychlor	0.0008	0.002	0.005	0.01	0.015	0.02
Endosulfan Sulfate	0.0008	0.002	0.005	0.01	0.015	0.02
Mirex	0.0008	0.002	0.005	0.01	0.015	0.02
Appendix IX Standards						
2,4'-DDD	0.0008	0.002	0.005	0.007	0.01	0.02
2,4'-DDE	0.0008	0.002	0.005	0.007	0.01	0.02
2,4'-DDT	0.0008	0.002	0.005	0.007	0.01	0.02
Chlorobenzilate	0.008	0.02	0.05	0.07	0.1	0.2
Chlorpyrifos	0.004	0.01	0.025	0.035	0.05	0.1
DBPP	0.04	0.1	0.25	0.35	0.5	1.0
Diallate	0.08	0.2	0.5	0.7	1	2
Dicofol	0.008	0.02	0.05	0.07	0.1	0.2
Isodrin (calib from AB mix)	0.004	0.01	0.025	0.035	0.05	0.1
Kepone	0.008	0.02	0.05	0.07	0.1	0.2
Multicomponent Standards						
Chlordane (Technical)	0.004	0.01	0.04	0.1	0.2	0.4
Toxaphene	0.02	0.10	0.2	0.4	1.0	2.0
Surrogates are included the AB Mix calibration mix at the following levels:						
Tetrachloro-*m*-xylene	0.0008	0.002	0.005	0.01	0.015	0.02
Decachlorobiphenyl	0.0008	0.002	0.005	0.01	0.015	0.02

Table 5. Column Degradation Evaluation Mix

Component	Concentration (µg/mL)
4,4'-DDT	0.040
Endrin	0.020

Table 6. LCS/Matrix Spike and Surrogate Spike Levels

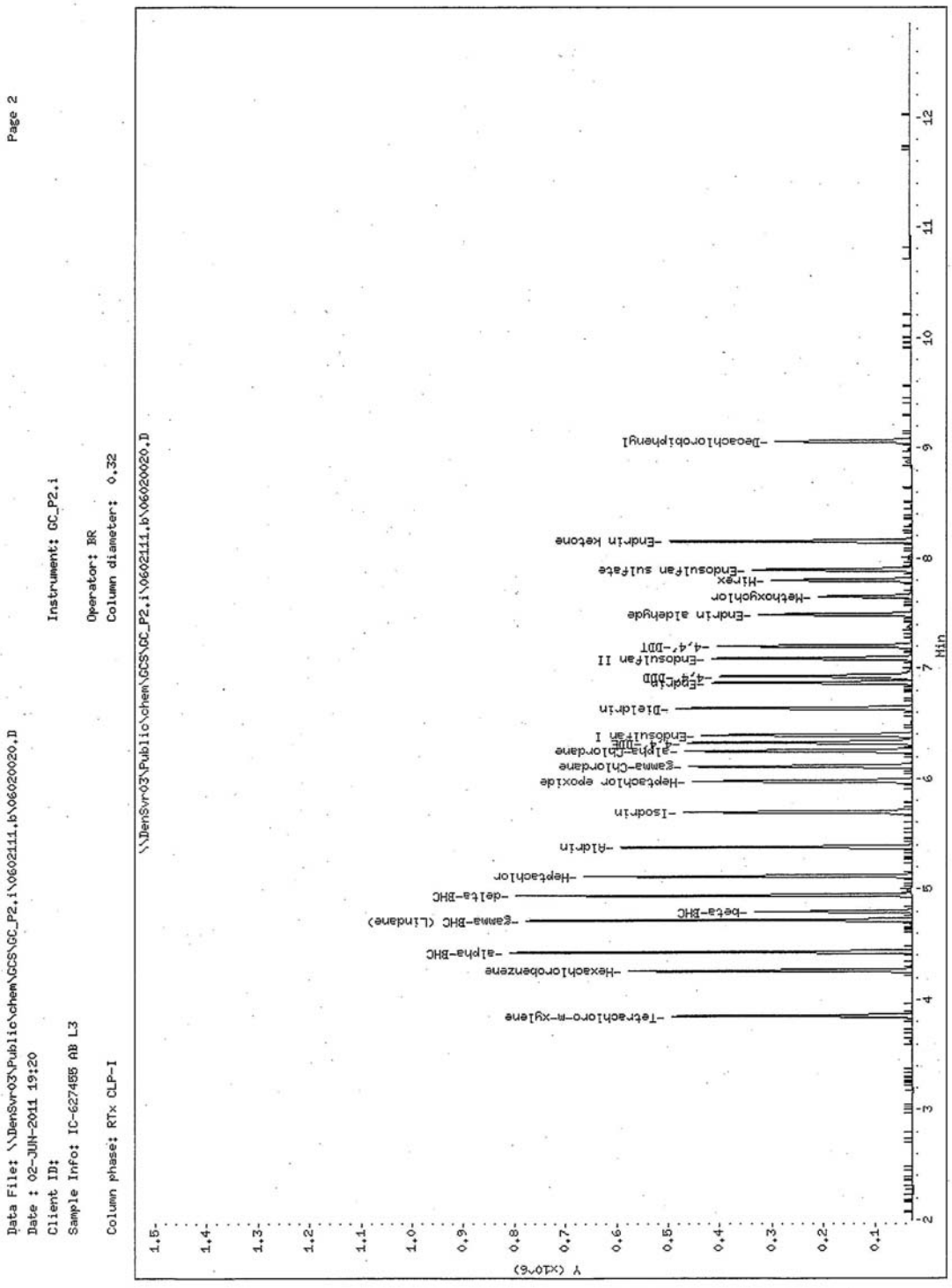
Compound	(µg/L)	(µg/kg)
Aldrin	0.5	16.67
α-BHC	0.5	16.67
β-BHC	0.5	16.67
δ-BHC	0.5	16.67
γ-BHC (Lindane)	0.5	16.67
α-Chlordane	0.5	16.67
γ-Chlordane	0.5	16.67
4,4'-DDD	0.5	16.67
4,4'-DDE	0.5	16.67
4,4'-DDT	0.5	16.67
Dieldrin	0.5	16.67
Endosulfan I	0.5	16.67
Endosulfan II	0.5	16.67
Endosulfan Sulfate	0.5	16.67
Endrin	0.5	16.67
Endrin Aldehyde	0.5	16.67
Endrin Ketone	0.5	16.67
Heptachlor	0.5	16.67
Heptachlor Epoxide	0.5	16.67
Methoxychlor	0.5	16.67
Toxaphene (when required)		
Surrogates		
Decachlorobiphenyl	0.2	6.67
Tetrachloro-*m*-xylene (TCMX)	0.2	6.67

Table 7. Evaluation Criteria and Corrective Actions for Continuing Calibration Verification

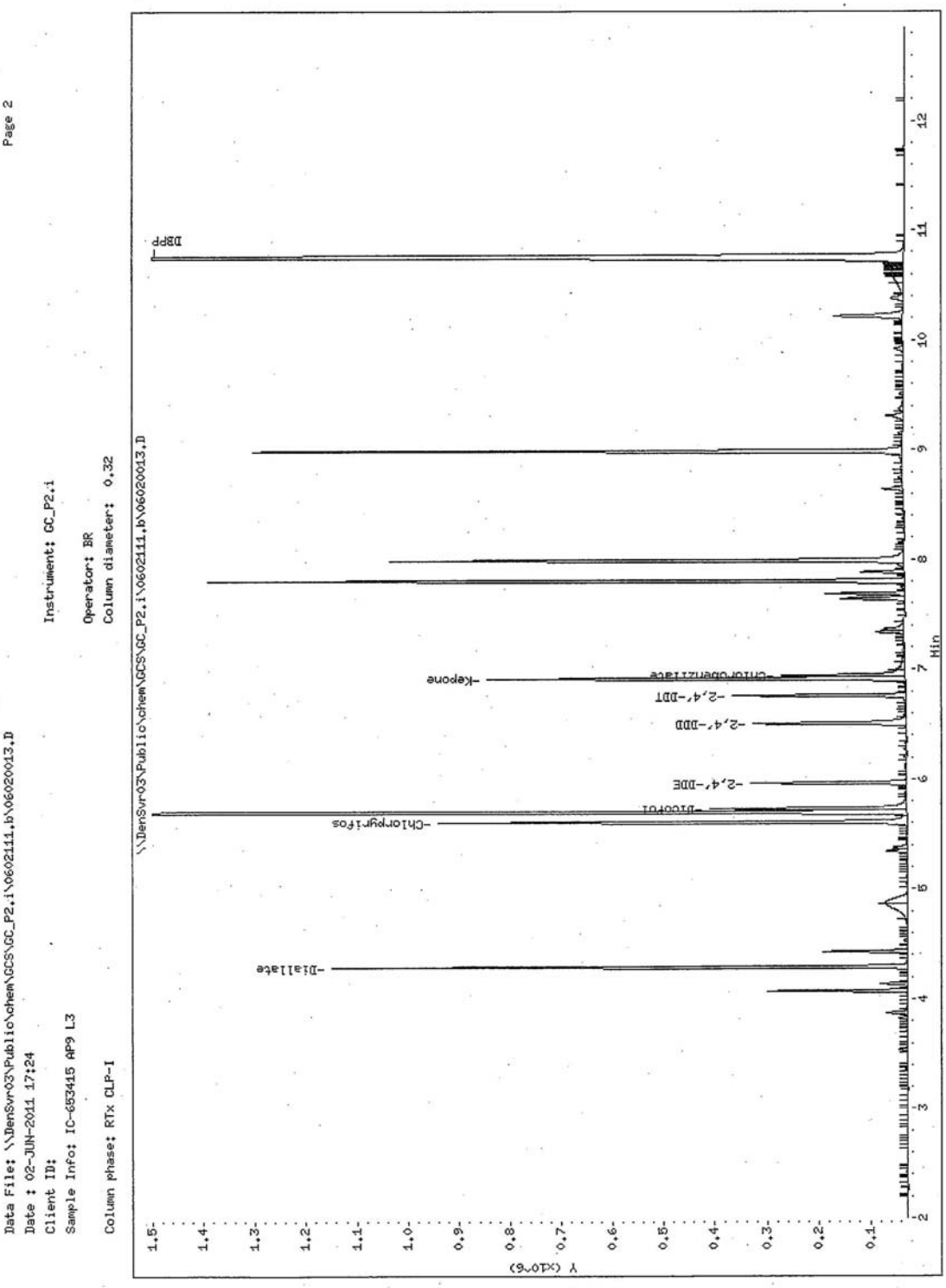
Evaluation Criteria for a Specific Analyte				Evaluation / Corrective Actions
Average %D	**Individual %D**	**RL Standard**	**Client Samples**	
N/A	± 15%	N/A	≥ RL	Calibration is verified for the analyte(s) detected in the sample; no action required.
N/A	Outside of ± 15%	N/A	≥ RL	Calibration is not verified for the analyte(s) detected in the sample. The sample must be re-analyzed using a verified calibration.
± 15%	± 30%	N/A	ND	Calibration is acceptable because analytes were not detected in the sample. An NCM is required.
Outside of ± 15%	N/A	N/A	N/A	Calibration is <u>not</u> verified and corrective action must be taken. NOTE: The exception to this may be those cases where the client has requested a small subset of the analytes typically measured by the method and the %D for each of those analytes is within ± 15%. Corrective action may include clipping the column, changing the liner, or other minor instrument adjustments, followed by reanalyzing the standard twice. If both results pass acceptance criteria, the calibration may be used to process samples. If the overall average %D still varies by more than ±15%, a new calibration curve must be prepared. Reanalyze any samples that were either preceded by or followed by the failed CCV using a verified calibration.
± 15%	< -30% (low)	Detected	ND	Sample results are acceptable because the RL standard indicates that the analyte would have been detected if present in the sample. Explain in an NCM.
± 15%	< -30% (low)	ND	ND	Analyte was not detected in the RL standard, possibly as the result of a calibration drift in the negative direction, and therefore one cannot be sure that the analyte would have been detected in the sample if present. Reanalyze samples with verified calibration.
± 15%	> +30% (high)	N/A	ND	Sample results are acceptable because the CCV failed high, so if the analyte were present in the sample, it would definitely have been detected. Explain in an NCM.

Note: Some programs (e.g., South Carolina) do not allow the average percent difference to be used in evaluating calibration verification standards. Please see the QAS's in the public folders for the current requirements.

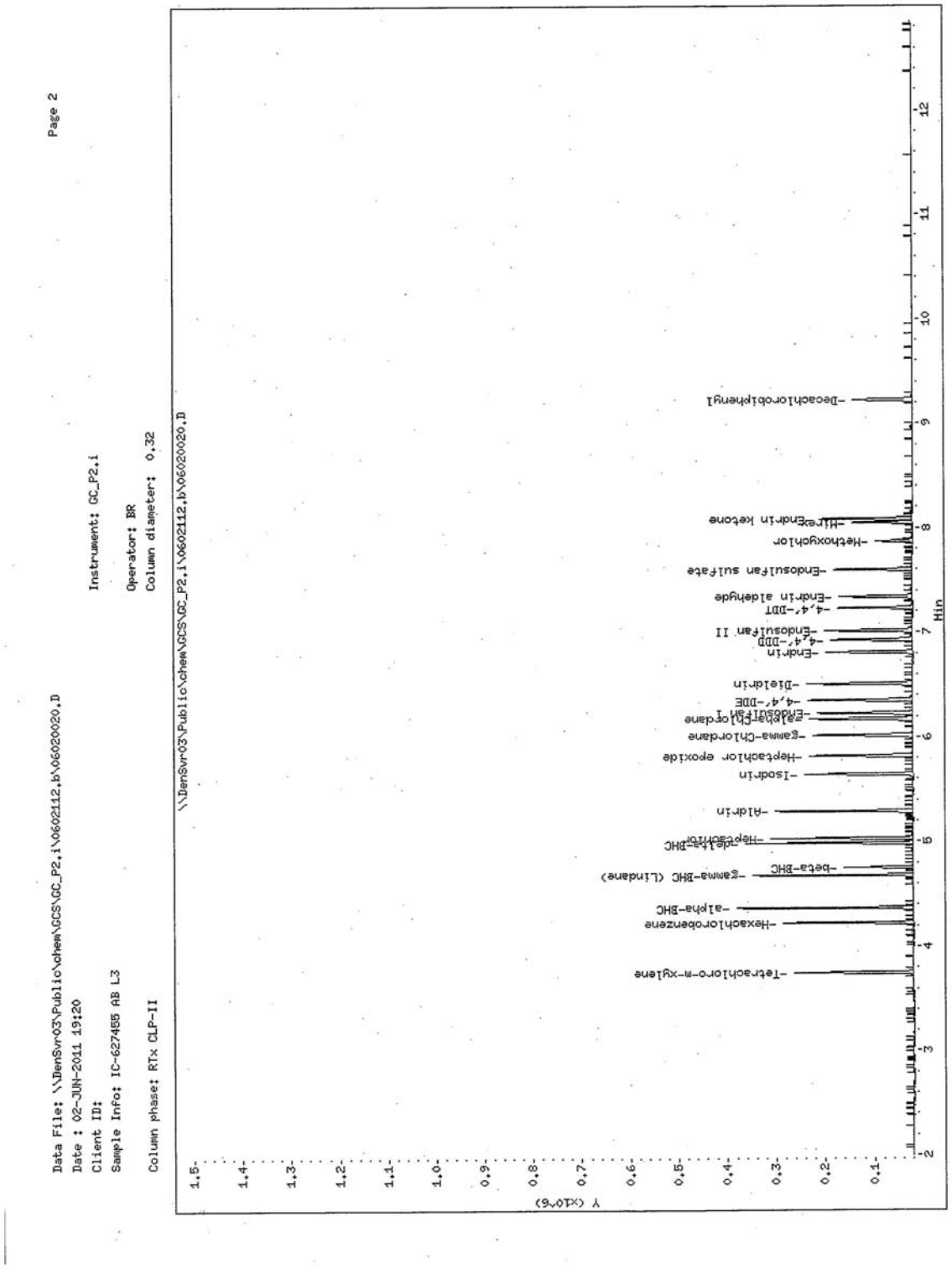
Attachment 1
Example Chromatogram – RTx CLP-I – AB Standard



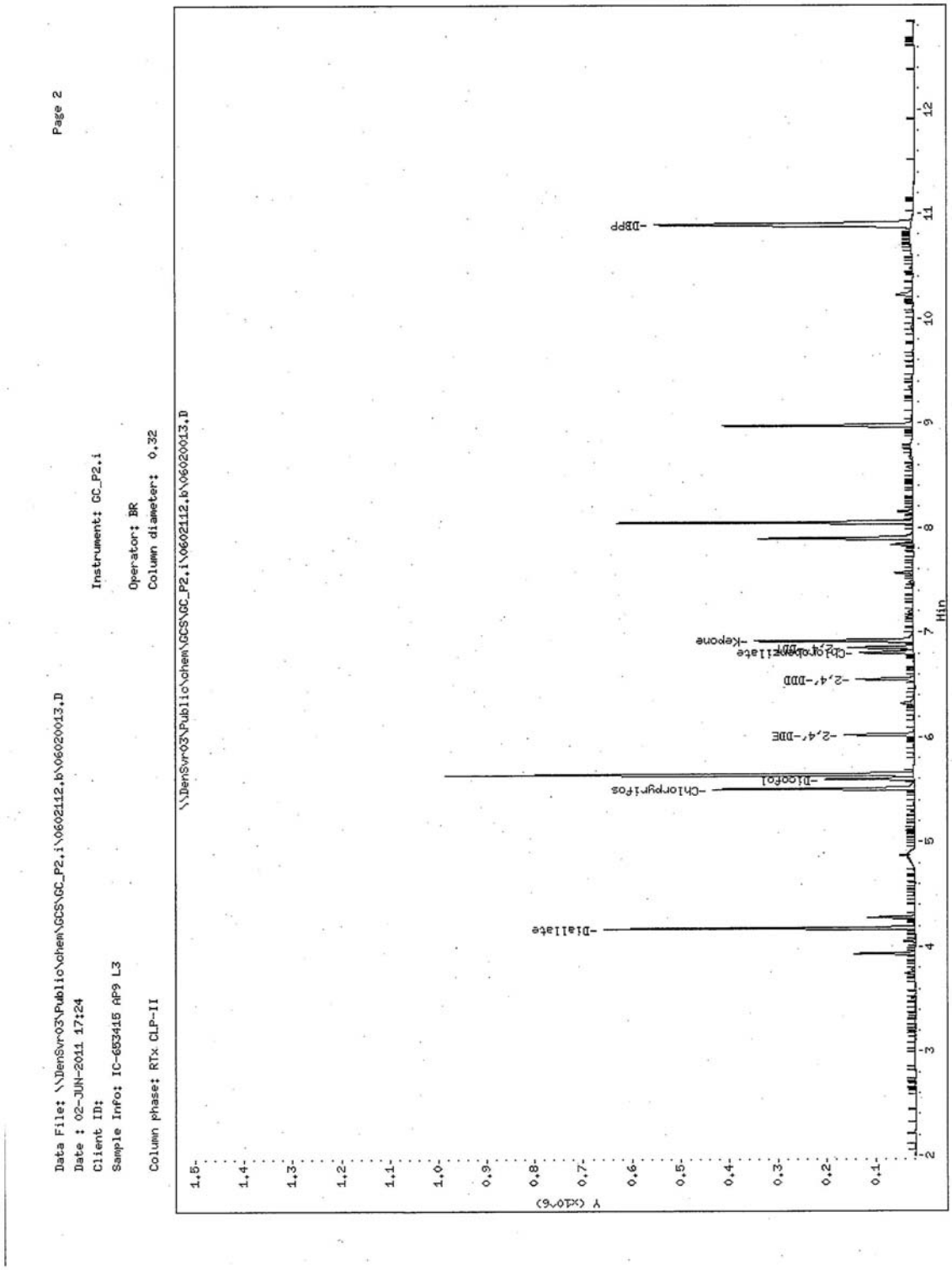
Attachment 2
Example Chromatogram – RTx CLP-I – AP9 Standard



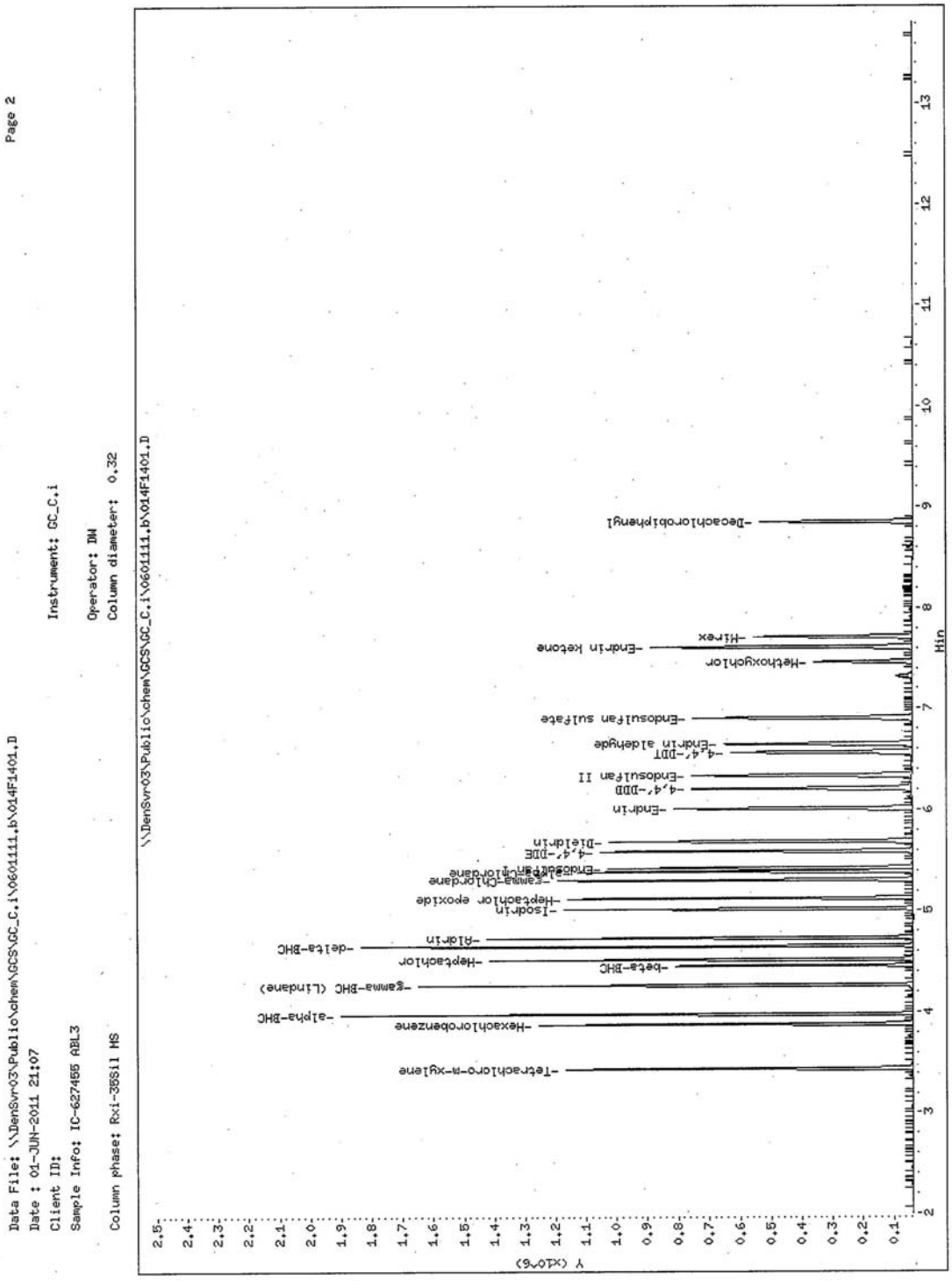
Attachment 3
Example Chromatogram – RTx CLP-II – AB Standard



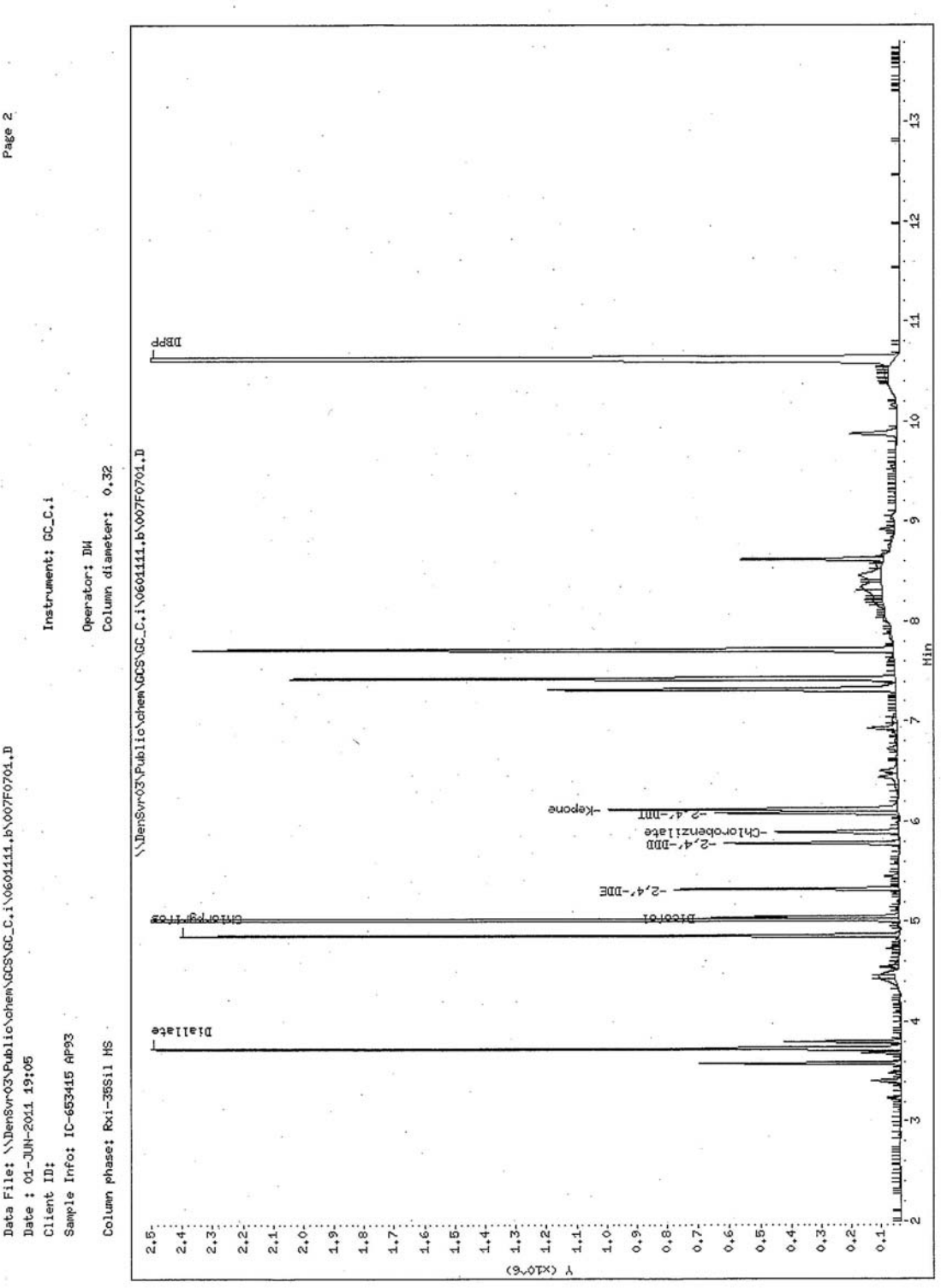
Attachment 4
Example Chromatogram – RTx CLP-II – AP9 Standard



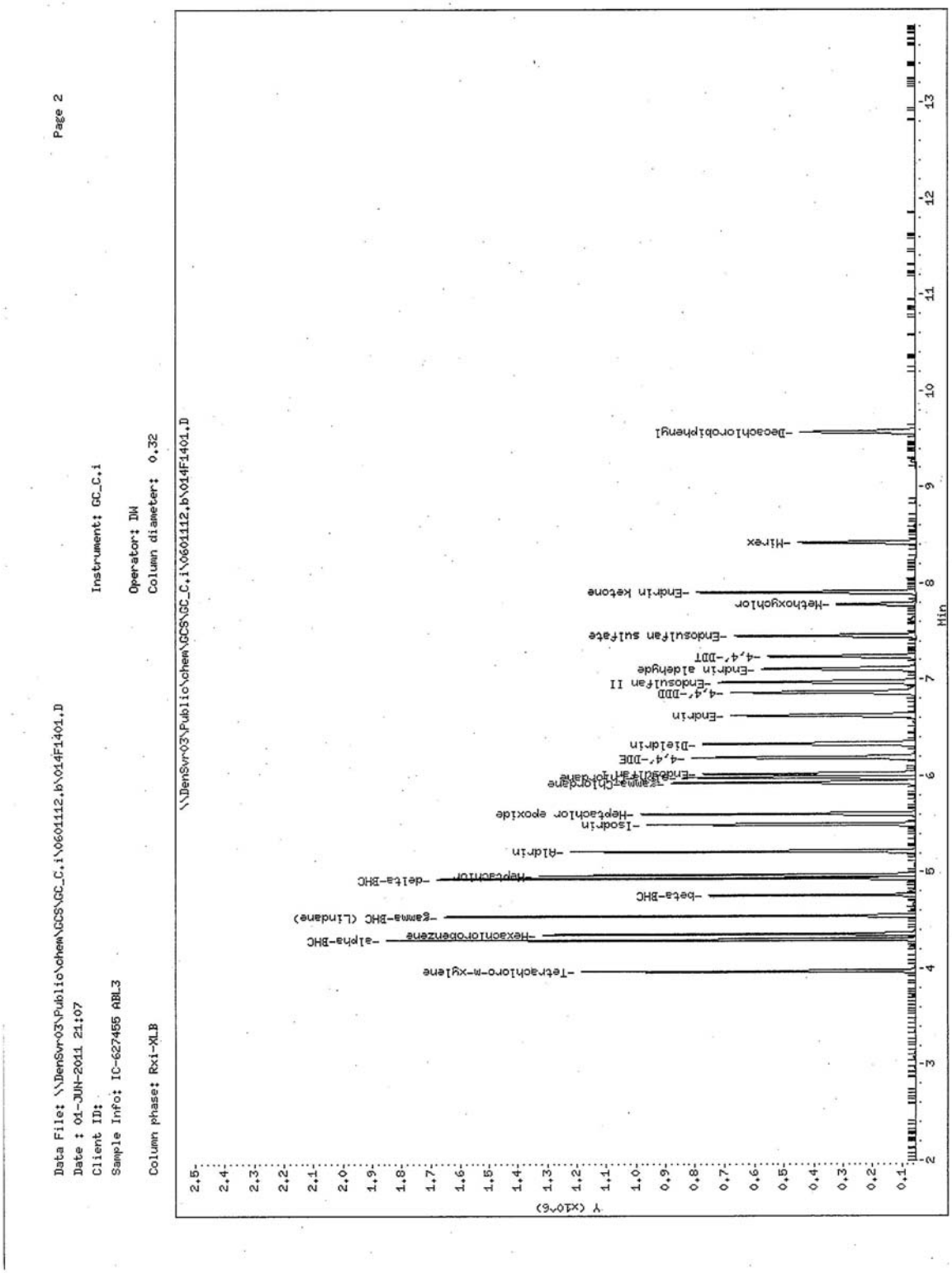
Attachment 5
Example Chromatogram – Rxi-35Sil MS – AB Standard



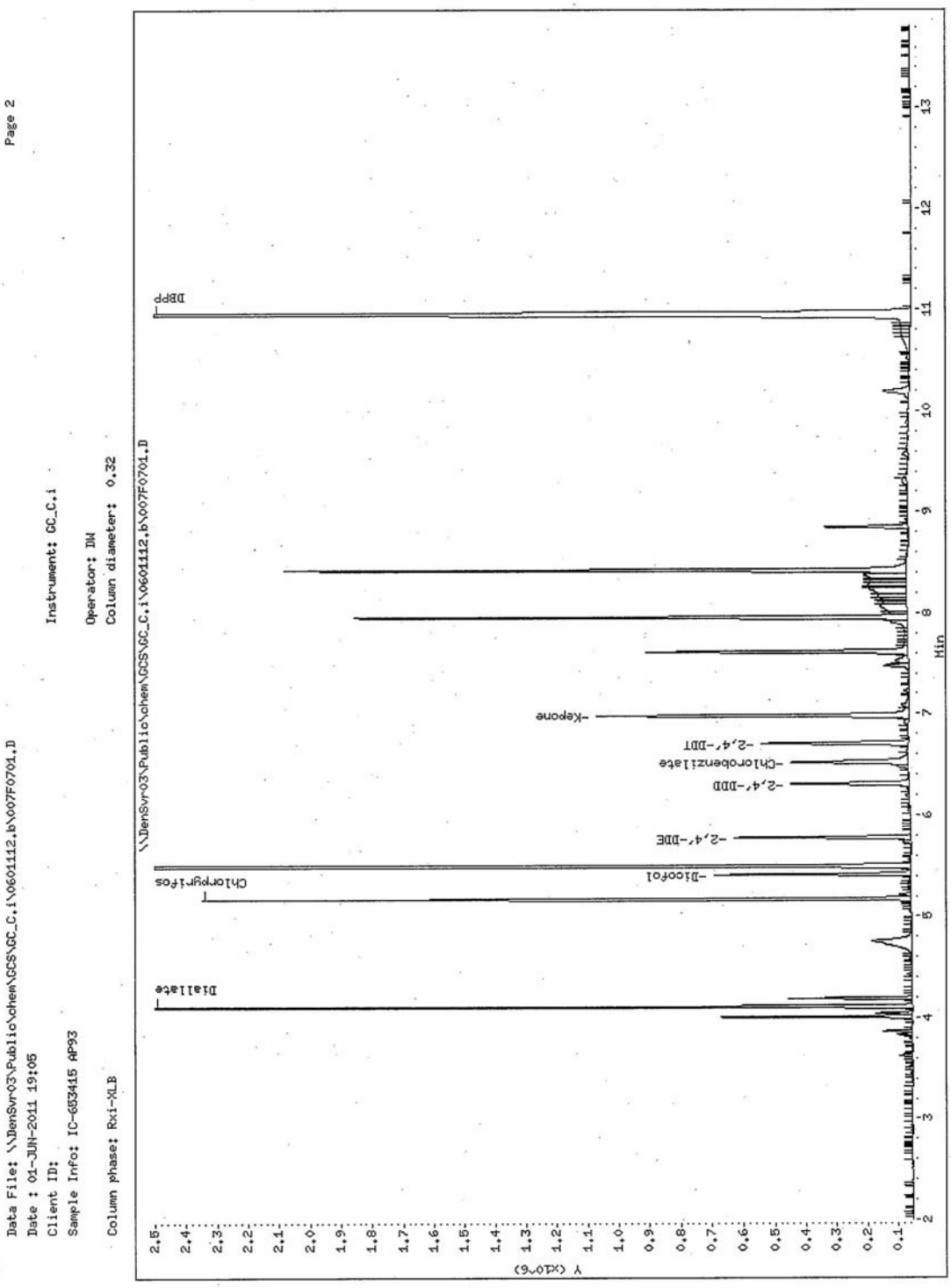
Attachment 6
Example Chromatogram – Rxi-35Sil MS – AP9 Standard



Attachment 7
Example Chromatogram – Rxi-XLB – AB Standard



Attachment 8
Example Chromatogram – Rxi-XLB – AP9 Standard



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Title: Polychlorinated Biphenyls (PCBs) by GC/ECD [SW846 Methods 8082 and 8082A]

Approvals (Signature/Date):

Dennis Jonsrud — 5-22-12 — Date Technical Manager	Adam Alban — 31 May 12 — Date Health & Safety Manager / Coordinator
John P. Morris — 5/21/12 — Date Quality Assurance Manager	Robert C. Hanisch — 5/31/12 — Date Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This SOP describes the procedure for the determination of concentrations of polychlorinated biphenyls (PCB) as Aroclors using the methodology prescribed in EPA SW-846 Method 8082 and 8082A.

1.2 This procedure is applicable to the analysis of extracts of aqueous, solid, and oil samples. When utilized for the analysis of oils, additional cleanup procedures may be required. This procedure also defines the conditions required when using a large volume injection.

1.3 This SOP does not include the procedures for extracting environmental samples. Refer to TestAmerica SOPs DV-OP-0006, DV-OP-0007, DV-OP-0015, DV-OP-0016 and DV-OP-0023 for sample preparation procedures. Refer to SOP DV-OP-0012 for waste dilutions.

1.4 Additional information is provided in this SOP for the inclusion of the analysis of polychlorinated terphenyls (PCT) by the same protocols used for the determination of Aroclors.

1.5 This SOP does not include the determination of the concentration of PCB congeners.

1.6 Analytes, Matrix(s), and Reporting Limits

1.6.1 Tables 1 and LVI-1 list the specific Aroclors that are determined using this procedure and their associated reporting limits (RLs).

2.0 Summary of Method

2.1 Preparation

2.1.1 Aqueous Samples

PCBs are extracted from a one-liter aqueous sample with methylene chloride using a separatory funnel (SW-846 Method 3510). The extract is evaporated to approximately 25 mL and exchanged to hexane. The final extract volume is 10 mL, however depending on special client requirements the final extract volume can also be 1 mL or 5 mL. The extraction procedure is detailed in SOP DV-OP-0006.

2.1.2 LVI Aqueous Samples

PCBs are extracted from a 35 mL aqueous sample with 2 mL of hexane (SW-846 Method 3511). The extraction procedure is detailed in SOP DV-OP-0023.

2.1.3 Solid Samples

PCBs are extracted from solid materials using either sonication or microwave extraction. If sonication extraction is selected the samples are extracted with a 50:50 Acetone:Methlyene Chloride mixture, concentrated down to approximately 25 mL, exchanged with hexane, and brought to a 10 mL final volume. See DV-OP-0016 and DV-OP-0007 for details. If microwave extraction is selected the samples are extracted with a 50:50 Acetone:Hexane mixture, and concentrated down to a 10 mL final volume. See DV-OP-0015 and DV-OP-0007 for details.

2.1.4 Oil Samples

Oil samples are typically prepared by diluting 1 gram of sample to a final volume of 10 mL with hexane. The extraction procedure is detailed in SOP DV-OP-0012.

2.1.5 Wipe Samples

Wipes are typically collected using either filter paper or gauze. These samples can then be extracted using the procedure outlined in SOP DV-OP-0016.

2.1.6 Cleanup Procedures

Cleanup options are discussed in Section 4 below. Instructions for performing various cleanup procedures are detailed in SOP DV-OP-0007.

2.2 Analysis

Samples are analyzed using a gas chromatograph with dual electron capture detectors (ECDs). Specific Aroclor mixtures are identified by the pattern of peaks compared to chromatograms of reference standards. The concentrations of Aroclors in the sample extract are determined using an external standard calibration. Second column confirmation is only performed when requested by the client or as a program requirement. The presence of multiple peaks in the sample serves as confirmation of analyte presence.

3.0 Definitions

3.1 Polychlorinated biphenyls (PCBs): PCBs are a class of organic compounds with 1 to 10 chlorine atoms attached to biphenyl, with a general chemical formula of $C_{12}H_{10-x}Cl_x$. There are 209 possible congeners.

3.2 Aroclor: PCBs were produced as technical mixtures by the chlorination of biphenyl. Production processes were designed to produce mixtures with characteristic chlorine contents. In the United States, most of the PCBs in the environment are in the form of Aroclors, which were produced by Monsanto from the 1930s through 1977. Each Aroclor mixture is identified by a four-digit number, the first two digits of which indicate the number of carbons in the biphenyl ring, i.e., 12, and the second two of which indicate the weight percent of chlorine. For example, Aroclor 1254 has

12 carbons and 54% by weight chlorine. The exception is Aroclor 1016, which has 12 carbons and 42% by weight chlorine.

NOTE: Each specific Aroclor produces a characteristic gas chromatographic pattern that represents the relative amounts of PCB congeners in the formulation. The formulation of the mixtures from batch to batch was fairly consistent, but never exactly the same. In almost all cases, the gas chromatogram can be used as a fingerprint to identify the specific Aroclor. Exceptions occurred for Aroclors 1254 and 1221. In each case, at least one batch was produced under different conditions, which resulted in an Aroclor mixture with the same approximate chlorine content, but with a significantly different distribution of congeners. These odd batches of 1254 and 1221 produce chromatographic patterns that are very different from the typical formulations. Standards for these odd batch Aroclors can be used to aid in the qualitative identification of Aroclors in environmental samples.

3.3 AR1660: Laboratory designation for the mixture of Aroclors 1016 and 1260.

3.4 AR2154: Laboratory designation for the mixture of Aroclors 1221 and 1254.

3.5 AR3262: Laboratory designation for the mixture of Aroclors 1232 and 1262.

3.6 AR4268: Laboratory designation for the mixture of Aroclors 1242 and 1268.

3.7 Polychlorinated Terphenyls: Polychlorinated terphenyls (PCTs) are chemically related to PCBs with the exception that PCTs have an additional phenyl group. The PCTs included in this analysis are AR 5432, AR 5442, and AR 5460. The preparation and analysis is treated the same as for the PCB Aroclor analysis.

4.0 Interferences

4.1 Hydrocarbons can co-elute and thereby mask the Aroclor pattern. The laboratory uses acid cleanup with concentrated sulfuric acid to remove hydrocarbons from solid and oil sample extracts, and for water samples when extracts have noticeable color or whenever there is clear evidence of interferences in the initial sample chromatograms. Acid cleanup removes low-to-medium molecular weight polar organic interferences from sample extracts. Detailed instructions for performing acid cleanup are provided in SOP DV-OP-0007.

All QC is brought through the cleanup process and reported with the samples. An aliquot of all samples and QC is set aside and not brought through the cleanup process. If the QC is out of criteria then the QC that wasn't brought through the cleanup process will be analyzed and used to verify the batch for the samples not brought through clean-up.

4.2 Sulfur will interfere and can be removed using procedures described in SOP DV-OP-0007.

4.3 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Any affected samples are re-analyzed.

4.4 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector.

4.4.1 Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.4.2 Single-component chlorinated pesticides, if present, may co-elute with individual PCB congeners and interfere with the identification and/or quantitation of the aroclors. This can be addressed by analyzing a chlorinated pesticide mixed standard prior to an initial calibration to identify where potential interferences might occur.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (M-E-001 DV), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.1.4 All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the TestAmerica Denver Radiation Safety Officer and the TestAmerica Corporate EH&S Director shall be immediately notified and a letter sent to the Colorado Department of Public Health and Environment.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of

the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Serious or Significant Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hydrogen gas	Explosive	None	The main hazard is flammability. Exposure to moderate concentrations may cause dizziness, headache, nausea, and unconsciousness. Exposures to atmospheres less than 8 to 10% oxygen will bring about sudden unconsciousness, leaving individuals unable to protect themselves. Lack of sufficient oxygen may cause serious injury or death.
Sulfuric Acid	Corrosive Carcinogen	1 mg/m3	Inhalation may cause irritation of the respiratory tract with burning pain of the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema. Causes chemical burns to the respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Causes skin burns. Causes severe eye burns. May cause irreversible eye injury, blindness, permanent corneal opacification.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

A gas chromatographic system with dual columns and dual ECD (^{63}Ni) detectors, and a data system capable of measuring peak area and/or height.

6.2 Computer Software and Hardware

6.2.1 Please refer to the master list of documents and software located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

6.3 Columns

6.3.1 Primary Column: CLPI, 30 m x 0.32 mm id, 0.5 µm coating.

6.3.2 Secondary Column: CLPII, 30 m x 0.32 mm id, 0.25 µm coating.

6.3.3 Additional columns that can be used for confirmation include 30m x 0.32mm id HP-5 or HP-1701.

6.4 Supplies

6.4.1 Autosampler vials, crimp caps with PTFE-faced septa.

6.4.2 Y-splitter, septa, guard columns, ferrules, Siltek injection port liners, Siltek glass wool.

6.4.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.4.4 Various class A volumetric flasks from 5 mL to 250 mL.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Acetone, 99.4% for organic residue analysis. Each lot is tested for purity prior to use per SOP S-T-001.

7.1.2 Hexane, pesticide grade. Each lot is tested for purity prior to use per SOP S-T-001.

7.1.3 Carrier Gas: ≥ 99.99999% pure hydrogen

7.1.4 Make-up Gas: ≥ 99.99980% pure nitrogen

7.2 Stock Standards

7.2.1 All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

7.2.2 All standards must be refrigerated at 0-6 ºC. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before use.

7.2.3 Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of opening or earlier if the vendor indicates an earlier date.

7.2.4 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. The standards must be replaced at least every six months, or sooner if comparison with check standards indicates a problem.

7.3 PCB and Surrogate Stock Calibration Standards

7.3.1 Stock A

For each of the Aroclors listed in Tables 1 and LVI-1, a commercially prepared stock standard solution is obtained. Each stock standard contains the specific Aroclor in pesticide-grade hexane (or in some cases, isooctane) at a concentration of 1,000 µg/mL. The current primary stock source is Accu Standard (AR1221/C-221S-H-10x; AR1016/C-216S-H-10X; AR1232/C-232S-H-10X; AR1242/C-242S-H-10X; AR1248/C-248S-H-10X; AR1254/C-254S-H-10X; AR1260/C-260S-H-10X; AR1262/C-262S-H-10X; AR1268/C-268-H-10X).

7.3.2 Surrogate Stock B

A commercially prepared stock standard solution is obtained that contains the surrogate compounds tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) in acetone, each at a concentration of 200 µg/mL. The current surrogate source is Accu Standard CLP-032-R-Accu.

7.3.3 PCT Stock

A commercially prepared stock standard solution is obtained that contains the individual PCT compounds at a concentration of 35 ug/mL in hexane. The current vendor is Accustandard and the catalog numbers are AR 5432 T432S, AR 5442 T442S, and AR 5460 T460S.

7.4 Intermediate and Working Level Calibration Standard Solutions

7.4.1 Stock C (Level 6 Calibration) Standard Solutions

A Stock C standard solution is prepared for the various Aroclors or combination of Aroclors as summarized in the following table. In each case, the Stock C standard solution is also the highest concentration (i.e., Level 6) calibration standard.

Stock C	Recipe	Conc (µg/mL)	Final Vol (mL)	Final Concentrations (µg/mL)	
AR_1660	0.1 mL of Aroclor 1016 Stock A	1000	100	Aroclor 1016	1.0
	0.1 mL of Aroclor 1260 Stock A	1000		Aroclor 1260	1.0
	0.025 mL of surrogate Stock B	200		TCMX	0.05
				DCB	0.05

Stock C	Recipe	Conc (µg/mL)	Final Vol (mL)	Final Concentrations (µg/mL)	
AR_2154	0.1 mL of Aroclor 1221 Stock A	1000	100	Aroclor 1221	1.0
	0.1 mL of Aroclor 1254 Stock A	1000		Aroclor 1254	1.0
AR_3262	0.1 mL of Aroclor 1232 Stock A	1000	100	Aroclor 1232	1.0
	0.1 mL of Aroclor 1262 Stock A	1000		Aroclor 1262	1.0
AR_4268	0.1 mL of Aroclor 1242 Stock A	1000	100	Aroclor 1242	1.0
	0.1 mL of Aroclor 1268 Stock A	1000		Aroclor 1268	1.0
AR_1248	0.1 mL of Aroclor 1248 Stock A	1000	100	Aroclor 1248	1.0

7.4.2 AR_1660 Calibration Levels

A total of 7 calibration standards are prepared for AR_1660 as summarized in the following table. As needed, the following table can be used to prepare calibration standards for any of the Aroclors, but only the AR_1660 calibration standards include the surrogates. In all cases, measured volumes of the Stock C standard are diluted using pesticide-grade hexane to the final volume indicated in the following table.

Level	Vol of Stock C Used (mL)	Final Volume (mL)	Final PCB Conc (µg/mL)	Final Surrogate Conc (µg/mL)*
1	0.25	10	0.025	0.00125
2	0.5	10	0.050	0.0025
3	1.0	10	0.10	0.005
4	2.5	10	0.25	0.0125
5 (CCV)	50.0	100	0.50	0.025
6	7.5	10	0.75	0.0375
7 (Stock C)	--	--	1.0	0.0500

* Surrogates are in the AR_1660 calibration solutions only. None of the other Aroclor calibration solutions contain the surrogate compounds.

7.4.3 Working Single-Point PCB Calibration Standards

The Level 5 standard in the table above is used for single-point calibrations of the individual Aroclors. These standards are also used as pattern recognition standards.

7.4.4 Polychlorinated Terphenyl Calibration Levels

A total of 7 calibration standards are prepared for PCTs as summarized in the following table. As needed, the following table can be used to prepare calibration standards for any of the PCTs. The level 7 standard is prepared from the stocks described in section 7.3.3 by diluting 1 mL of the stock to 35 mL final volume with hexane. The final concentration of the level 7 standard is 1.0 ug/mL. In all cases, measured volumes of the Level 7 standard are

diluted using pesticide-grade hexane to the final volume indicated in the following table.

Level	Vol of Level 7 Used (mL)	Final Volume (mL)	Final PCT Conc (µg/mL)
1	0.25	10	0.025
2	0.5	10	0.05
3	1	10	0.10
4	2.5	10	0.25
5 (CCV)	5	10	0.50
6	7.5	10	0.75

7.5 Second-Source Standards for Initial Calibration Verification (ICV)

These standards are purchased from a vendor different from the one that supplied the stock calibration standards.

7.5.1 Second-Source Stock A' Aroclor Standard Solutions

Commercially prepared solutions in pesticide-grade hexane (or isooctane) are routinely obtained for Aroclors 1016 and 1260. The Aroclor concentration in each solution is 100 µg/mL. A second source may be obtained for the other Aroclors, if necessary. The current second source is Ultra Scientific (AR1221/PP-291; AR1016/PP281; AR1232/PP301; AR1242/PP311; AR1248/PP341; AR1254/PP-351; AR1260/PP362; AR1262/PP370; AR1268/PP380.

7.5.2 Second-Source Surrogate Stock B' Standard Solution

A commercially prepared solution is obtained containing TCMX and DCB each at a concentration of 200 µg/mL. The current second source surrogate is Ultra Scientific ISM-320.

The working level second-source ICV standard is prepared by combining 0.025 mL of Aroclor 1016 Stock A', 0.025 mL of Aroclor 1260 stock A', and 0.00625 mL of surrogate Stock B', and diluting to a final volume of 10 mL with pesticide-grade hexane. This results in a concentration of 0.25 µg/mL for each of the Aroclors and 0.125 µg/mL for each surrogate. If a second source verification standard is prepared for any of the Aroclors other than the AR_1660 mixture, the surrogates are not added.

7.5.3 PCT Second Source Stock and working level.

A commercially prepared solution of each of the PCT mixes is obtained from a different vendor and typically prepared at a concentration of 100 ug/mL in hexane. The current second source vendors and catalog numbers are AR 5432 Chem Services F290RPS, AR 5442 Chem Services F860RPS, and AR 5460 Chem Services F292RPS.

The working level PCT standard is prepared at a concentration of 0.25 ug/mL by diluting 0.025 mL of each stock to a final volume of 10 mL.

7.6 Continuing Calibration Verification Standard (CCV), 0.5 µg/mL

The working CCV solution is the same as the Level 5 initial calibration standard, as shown in the table in Section 7.4.2.

7.7 RL Standard

The lowest concentration calibration standard (i.e., Level 1) is used as the RL Standard.

7.8 Laboratory Control Standard (LCS) Spiking Solution (AR1660)

NOTE: The LCS/MS spiking solution is prepared and used as part of the scope of the organic preparation SOPs DV-OP-0006, DV-OP-0012, DV-OP-0015, and DV-OP-0016. The following information is provided for reference only.

The working level LCS solution is made from a source different from the source used to make the primary calibration standards. In general it is made up at a concentration of 2 µg/mL in a water-soluble solvent such as acetone. For oil samples extracted by waste-dilution, the standard is made in hexane. The standard contains Aroclors 1016 and 1260 only. Typically 1 mL of this standard is added to 1 liter of water samples, 30 g of soil samples, or 1 g of oil samples. The current LCS vendor is Ultra Scientific PPM-8082 at a concentration of 1000ug/ml. The solution is prepared by diluting 0.5 ml of this stock into 250 ml with acetone solvent.

7.9 Matrix Spike (MS) Spiking Solution:

The working matrix spike solution is the same as the LCS spike solution. Matrix spike samples are prepared by adding 1.0 mL of the working solution to a second one-liter aliquot of the selected aqueous sample, or to a 30-gram subsample of the selected soil sample. The MS duplicate (MSD) is prepared in the same way using a third aliquot of the selected sample.

7.10 Surrogate Spike Solution

7.10.1 Stock Surrogate Spike Solution:

A commercially prepared solution containing 200 µg/mL each of decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX) in acetone is purchased.

7.10.2 Working Surrogate Spike Solution

NOTE: Samples are spiked with the surrogate compounds during sample preparation, which is described in the organic preparation SOPs DV-OP-0006, DV-OP-0012, DV-OP-0015 and DV-OP-0016. The following information is provided for reference only.

The working level surrogate solution is made up to contain DCB and TCMX at a concentration of 0.2 µg/mL. For water and soil samples the solution is made in a water-soluble solvent like acetone. For all oil samples extracted by waste dilution the solution is made in hexane.

7.11 Primer Mix

The primer mix typically consists of a mixture of CCV standards and/or old calibration standards. The concentrations of the components of the primer mix are not critical. The primer mix is injected one or more times prior to analyzing standards and samples to ensure that the chromatographic system is stable, i.e., that retention times are reproducible.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[2]	Reference
Water[1]	Amber glass	1 Liter	Cool, $\leq 6^{o}C$	1 Year to extraction 40 days to analysis	SW-846
Water[3]	3x40 mL vial	40 mLs	Cool, $\leq 6^{o}C$	1 Year to extraction 40 days to analysis	SW-846
Solid	Glass	8 oz	Cool, $\leq 6^{o}C$	1 Year to extraction 40 days to analysis	SW-846

[1]To achieve routine reporting limits, a full one liter of sample is required. Additional one-liter portions are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes.

[2]California, Connecticut, Pennsylvania and South Carolina do not allow the 1 year holding time. For work performed in these states, the extraction holding time is 7 days for water and 14 days for solid.

[3]Samples collected in 40 mL vials will be extracted by SW846 method 3511 followed by analysis using the LVI procedure described in this SOP.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Assurance Program.*

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, *Requirements for Federal Programs*.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and in the Quality Assurance Summaries (QAS) available in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). The current MDL value is maintained in the TestAmerica Denver LIMS. In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. A batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The minimum batch QC in each run is an acceptable method blank or instrument/calibration blank. See QA Policy DV-QA-003P for further details.

9.4 Method blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water (for aqueous sample batches) or Ottawa sand (for solid sample batches) to which the surrogate compounds are added. The method blank is subject to the entire extraction and analysis process.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above one-half the reporting limit (RL) or above one-tenth of the concentration found in the associated samples.

Corrective Action: If the method blank exceeds allowable levels, the source of the contamination must be investigated and all associated samples that produced detections for the contaminant must be re-extracted and reanalyzed. Any samples that produce concentrations more than 10 times the concentration of the same compound as the blank contaminant may be reported with proper flagging and narration.

9.5 Laboratory Control Sample (LCS)

One LCS is prepared and analyzed with each batch of samples. The LCS is

prepared as described in Section 7.8. The LCS is subject to the entire extraction and analysis process.

Acceptance Criteria: The LCS recovery must be within the established control limits. The laboratory's standard control limits are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise. Current control limits are maintained in LIMS.

Corrective Action: If recoveries are not within the established limits, the analytical system is out of control and corrective action must occur. All associated samples must be re-extracted and reanalyzed. If the LCS exceeds the upper control limit then all samples that do not contain detections for the affected compound may be reportable with client consent and proper flagging and narration.

9.6 Matrix Spike (MS) and Matrix Spike Duplicate Samples (MSD)

One MS/MSD pair is required with each analytical batch. Note that some programs (e.g., North Carolina and South Carolina) require preparation and analysis of an MS/MSD pair at a 10% frequency. Preparation of the MS is described in Section 7.9. The MSD is another aliquot of the sample selected for the MS that is spiked in the same manner as the MS.

Acceptance Criteria: The MS and MSD recoveries must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise. The relative percent difference (RPD) between the MS and MSD must be less than the established limit, which is based on statistical analysis of past results, unless otherwise dictated by project requirements. Current control limits are maintained in LIMS.

Corrective Actions: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-extracted and reanalyzed.

If the recovery for any component is outside control limits for both the MS and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-extraction and reanalysis of the batch.

The MS must be analyzed at the same dilution level as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.7 Surrogates

Each field sample, QC sample, and each calibration standard that is used for the AR_1660 initial calibration, is spiked with surrogate compounds decachlorobiphenyl (DCB) and trichloro-m-xylene (TCMX). The surrogate spike solution is prepared as described in Section 7.10.

Acceptance Criteria: The surrogate recoveries must be within the established control limits, which are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise.

Corrective Action: If recoveries of the surrogates in blanks are outside of the control limits, check for calculation or instrument problems. High recoveries might be acceptable if the surrogate recoveries for the samples and other QC in the batch are acceptable. Low surrogate recoveries in the blank require re-extraction and reanalysis of the associated samples especially those that have detections for the targeted compounds that are found in the blank. Samples that are ND may be reportable with proper flagging and NCM.

For field samples, surrogate recovery is calculated and reported for DCB only. TCMX may also be added. However, if both surrogate compounds are added, and recoveries calculated, and either surrogate fails to fall within the control limits, corrective actions are required (this also applies to programs that require the use of only one surrogate). Samples with surrogate recoveries that are above the upper control limit may be reportable with flagging and narration if they do not have reportable detections.

If matrix interference is not obvious from the initial analysis, it is only necessary to re-extract and reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effects, as long as the extraction/instrument system is proven to be working properly.

10.0 Calibration and Standardization

10.1 TestAmerica Denver gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

10.1.1 Use the ChemStation chromatography data system to set up GC

conditions for calibration. See Tables 2 and LVI-2 for typical operating conditions. The conditions described in Table LVI-2 are to be used when performing the large volume injection approach.

10.1.2 Transfer calibration standard solutions into autosampler vials and load into the GC autosampler. Use the ChemStation software to set up the analytical sequence.

10.1.3 Unprocessed calibration data are transferred to the TARGET DB database for processing. After processing the calibration data, print the calibration report and review it using the calibration review checklist (GC and HPLC Data Review Checklist - ICAL). Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration reports are scanned and stored as Adobe Acrobat files on the Public Drive.

10.2 A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new columns.

10.3 Initial Calibration (ICAL)

10.3.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and under the public folder, *Arizona Calibration Training*.

10.3.2 An external standard calibration using seven concentration levels of the AR_1660 mixture is routinely performed. (At least five calibration levels are required.) This provides concentration levels for Aroclor 1016, Aroclor 1260, and the surrogate compounds DCB and TCMX.

NOTE: See Tables 3 and LVI-3 for Calibration Levels. Calibration levels defined in Table LVI-3 are appropriate when the large volume injection approach is used.

NOTE: Prior to analysis of the initial calibration standards it is recommended that a chlorinated pesticide standard (Method 8081) be analyzed as a locater standard to identify potential interferences in samples due to the presence of chlorinated pesticides.

10.3.3 All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.3.4 The calibration curves for Aroclors 1016 and 1260 and the surrogate compounds are modeled either as average calibration factors or as calibration curves using a systematic approach to selecting the optimum calibration function.

10.3.5 The calibration for each of the other Aroclors (see Table 1 or LVI-1) is initially determined using a single, mid-level calibration standard. As needed, the laboratory may generate a multi-point calibration for other

commonly detected Aroclors, such as 1221, 1254, and 1248. When additional multi-point calibrations are developed for the other Aroclors, a second-source ICV standard is also analyzed.

NOTE: Samples from sites known to be contaminated with specific Aroclors should be analyzed using a multi-point calibration curve for the identified Aroclors. This information is provided to the analyst through special instructions in LIMS.

NOTE: Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of the preparation of the calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed

10.3.6 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious mis-injection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

10.3.6.1 The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and

10.3.6.2 The lowest remaining calibration point is still at or below the project reporting limit; and

10.3.6.3 The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and

10.3.6.4 The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average calibration factors or linear regressions, or six levels for second-order curve fits.

10.3.6.5 If a data point is rejected, it must be documented in the sequence log and on an NCM which is filed with each project that is reported from the calibration.

10.3.7 The high and low standard for the initial calibration of the AR_1660 mixture defines the acceptable quantitation range for all of the Aroclors. The low calibration standard must be at or below the RL. If a sample extract contains any Aroclor at a concentration that exceeds the upper range of the calibration, then the extract must be diluted and reanalyzed.

10.3.8 Select 5 major peaks in the analyte pattern (only 3 peaks are usable for Aroclor 1221). The peaks that are chosen should have responses that are at least 25% of the response for the largest peak in the Aroclor pattern. Try

to include one peak that is unique (differs in size or location relative to the other common Aroclors) to the Aroclor being quantitated. Calculate the response of each of the major peaks for each Aroclor, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be co-eluting with contaminant peaks from the quantitation (i.e., peaks that are significantly larger than would be expected from the rest of the pattern).

NOTE: A minimum of three accurate peaks must be used to quantify an Aroclor (two for Aroclor 1221).

10.4 External Standard Calibration

External standard calibration involves the comparison of instrument responses from the samples to the responses from the target compounds in the calibration standards. The area (or height) of a peak in a sample chromatogram is compared to the area (or height) of the peak in the standard chromatograms that appears at the same retention time. The ratio of the detector response to the concentration of the target analyte in the calibration standard is defined as the calibration factor (CF) and is calculated as follows:

$$CF = \frac{A_s}{C_s}$$ Equation 1

Where:

A_s = Peak area (or height) of the target analyte in the calibration standard.

C_s = Concentration of the target analyte in the calibration standard (µg/mL).

10.5 Establishing the Calibration Function

Calibrations are modeled either as average calibration factors or as linear regression curves, using a systematic approach to select the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until calibration acceptance criteria are met.

10.5.1 Linear Calibration Using Average Calibration Factor

The calibration factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

10.5.1.1 The average calibration factor is calculated as follows:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$ Equation 2

Where:

CF_i = The calibration factor for the i^{th} calibration level.

n = The number of calibration levels.

10.5.1.2 The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{CF}} \times 100\% \qquad \text{Equation 3}$$

Where SD is the standard deviation of the average RF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)^2}{n-1}} \qquad \text{Equation 4}$$

10.5.2 Evaluation of the Average Calibration Factor

Plot the calibration curve using the average CF as the slope of a line that passes through the origin. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Acceptance Criteria: The RSD must be ≤ 20%. SW-846 Method 8000B allows evaluation of the grand average across all compounds, but some programs (e.g., DoD, Arizona and South Carolina require evaluation of each compound individually). Check project requirements.

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.5.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated is the dependent variable (y) and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = ax + b$$ Equation 5

Where:

y = Instrument response (peak area or height).

x = Concentration of the target analyte in the calibration standard.

a = Slope of the line.

b = The y-intercept of the line.

For an external standard calibration, the above equation takes the following form:

$$A_s = aC_s + b$$ Equation 6

Where:

A_s = Peak area (or height) of the target analyte in the calibration standard.

C_s = Concentration of the target analyte in the calibration standard (µg/mL).

10.5.4 Evaluation of the Linear Least-Squares Regression Calibration Function

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of this for weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit (RL), and preferably less than the MDL.

Also examine the residuals, but with particular attention to the residuals at the bottom of the curve. If the intercept or the residuals are large, the calibration should be repeated since a higher order regression is not allowed for this method.

The linear regression must have a correlation coefficient (r) ≥ 0.99. Some programs (e.g., USACE, AFCEE and DoD) require a correlation coefficient ≥ 0.995.

Corrective Action: If the correlation coefficient falls below the acceptance limit, the linear regression is unacceptable and the calibration should be repeated since a higher order regression is not allowed for this method.

10.5.5 Second-order regressions and polynomial regression fits of third order or higher are not allowed for this method.

10.6 Second-Source Initial Calibration Verification (ICV)

The second-source ICV standard usually consists of Aroclors 1016 and 1260 only. The stock standards are obtained from a source different than that of the standards used for the calibration. The preparation of the ICV standard is described in Section 7.5. The concentration of each Aroclor in the ICV is 0.25 µg/mL; the concentration of each surrogate is 0.125 µg/mL. The ICV standard is analyzed immediately following the completion of the initial calibration. If any changes are made to the calibration curve types then the ICV must be recalculated to the final form of the ICAL.

If it is necessary to generate a multi-point calibration for any of the other Aroclors, then an ICV standard containing the specific Aroclor(s) is analyzed immediately following the calibration.

Acceptance Criteria: The result for the target analyte(s) in the ICV standard must be within ± 15% of the expected value. Method 8082A allows a control of ± 20%.

Corrective Action: If this is not achieved, the ICV standard, calibration standards, and instrument operating conditions should be checked. Correct any problems and rerun the ICV standard. If the ICV still fails to meet acceptance criteria, then repeat the ICAL.

10.7 Continuing Calibration Verification (CCV), 0.50 ug/mL.

10.7.1 12-Hour Calibration Verification

The 12-hour calibration verification sequence consists of, at a minimum, an instrument blank and the mid-level calibration standard. The 12-hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter when samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration sequence.

NOTE: It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.7.2 It may be appropriate to analyze a mid-level standard more frequently than every 12 hours. The mid-level calibration standard is analyzed as the continuing calibration verification (CCV) standard (see Section 7). At a minimum, this is analyzed after every 20 samples, including matrix spikes,

LCSs, and method blanks. Some programs, specifically drinking water programs, require a CCV after every 10 samples to minimize the number of samples requiring re-injection when QC limits are exceeded. If 12 hours elapse, analyze the 12-hour standard sequence instead.

10.7.3 RL Standard

It may be appropriate to analyze a standard prepared at or very near the reporting limit (RL) for the method between every 10 sample injections (see Section 7.7). This standard can be used to rule out false negatives in client samples in cases where the %D for one or more of the analytes in a bracketing CCV falls below the lower acceptance limit and the samples contain no analytes above the reporting limit. The results for the RL standard are not evaluated unless the previous CCV fails acceptance criteria or in the case of matrix effect to confirm the ability to see at the reporting limit.

This procedure is not used when Method 8000C is required.

10.7.4 Acceptance Criteria for Continuing Calibration Verification (CCV)

10.7.4.1 Detected Analytes (≥ RL)

For any analyte detected at or above the reporting limit (RL) in client samples, the percent difference (%D) for that analyte in the preceding and following CCVs (i.e., bracketing CCVs) or 12-hour calibration, on the column used for quantitation, must be within ± 15%. Method 8082A requires a control of ± 20%. If a confirmation column is required (see Section 12.5), the CCV criteria must be met on both columns.

In some cases, the nature of the samples being analyzed may be the cause of a failing %D. When the %D for an analyte falls outside of acceptance criteria in the CCV, and that analyte is detected in any or all of the associated samples, then those samples must be reanalyzed to prove a matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect; if so desired by the client.

Refer to Section 11 for which result to report.

The %D is calculated as follows:

$$\%D = \frac{\text{Measured Conc} - \text{Theoretical Conc}}{\text{Theoretical Conc}} \times 100 \qquad \text{Equation 8}$$

10.7.4.2 Analytes Not Detected (< RL)

For any analyte not detected (ND) in client samples, the %D for that analyte in the bracketing CCVs should also be within acceptance criteria.

However, if the CCV %D exceeds the upper control of the acceptance criteria and the sample results are ND, it still may be possible to report sample results. In this case, the client should be consulted and an NCM written.

If the CCV % D falls below acceptance criteria and sample results are ND, but the target analytes are detected in the RL Standard, it may still be possible to report sample results, since the detection of the analyte(s) in the RL Standard indicate that there was sufficient sensitivity to detect the analyte(s) in the samples. In this case, the client should be consulted and an NCM written. This would only be used in cases where the matrix is affecting CCV recovery and dilution of the affected sample(s) is not an alternative.

NOTE: The state of Arizona requires the use of Method 8000C and does not allow the use of the average %D.

10.8 Retention Time (RT) Windows

10.8.1 Determine the retention time (RT) windows for the 5 major peaks selected for each Aroclor (3 peaks for Aroclor 1221). The AR1016 windows will be used to establish retention time windows for AR1221, AR1016, AR1232, AR1242, and AR1248. The AR1260 windows will be used to establish retention time windows for AR1254, AR1260, AR1262, and AR1268.

10.8.2 Determine new RT windows each time a new column is installed or annually.

10.8.3 Inject a standard containing all analytes at least once each day over a 72-hour period.

10.8.4 Calculate the mean and standard deviation of the three RTs for each analyte as follows:

$$\text{Mean RT} = \overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n} \quad \text{and} \quad SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RT_i - \overline{RT}\right)^2}{n-1}} \qquad \text{Equations 9 and 10}$$

Where:

RT_i = Retention time for the i^{th} injection.
n = Number of injections (typically 3).
SD = Standard deviation.

10.8.5 The width of the RT window for each analyte is set at ± 3 times the standard deviation of the RTs determined for each analyte over the 72-hour period. For multi-response analytes, use the RT of major peaks.

10.8.6 The center of the RT window for each analyte is the RT from the last of the three analyses of the standard.

10.8.7 The center of the window for each analyte is updated with the RT from the last of the three standards measured for the 72 hour RT study, the level 4 standard of the ICAL, or the CCV at the beginning of the analytical sequence. The width of each window remains the same until new windows are generated following the installation of a new column, or in response to an RT failure.

10.8.8 If the width of the RT window, as calculated above, is less than ±0.03 minute, use ± 0.03 minute as the RT window width. This allows for slight variations in RTs caused by sample matrix.

Acceptance Criteria: The RT for each compound in each CCV analysis must be within the RT windows established by the daily initial CCV.

Corrective Action: If a target analyte falls outside the established RT window in a CCV standard, either adjust the center of the window based on the CCV, or investigate the problem and calculate new RT windows. All samples analyzed after the last acceptable CCV must be reanalyzed.

10.8.9 Sample Retention Time Criteria

The surrogate must fall within the established RT window. Target analyte peaks must be within the established RT window to be reported as such. If the surrogate RT indicates a RT shift, it may be possible to accept a target analyte peak if it has not shifted relative to the surrogate peak. The presence of a definitive aroclor pattern will be positive evidence of a hit and may supersede RT window criteria. An NCM should be written to explain this case.

10.8.10 Daily Retention Time Windows

The centers of the retention time windows are adjusted at the beginning of each analytical sequence based on the daily initial CCV.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed

must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Preparation

11.3.1 Sample preparation for aqueous samples is described in SOP DV-OP-0006.

11.3.2 Sample preparation for solid samples is described in SOPs DV-OP-0016 and DV-OP-0015.

11.3.3 Cleanup and concentration of sample extracts are described in SOP DV-OP-0007. Note that it is highly recommended that all samples be checked for sulfur and cleaned up if necessary before the samples are analyzed on the instrument. Sulfur can contaminate the column and hinder the quantification of certain compounds.

11.3.4 The final extract volume in hexane is 10 mL.

11.3.5 Use hexane to dilute sample extracts, if necessary.

11.4 Gas Chromatography

Chromatographic conditions for this method are presented in Tables 2 and LVI-2. Use the ChemStation interface to establish instrument operating conditions for the GC. Raw data obtained by the ChemStation software is transferred to the TARGET DB database for further processing. The data analysis method, including peak processing and integration parameters, calibration, RT windows, and compound identification parameters, is set up in the TARGET DB software.

11.5 Sample Introduction

All extracts and standards are allowed to warm to room temperature before injection. An autosampler is used to introduce samples into the chromatographic system by direct injection of 1 or 2 µL of the sample extract. For LVI analysis 10 µL of sample extract is introduced into the chromatographic system. Samples, standards, and QC samples must be introduced using the same procedure. Use the ChemStation interface to set up and run the analytical sequence. Sample injection and analysis are automated and may proceed unattended.

11.6 Analytical Sequence

An analytical sequence starts with a minimum five-level initial calibration (ICAL) or a daily calibration verification. Refer to Tables 3 and LVI-3 for the calibration levels used.

11.6.1 The daily calibration verification includes analysis of the 12-hour calibration sequence (Section 10.7.1) and updating the retention time windows (Section 10.8.7)

11.6.2 If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration

verification. Any samples that were not bracketed by a closing CCV must be reanalyzed in the new 12 hour sequence.

11.6.3 The following is a typical analytical sequence for routine sample analysis:

- Primer (Injection of any standard that contains any of the analytes to establish the stability of the chromatographic system.)
- Hexane instrument blank
- Daily initial CCV (Unless an ICAL is performed, which is immediately followed by the second-source initial calibration verification.)
- 10 sample injections (The first set of samples analyzed usually includes the method blank and the LCS, and may include matrix spikes.)
- CCV
- Followed by cycles of 10 sample injections and a CCV, as needed
- Closing CCV, instrument blank, and RL Standard

11.7 Retention Times

The centers of the RT windows determined in section 10.8 are adjusted to the RT of each individual peak as determined in the 12-hour calibration verification. The RT window must be updated at the beginning of each analytical sequence.

11.8 When a sample result exceeds the upper calibration range, then that sample extract is diluted to obtain a result in the upper half of the calibration range and reanalyzed. Any samples that were analyzed immediately following the high sample are evaluated for carryover. If the samples had target analyte detections at or above the RL, the samples must be reanalyzed to rule out carryover.

11.9 Upon completion of the analytical sequence, transfer the raw chromatography data to the TARGET DB database for further processing. Review chromatograms online and determine whether manual data manipulations are necessary. All manual integrations must be justified and documented. See DV-QA-011P for requirements for manual integration. Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature. Alternatively, the manual integration may be processed manually. In the latter case, print both the both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration.

11.10 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD.

11.10.1 The data package should consist of the checklist, sequence(s), ICAL cover, ICAL summary and history used for data quantitation and the prep batch paperwork.

11.10.2 Perform a level 1 data review and document the review on the data review

checklist, GC Data Review Checklist/Batch Summary (See SOP DV-QA-0020.)

11.10.3 Submit the data package and review checklist to the Data Review Group for the level 2 review. All manual integrations must be evaluated by the peer reviewer and this review must be documented by date and initial on the annual integration summary report and/or the level 2 review checklist. For Federal projects and certain client specified projects, the documentation of the manual integration review must be scanned and attached to the project tin the LIMS to be included with the Level 4 data package. The level 2 review is documented on the review checklist initiated at the level 1 review. The data review process is explained in SOP DV-QA-0020.

12 Calculations / Data Reduction

12.1 Detailed equations can be found in the corporate SOP CA-Q-S-005 “Calibration Curves” and under the public folder, Arizona Calibration Training.

12.2 Qualitative Identification of Aroclors

Retention time windows are used for identification of Aroclors, but the “fingerprint” produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3 Quantitation of Aroclors

Quantitation of Aroclors is accomplished using 5 major peaks (3 peaks for Aroclor 1221). The peaks must be within the established retention time windows. If there is an interference that affects the accuracy of results, the analyst may use as few as 3 major peaks (2 peaks for Aroclor 1221). The same peaks that are used for sample quantitation must be used for standards and QC quantitation.

12.4 Second column confirmation of Aroclors is performed only when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

NOTE: USACE and DoD projects require the use of second-column confirmation of Aroclors unless the project work plans (SOW, SAP, QAPP, etc.) specify single-column analysis.

NOTE: South Carolina requires second column confirmation.

NOTE: DOE requires second column confirmation with flagging if the results vary by more than 25% RPD.

NOTE: Method 8082A indicates that second column confirmation is necessary when the sample composition is not well characterized.

12.5 Dual Column Quantitation

NOTE: Dual column quantitation is not routinely performed for PCB analysis. This section is included for those clients/projects that require dual column confirmation.

12.5.1 A primary column is designated. If the continuing calibration fails for one of the columns then the appropriate corrective action must be taken. The result from the primary column is normally reported. The result from the secondary (confirmatory) column is reported if any of the following is true:

12.5.1.1 There is obvious chromatographic interference on the primary column.

12.5.1.2 The difference between the result for the primary column and the result for the secondary column is > 40% and chromatographic interference is evident on the primary column.

12.5.2 Dual Column Results With > 40% RPD

12.5.2.1 If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, the higher of the two results is reported unless there is obvious interference documented on the chromatogram.

12.5.2.2 If there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

12.5.2.3 If there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.

12.5.2.4 The RPD between two results is calculated using the following equation:

$$\%RPD = \frac{\left|R_1 - R_2\right|}{\frac{1}{2}\left(R_1 + R_2\right)} \times 100\% \qquad \textit{Equation 14}$$

Where R_1 is the result for the first column and R_2 is the result for the second column.

12.5.3 If total Aroclors and dual column quantitation is requested, then total aroclors will be calculated for each column by summing the detections on each column. If the results for total aroclors from the primary column differs from the total aroclor result from the secondary column by more than 40%, the total aroclors result from the primary column will be reported and the data will be flagged accordingly.

12.6 Surrogate Recovery

12.6.1 Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB).

12.6.2 In cases where the addition of the surrogate tetrachloro-*m*-xylene (TCMX) is required, its recovery is calculated and reported. In cases where both surrogates are added and recoveries calculated, the recovery of each surrogate is evaluated and corrective action must be taken if either surrogate recovers outside of the established control limits and matrix interference is not evident. Depending on project requirements, corrective action may be necessary only if DCB and TCMX are both outside of acceptance limits.

12.7 Calibration Range and Sample Dilutions

12.7.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for any analyte, they must be reanalyzed to rule out carryover unless other objective evidence indicates that the detection is not the result of carryover. Such evidence may include an observation where carryover was not observed when blanks or other samples were analyzed after a sample with similar high concentration or when the detection in the sample with suspected carryover is much higher than the expected amount of the carryover (i.e., the suspect sample's concentration is similar to or higher that the sample run previous to it). It may also be necessary to dilute samples because of matrix interferences.

12.7.2 If the initial diluted run has no hits or hits below 20% if the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.7.3 Guidance for Dilutions Due to Matrix Interference

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

12.7.4 Reporting Dilutions

Some programs (e.g., South Carolina, DoD, and AFCEE) and some projects require reporting of multiple dilutions (check Method Comments in

LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported.

12.8 Interferences are Observed in Samples

12.8.1 Dual column analysis does not entirely eliminate interfering compounds. Complex samples with high background levels of interfering organic compounds can produce false positive and/or false negative results. The analyst must use appropriate judgment to take action as the situation warrants.

12.8.2 Suspected Negative Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. Elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.8.3 Suspected Positive Interferences

If no further cleanup is reasonable and interferences are evident that are suspected of causing false positive results, consult with the laboratory Project Manager to determine if analysis using additional confirmation techniques is appropriate for the project. Use of additional confirmation columns is another possible option. At a minimum, the Data Review Template prepared by the analyst should include the following comment for inclusion in the case narrative:

"Based on review of the chromatograms for samples _____, it is my opinion that the evident interferences may be causing false results.

Date _____ Analyst _____"

12.9 Identifying and Reporting PCBs

12.9.1 In samples where the PCB pattern matches an individual Aroclor reasonably well, the samples should be quantified and reported as usual. When there are numerous PCB peaks present but there are no good matches to any individual Aroclor, choose the Aroclor (or Aroclors) that most closely match the sample and quantify the peaks as that Aroclor. The sample should not be reported as "not detect" based solely on the absence of a good match to a single Aroclor mixture. Multiple Aroclors should only be reported if their patterns are reasonably well separated. For example, 1232 and 1254 could be reported together, but not 1242 and 1248. See Attachment 1 for additional information on identifying Arochlors.

NOTE: When reporting and quantifying PCBs that do not closely match an Aroclor standard, it is absolutely essential and mandatory that this is explained in the report narrative.

12.9.2 Some example text that can be used in the report narrative is presented below:

Sample XXXX appears to contain PCBs based on the presence of numerous PCB peaks. However, due to weathering or other environmental processes, the PCBs in the sample do not closely match any of the Aroclor standards we use to calibrate our instruments. We quantified and reported the sample as Aroclor ZZZZ (or as a mixture of Aroclors ZZZZ and YYYY). Due to the poor match with the Aroclor standard(s), there is increased qualitative and quantitative uncertainty associated with this result. This approach is consistent with the guidance in section 7.9.3 of SW846 method 8082A. If these results do not meet the needs of your project then we would suggest a further analysis of the sample. Depending on the objectives, this may include congener-specific analysis by 8082A; or analysis a more specific method (e.g., method 1668 or an adaptation of method 8270) for PCB congeners or PCB homolog totals.

Some clients may insist on ND reporting if the patterns are not clear. In that event, we should add information to the project file to indicate that the information in this guidance have been communicated to the client, together with the client's instructions. In addition, in the event of a poor match to patterns, we would still insist on a narrative as suggested in the previous paragraph.

12.9.3 Sample Matrix Issues

12.9.3.1 In some cases when analyzing for multi-component analytes, the sample matrix is so complex that it would obliterate any possible pattern that would allow us to identify the analyte. When this happens, it is true that the analyte is not detected at the normal detection limit. However, it is true that we could not have detected the analyte at the normal detection limit. Even if the analyte was present, we would not be able to recognize it.

12.9.3.2 When this occurs, the sample must be analyzed at a dilution that would allow us to detect the analyte, and the reporting limit should be the one appropriate for that dilution. Reporting a non-detect at the normal reporting limit is not an acceptable practice.

12.9.3.3 Some clients may insist on ND reporting if the patterns are not clear. In that event, add information to the project file to indicate that the information in this guidance have been communicated to the client, together with the client's instructions. In addition, in the event of a poor match to patterns, a narrative comment as suggested in the previous paragraph is still required.

12.9.4 Background on PCBs

12.9.4.1 PCBs were widely used in a variety of products prior to being banned in the 1970's. The most common usages were in electric motors and transformers. They were manufactured by gas phase chlorination of a biphenyl molecule. The nomenclature, in general, describes the weight percent of chlorine in the final product. Thus, Aroclor 1254 was produced by chlorinating a quantity of biphenyl until the resulting product was 54% chlorine by weight. Aroclor 1242 was 42% chlorine by weight.

12.9.4.2 PCBs were manufactured in batch processes, so there were slight variations between batches, but in general each Aroclor had a very reproducible pattern of chlorinated biphenyl isomers (congeners). With few exceptions, when we detect PCBs in the environment the initial contaminant was one of the Aroclors.

12.9.4.3 The one exception to the nomenclature of the Aroclors is Aroclor 1016. In the 1960's researchers started to find PCBs in fish tissue in the Great Lakes. The primary congeners appearing in the fish were pentachlorobiphenyls. The manufactures of PCBs devised a synthetic process that created an Aroclor with very similar properties to Aroclor 1242, but minimized the formation of pentachlorobiphenyl molecule. Aroclor 1016 41% chlorine by weight and as result it can be difficult to distinguish from 1242.

12.9.4.4 While the pattern of congeners was quite reproducible in the pure products once in the environment the pattern changes. The lesser chlorinated PCBs are more water soluble and are more volatile, while the more highly chlorinated PCBs bind to solids and sediments more strongly. As examples, landfill gas condensates tend to have a bias toward the lesser chlorinated congeners because they are more volatile. River sediments near source of PCBs tend to have a bias toward the more highly chlorinated congeners because the accompanying lesser chlorinated congeners were more water soluble. Downstream from the source of contamination, however, there will be a bias toward the less chlorinated congeners because the more heavily chlorinated congeners were trapped in the sediments near the outfall. Anaerobic and aerobic microbial degradation reduce the concentrations of some congeners and an increase in concentrations of others. Although they are rarely the primary mechanisms, oxidative and photolytic processes are also selective, impacting some congeners more than others.

12.9.4.5 As a result, PCBs in the environment rarely have an exact match to the Aroclor standards that we use to calibrate our instruments. There is inevitably some level of judgment required to choose the Aroclor that has the best match to the sample in questions. Sometimes this is straightforward, but other times the judgment is difficult and can be controversial. In the worst cases, we can

have situations where there are clearly PCB peaks throughout a chromatogram, but there is no good match with any of the Aroclors. It is recommended that at a minimum there must be some peak groupings present that are characteristic of an aroclor pattern in order to indicate a positive detection.

12.10 Calculations

12.10.1 Concentration of Analyte in Sample Extract

Depending on the calibration function used, the concentration of the analyte in the sample extract is calculated as follows (see Section 10.5 for details on establishing the calibration function):

12.10.1.1 Average Calibration Factor:

$$C_e = \frac{A_e}{\overline{CF}} \qquad \text{Equation 12}$$

12.10.1.2 Linear Regression:

$$C_e = \frac{[A_e - b]}{a} \qquad \text{Equation 13}$$

Where:

C_e = Concentration of the analyte in the sample extract (ng/mL).

A_e = Peak area for the analyte in the sample extract injection.

b = y-intercept of the calibration fit.

a = Slope of the calibration fit.

12.10.2 Concentration of Analyte in Original Sample

The concentration of the analyte in the original sample is calculated as follows:

$$C_{sample} = \frac{C_e}{1000\ \frac{ng}{\mu g}} \times \frac{V_e}{V_s} \times DF \qquad \text{Equation 14}$$

Where:

C_{sample} = Concentration of analyte in original sample (µg/L or µg/kg).

C_e = Concentration of analyte in sample extract injected in GC (ng/mL).

$1000\frac{ng}{\mu g}$ = Factor to convert ng/mL to µg/mL.

V_e = Volume of sample extract (mL).

V_s = Volume (or weight) of original sample (L or kg).
DF = Dilution Factor (post extraction dilutions)

12.10.3 LCS and Surrogate Spike Recovery Calculation

LCS and surrogate spike recoveries are calculated using the following equation:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\% \qquad \textit{Equation 15}$$

12.10.4 MS and MSD Recovery Calculation

Matrix spike recoveries are calculated as follows:

$$\text{MS or MSD } \%\text{Recovery} = \left(\frac{SSR - SR}{SA} \right) \times 100\% \qquad \textit{Equation 16}$$

Where:

SSR = Measured concentration in spiked sample.
SR = Measured concentration in unspiked sample.
SA = Concentration of spike added to sample.

12.10.5 MS/MSD RPD Calculation

The relative percent difference between the MS and MSD is calculated as follows:

$$\%RPD = \frac{\left| R_1 - R_2 \right|}{\frac{1}{2}\left(R_1 + R_2 \right)} \times 100\% \qquad \textit{Equation 17}$$

Where *R1* is the result for the MS and *R2* is the result for the MSD.

12.11 All data are subject to two levels of review, which is documented on a checklist, as described in SOP DV-QA-0020.

13 Method Performance

13.1 Method Detection Limit Study (MDL)

An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy DV-QA-005P. Each of the other aroclors have an MDLV performed annually to satisfy NELAC 2003 requirement. For DoD, AFCEE, DOE and Texas TRRP projects, AR_1660 MDLVs and LOQVs are performed quarterly. MDLs and LOQs are stored in LIMS.

13.2 Demonstration of Capabilities

An initial demonstration of capability for each method must be performed prior to analyzing samples in accordance with DV-QA-0024.

13.2.1 For the standard analyte list, the initial demonstration consists of the preparation and analysis of a QC check sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study (described in Section 12.1).

13.2.2 Four aliquots of the QC check sample are analyzed with the same procedures used to analyze samples, including sample preparation.

13.2.3 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples.

13.2.4 For non-standard analytes an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is successful analysis of an extracted standard at the reporting limit and a single point calibration.

13.3 Training Requirements

13.3.1 The Group/Team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.3.2 Each analyst performing the method must complete an initial demonstration of capability (IDOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

14 Pollution Control

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual (M-E-001 DV) for "Waste Management and Pollution Prevention.

14.2 Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Waste hexane solvent: Flammable Solvent – Waste Stream C

15.2.2 Vials containing extracts in hexane: Expired Extract Vials – Waste Stream A

15.2.3 Concentrated sulfuric acid and hexane from sample cleanup: Concentrated Acids with Organics - Waste Stream V

15.2.4 Expired reagents and standards – Contact Waste Coordinator

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.2.5 Samples containing polychlorinated biphenyls (PCB's) at concentrations ≥50 ppm are regulated under the Toxic Substance Control Act (TSCA) and must be segregated from all other waste streams. Analysts are responsible for contacting the Group Leader, Sample Control, and the Waste Coordinator immediately if a sample falls into the TSCA category.

16 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.1.1 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

16.1.2 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

16.1.3 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

16.1.4 Method 3546, Microwave Extraction, Revision 0, February 2006.

16.1.5 Method 3580A, Waste Dilution, Revision 1, July 1992.

16.1.6 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

16.1.7 Method 3665A, Sulfuric Acid/Permanganate Cleanup, Revision 1, December 1996.

16.1.8 Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, December, 1996.

16.1.9 Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 1, February 2007

16.1.10 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

16.1.11 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

17 Method Modifications

Item	Method	Modification
1	8082	Method 8082 includes an internal standardization option. Because of the high probability of interferences affecting internal standards, this is strictly an external standard SOP.
2	8000B	Method 8000 allows for use of a second order or third order calibration curve. TestAmerica Denver does not allow for any curvilinear calibrations for the analysis of arochlors.

18 Attachments

Table 1: Analyte List and Standard Reporting Limits

Table 2: Typical Instrument Conditions

Table 3: Calibration Levels (µg/mL)

Table 1-LVI: Analyte List and Standard Reporting Limits using Large Volume Injection

Table 2-LVI: Typical Instrument Conditions using Large Volume Injection

Table 3-LVI: Calibration Levels (µg/mL) using Large Volume Injection

Attachment 1: Arochlor Identification 101

19 Revision History

Revision 6, June 15, 2012

- Added Tables 1-LVI, 2-LVI, and 3-LVI for large volume injection

Revision 5.1, January 16, 2012

- Changed extraction holding time for water and solid to 1 year with exclusion for California, Connecticut, Pennsylvania and South Carolina (Section 8).
- Reformatted paragraphs throughout

Revision 5, December 2011

- Combined SOP DV-GC-0021 and DV-GC-0030 Rev. 0.2. Upon implementation of this revision of SOP DV-GC-0021, SOP DV-GC-0030 will be deactivated.

- Added details for analysis of polychlorinated terphenyls by this procedure (sections 1, 3, 7 and Table 1).
- Updated Section 6 to include reference to master list of documents, software and hardware and volumetric flasks.
- Updated refrigerator temperature references from 4 ± 2°C to 0-6°C throughout.
- Updated vendors and catalog numbers for standards (Section 7)
- Updated Section 9 for consistency with SOP DV-QA-003P.
- Added calibration section to describe calibration models.
- Revised Procedure (new section 11) to be consistent with other SOPs revised in the last year.
- Added detail about review process (Section 11.8)
- Revised Calculations section (new section 12) to address dual column quantitation, sample dilution, and recovery calculations.
- Revised section numbers for previous sections 12-18.
- Updated Method Modifications section
- Revised Table 1 and Table 2

Revision 4.1, December 2010

- Added QC criteria for cleanup procedures to section 4.1.
- Added section 11.1 to reference corporate SOP CA-Q-S-005 “Calibration Curves”

Revision 4.0, June 2010

- Annual Technical Review.
- Deleted the centering of the window requirement for “each subsequent 12-hour calibration verification” in Section 9.19.
- Added LOQV information in Section 12.1
- Added Attachment 1

Revision 3.1, June 2009

- Basic Annual Review

Revision 3, April 2008 updated to TestAmerica and reformatted.

- Updated formatting to comply with Policy DV-QA-001P.
- Added Section 1.3 to reference sample preparation SOPs..
- Added references to sample preparation SOPs to Section 2.
- In Section 3.2, added note to explain the existence of anomalous formulations of Aroclors.
- Updated information on standards in Section 7 to reflect current practice.

- Revised Section 9.1 to include reference to Policy DV-QA-024P for QC requirements for federal programs.
- In Section 10, deleted instructions for second-order calibration curves, which are not used for this method.
- Added the RL Standard to Sections 7.6 and 10.7.3.
- Added information for setting up and running specific laboratory instrumentation in section 10.1, 11.5, 11.6, and 11.10.
- Updated data analysis and calculations in Section 12 to reflect current practice.
- Updated Sections 13, 14, and 15 to reflect current practice.
- Expanded the references in Section 16.

Table 1. Analyte List and Standard Reporting Limits

Compound	Water Reporting Limit (µg/L)	Soil Reporting Limit (µg/kg)
Aroclor 1016	1.0	33
Aroclor 1221	1.0	47
Aroclor 1232	1.0	33
Aroclor 1242	1.0	33
Aroclor 1248	1.0	33
Aroclor 1254	1.0	33
Aroclor 1260	1.0	33
Aroclor 1262	1.0	33
Aroclor 1268	1.0	33
PCT 5432	0.5	50
PCT 5442	0.5	75
PCT 5460	0.5	50

Table 2. Typical Instrument Conditions

Parameter	Recommended Conditions
Injection Port Temperature:	250 °C
Detector Temperature:	325 °C
Temperature Program:	Instrument W 125 °C for 1 minute 8 °C/min to 275 °C for 0.1 minute 30 °C/min to 310 °C for 2 minutes Instrument P3 125 °C for 1.25 minutes 30 °C/min to 180 °C 12 °C/min to 280 °C 15 °C/min to 320 °C for 2.6 minutes
Column 1:	CLPI, 30 m x 0.32 mm id, 0.5 µm
Column 2:	CLPII, 30 m x 0.32 mm id, 0.25 µm
Injection:	1 or 2 µL
Carrier Gas:	Hydrogen
Make-up Gas:	Nitrogen
Y-splitter:	Restek or J&W or Supelco glass tee, single gooseneck liner

Table 3. Calibration Levels (µg/mL)

Aroclors	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Aroclor 1016	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1221	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1232	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1242	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1248	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1254	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1260	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1262	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1268	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Surrogates are included in the AR_1660 calibration mix at the following levels:							
Tetrachloro-m-xylene	0.00125	0.0025	0.005	0.0125	0.025	0.0375	0.05
Decachlorobiphenyl	0.00125	0.0025	0.005	0.0125	0.025	0.0375	0.05

Table 1-LVI. Analyte List and Standard Reporting Limits for Large Volume Injection

Compound	Water Reporting Limit (µg/L)	Soil Reporting Limit (µg/kg)
Aroclor 1016	0.5	5.0
Aroclor 1221	0.5	5.0
Aroclor 1232	0.5	5.0
Aroclor 1242	0.5	5.0
Aroclor 1248	0.5	5.0
Aroclor 1254	0.5	5.0
Aroclor 1260	0.5	5.0
Aroclor 1262	0.5	5.0
Aroclor 1268	0.5	5.0
PCT 5432	NA	NA
PCT 5442	NA	NA
PCT 5460	NA	NA

Table 2-LVI. Typical Instrument Conditions for Large Volume Injection

Parameter	Recommended Conditions
Injection Port Temperature:	250 °C
Detector Temperature:	325 °C
Temperature Program:	Instrument P3 125 °C for 1.25 minutes 30 °C/min to 180 °C 12 °C/min to 280 °C 15 °C/min to 320 °C for 2.6 minutes
Column 1:	Restek Rtx-CLPesticides 30m X 0.32 mm id, 0.5 µm (Cat# 11139 or equivalent)
Column 2:	Restek Rtx-CLPesticides2 30m X 0.32 mm id, 0.5 µm(Cat# 11324 or equivalent)
Injection Volume:	10 µL (Agilent 25 µL G4513-80241 or equivalent)
Carrier Gas:	Hydrogen
Make-up Gas:	Nitrogen
Y-splitter:	Restek Universal Presstight Connector (Cat# 20400 or equivalent)
Injection Port Liner:	Agilent 5190-2293 90011 or equivalent

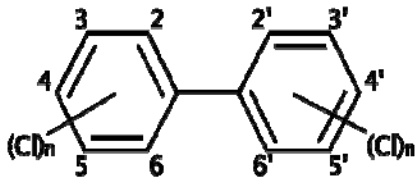
Table 3-LVI. Calibration Levels (µg/mL) for Large Volume Injection

Aroclors	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Aroclor 1016	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1221	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1232	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1242	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1248	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1254	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1260	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1262	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1268	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Surrogates are included in the AR_1660 calibration mix at the following levels:							
Tetrachloro-m-xylene	0.000125	0.00025	0.0005	0.00125	0.0025	0.00375	0.005
Decachlorobiphenyl	0.000125	0.00025	0.0005	0.00125	0.0025	0.00375	0.005

Attachment 1. Arochlor Identification 101

Aroclor identification 101

It can be difficult to correctly identify which Aroclor is present in a sample. This document provides a few guidelines. We are calling this document Aroclor identification 101 not because it is simple, but because Aroclor identification 201 and 301 (mixed, weathered Aroclors) are much more difficult still (sort of like P-Chem!)



First, we should consider what Aroclors actually are: They are mixtures of polychlorinated biphenyls.

Each phenyl ring can accommodate between zero and 5 chlorines. There are 209 possible isomers with 1-10 chlorines (the surrogate decachlorobiphenyl is the fully chlorinated molecule). Of these, about 130 are present in various Aroclor mixes, accounting for the complexity of the chromatograms. The first two digits of the Aroclor number refers to the number of carbon atoms, the last two refer to the degree of chlorination. Thus Aroclor 1248 has 12 carbon atoms in each molecule, and 48% chlorine by mass. So, as the last two digits increase, the overall degree of chlorination increases, the volatility decreases, and the pattern of peaks moves later in the chromatogram. Arochlor 1248 consists of approximately 1% monochlorobiphenyl, 13% dichlorobiphenyl, 45% trichlorobiphenyl, 31% tetrachlorobiphenyl and 10% pentachlorobiphenyl.

It is estimated that 1.25 billion pounds of PCBs were produced until Monsanto ceased production in 1977. PCBs are very persistent, so much of this material is still present in the environment.

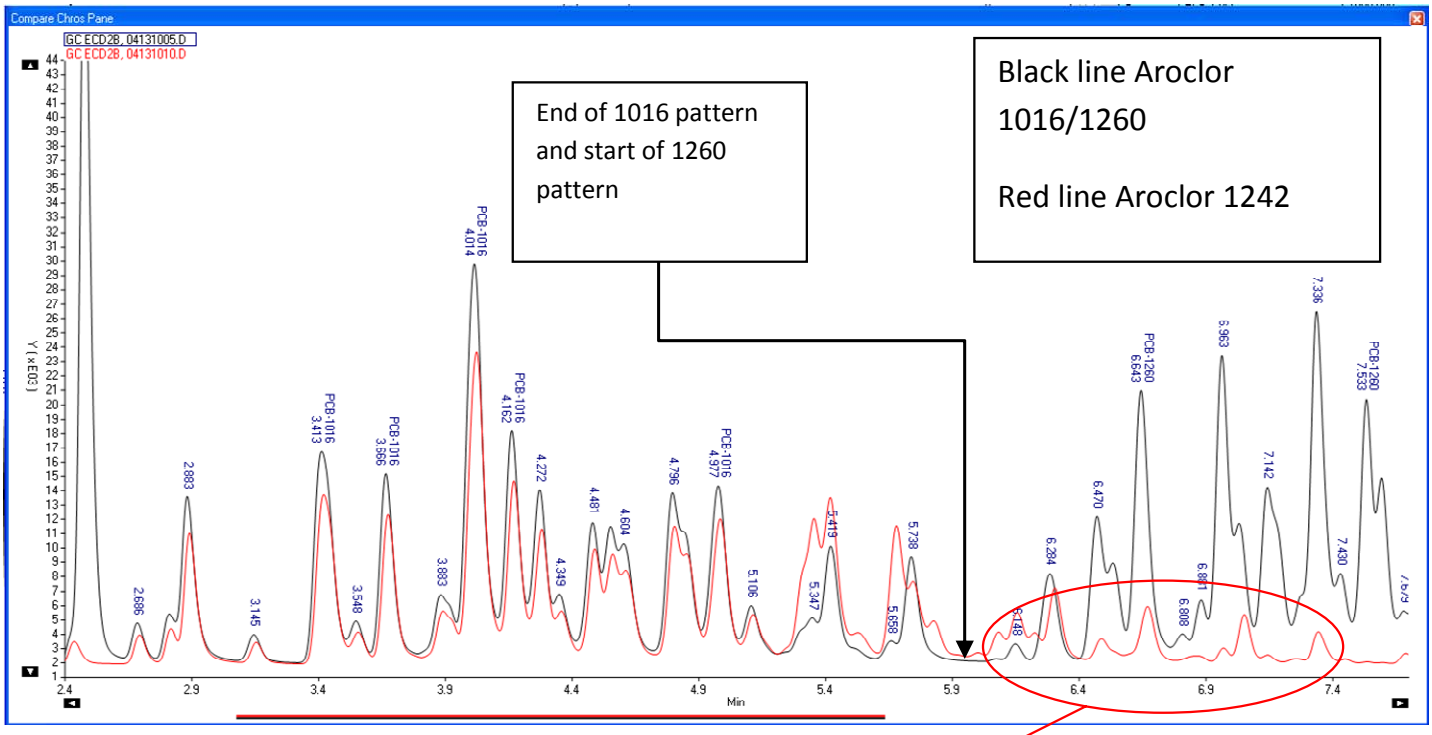
Example Chromatograms

In the following examples I'll refer to retention times frequently – your retention times will of course be different because of different chromatographic conditions but the same principles apply.

Attachment 1. Arochlor Identification 101 (Continued)

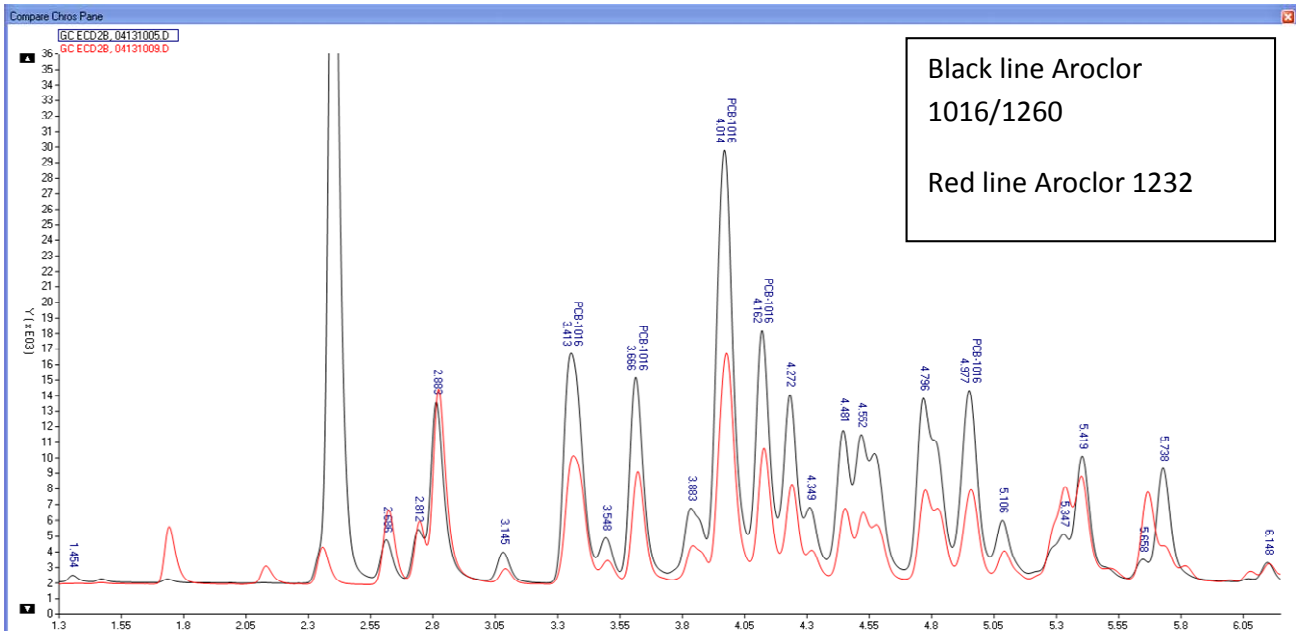
The first example considers Aroclor 1016 vs. 1242. Note that the early part of the chromatograms (2.5 – 5.3 minutes) are virtually identical. The key difference is the presence of some later eluting peaks (6.2-7.3 minutes in this chromatogram) in 1242 that are not present in 1016. This difference is masked by the fact that Aroclor 1260 is also present in this standard. Most labs analyze standards of 1016 and 1260 together – there is nothing wrong with this but it is a good idea to periodically (one run with each initial calibration?) analyze them separately so that you have a good idea of the two separate patterns.

The story of 1016 is interesting – in the early 1970's PCBs were starting to be found in fish in the Great Lakes. The more heavily chlorinated biphenyls were bioaccumulating more and were of greatest concern. So, Monsanto attempted to modify the manufacturing process to reduce the amount of pentachlorobiphenyls in Aroclor 1242, while still keeping the overall degree of chlorination similar. They were successful in this regard – Aroclor 1242 has about 10% pentachlorobiphenyls which show up between 6 and 7.4 minutes in the chromatogram below. Aroclor 1016 has 42% by weight chlorine (it does not follow the standard naming convention) but has no pentachlorobiphenyls.

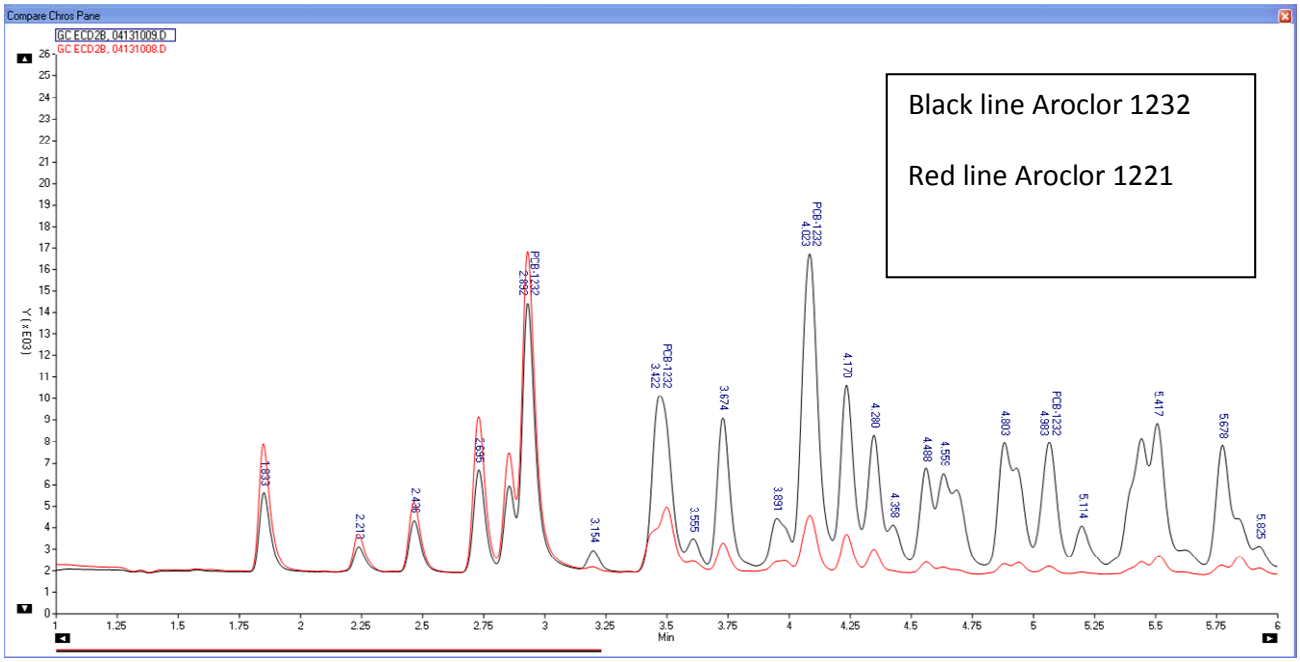


Attachment 1. Arochlor Identification 101 (Continued)

Here is 1016 vs. 1232. These are even more similar (the large peak at around 2.35 min is TCMX) but note the very early peaks present in 1232 and not in 1016, and also note that the front end is stronger in 1232 for example in 1232 the peak at 2.88 min is about the same size as those at 4.79 and 4.97, whereas in 1016 the later peaks are twice as large.

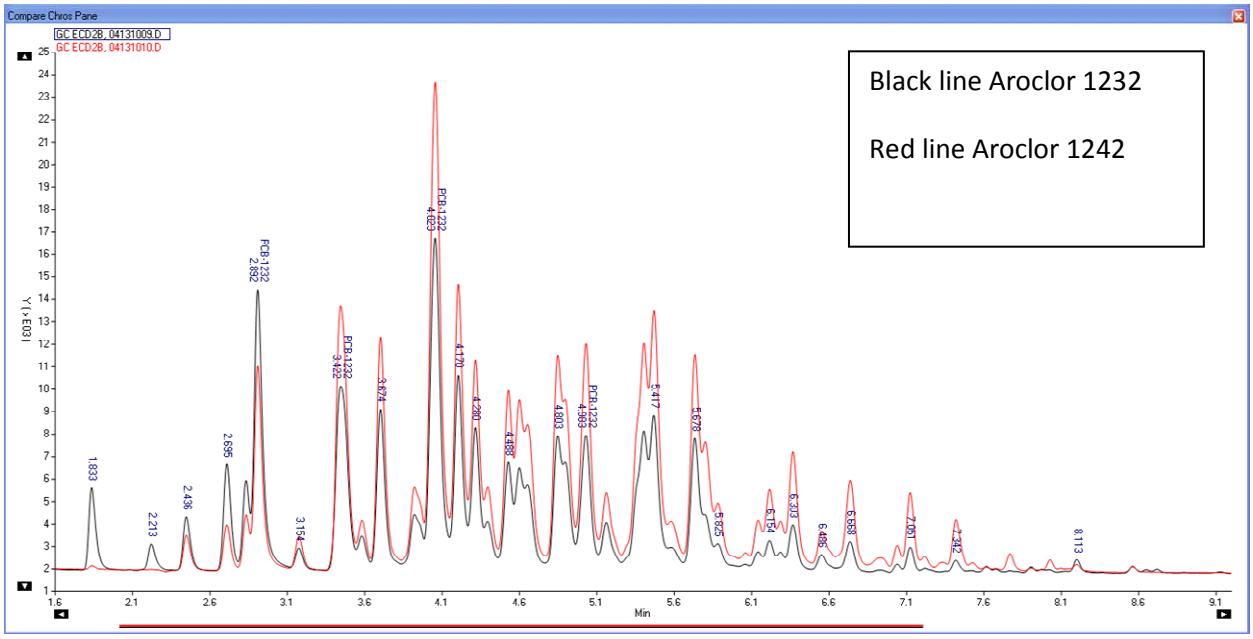


For 1221 vs. 1232, the front end of the chromatogram is identical, but 1232 has later peaks that are not present in 1221.

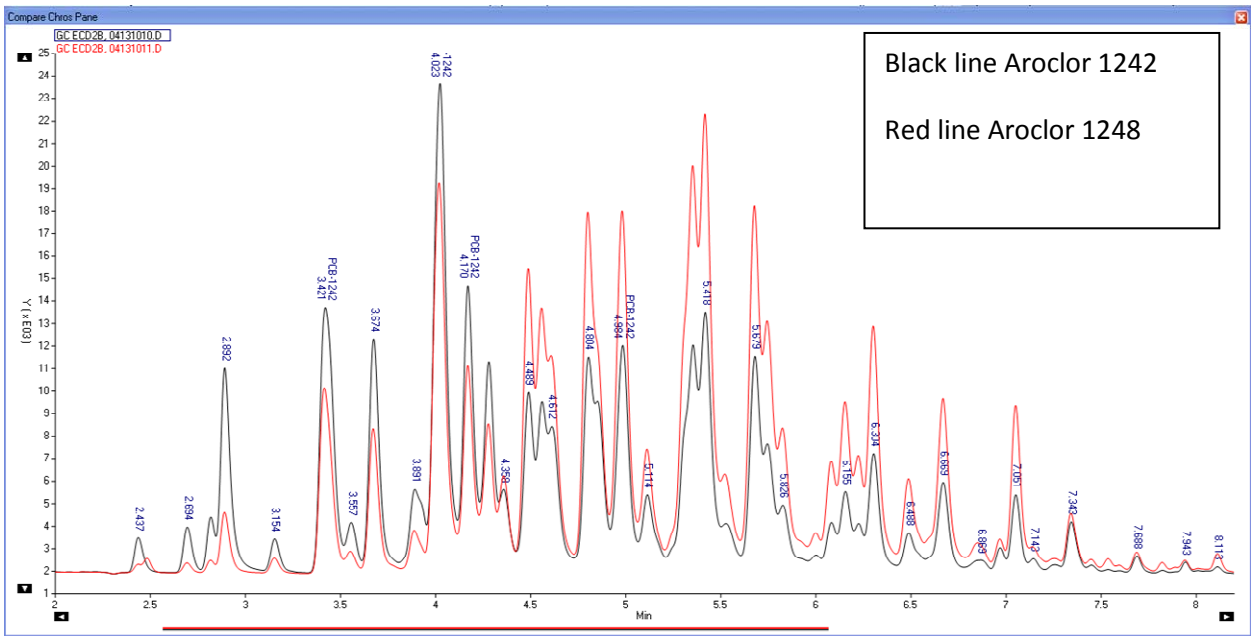


Attachment 1. Arochlor Identification 101 (Continued)

1232 and 1242 are best distinguished by the early peaks in 1232 (1.83, 2.13) that are not present in 1242. Also note that the peak at 2.89 is twice the height of that at 5.41 in 1232, whereas the 5.41 peak is slightly higher in 1242. This relative size of the front and back end of the envelope is a key tool for distinguishing Aroclors.

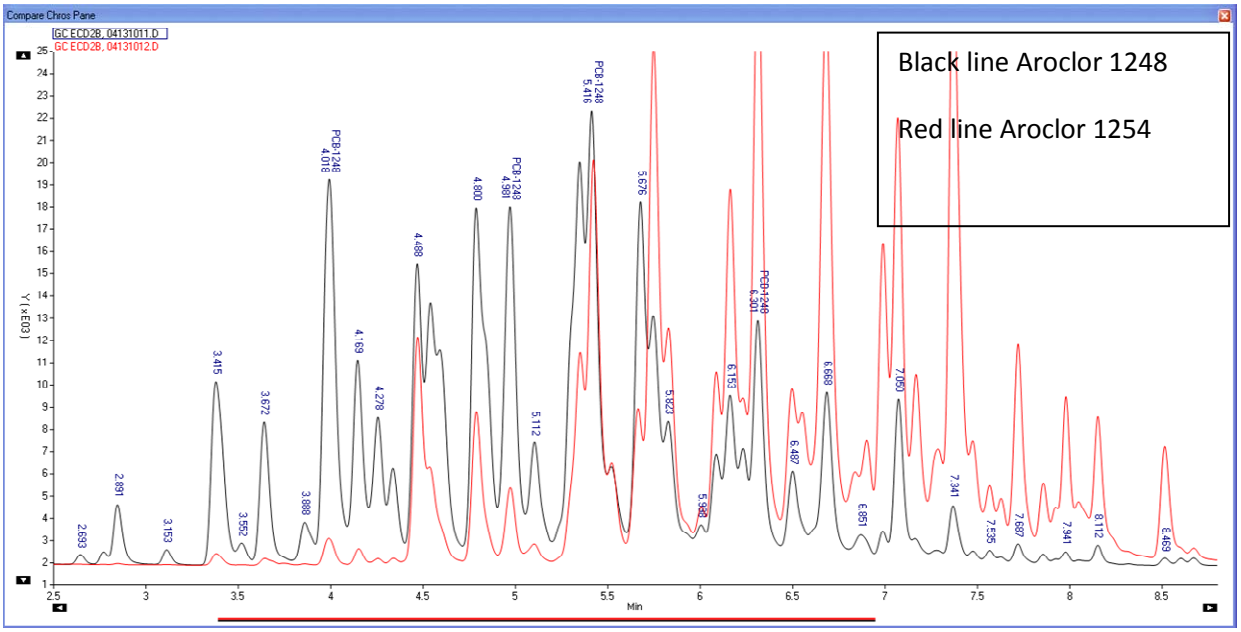


Aroclors 1242 and 1248 both have all of the same peaks, so the relative strength of the front and back of the envelope is the only way to distinguish. For example, in 1242, the peak at 3.42 is larger than that at 5.67, whereas for 1248, the 5.67 peak is considerably larger than the 3.42 peak.

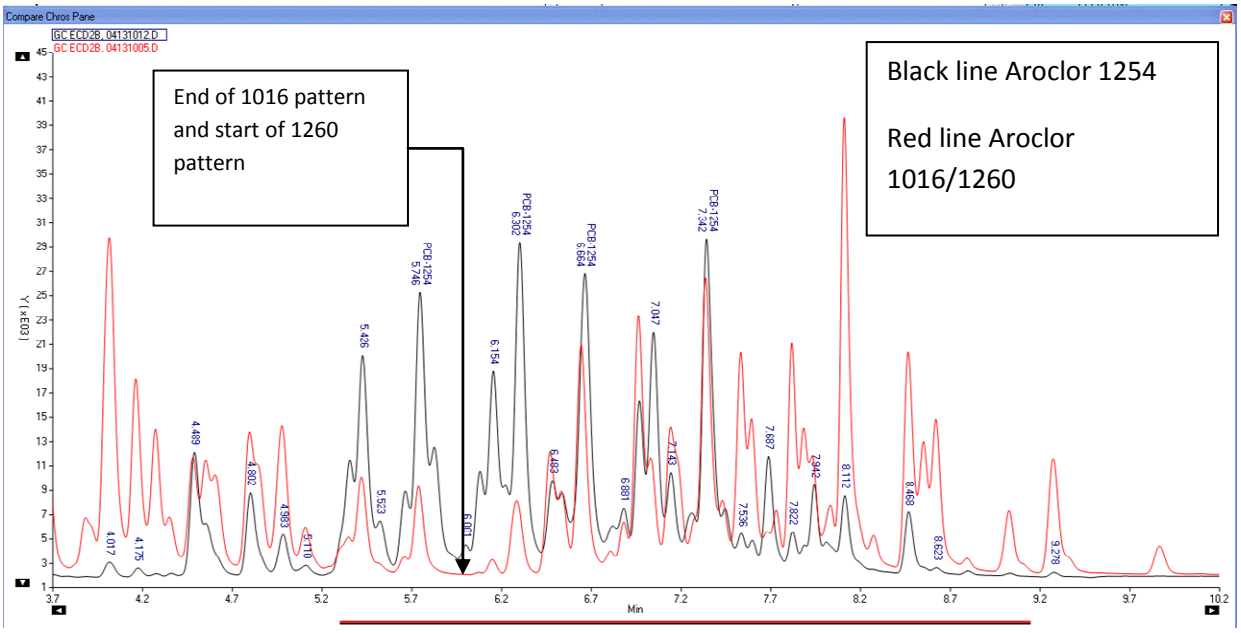


Attachment 1. Arochlor Identification 101 (Continued)

1248 vs 1254 is a relatively easy case, the front end of the envelope is much stronger in 1248.

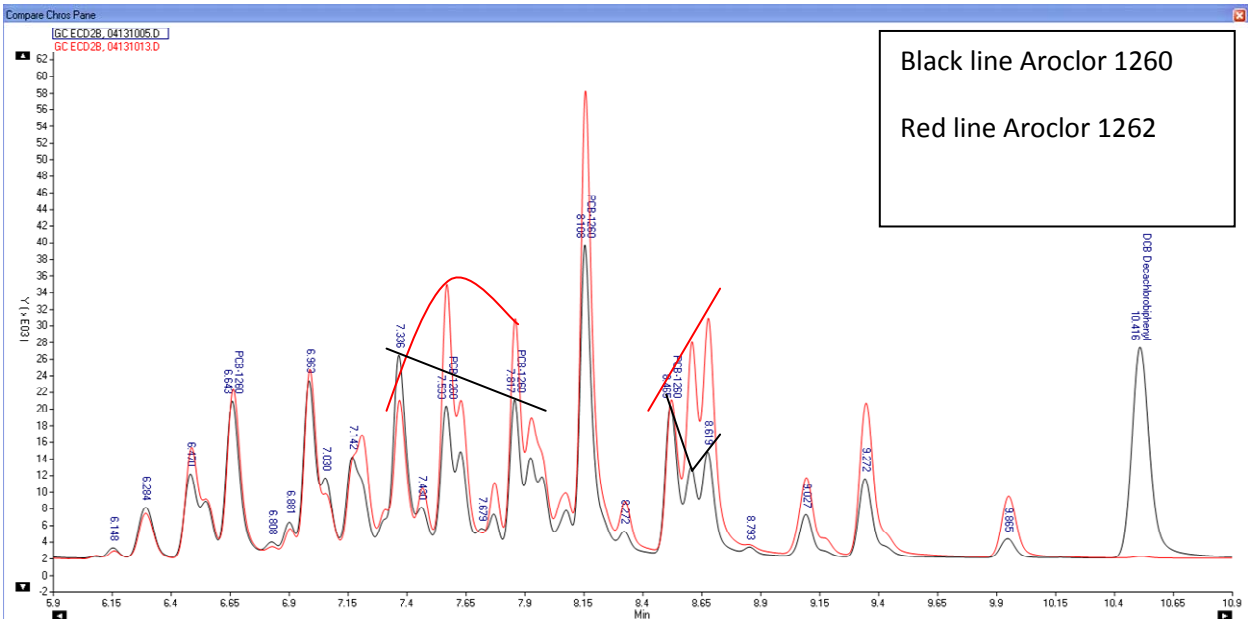


The 1254 vs. 1260 chromatograms are again a little masked by the inclusion of 1016 in the 1260 standard (peaks up to 5.9 min in the 1260 chromatogram actually belong to 1016). Keeping this in mind, the presence of peaks at 5.42 and 5.74 indicates 1254. The relative strength of peaks in the 7.5-9.3 range indicates 1260.

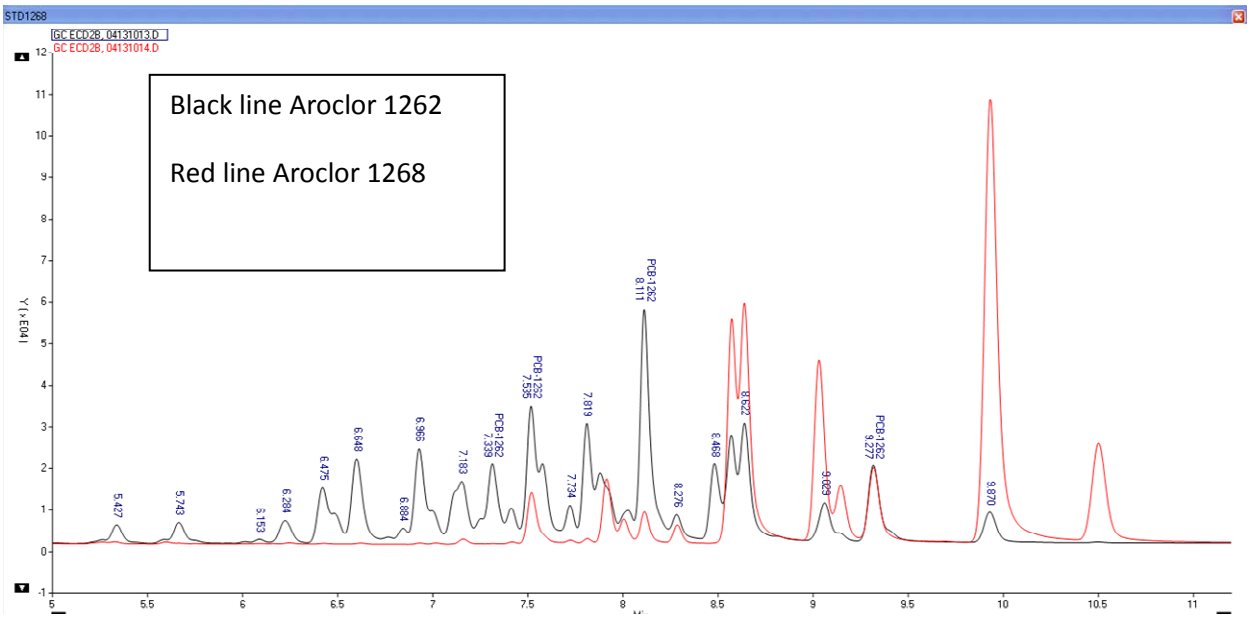


Attachment 1. Arochlor Identification 101 (Continued)

Many labs do not analyze for 1262, perhaps just as well since it is certainly challenging to distinguish from 1260. However, the shape of the envelope is again the key. Note that in 1262 the peaks around 8.6 minutes are as large as that at 6.96, whereas they are only half the size in 1260. Also note the shape of the envelope for the peaks in the 7-8 minute range – bow shaped for 1262 and a straight declining line for 1260. The envelope shape is also quite different in the 8.4-8.7minute range.

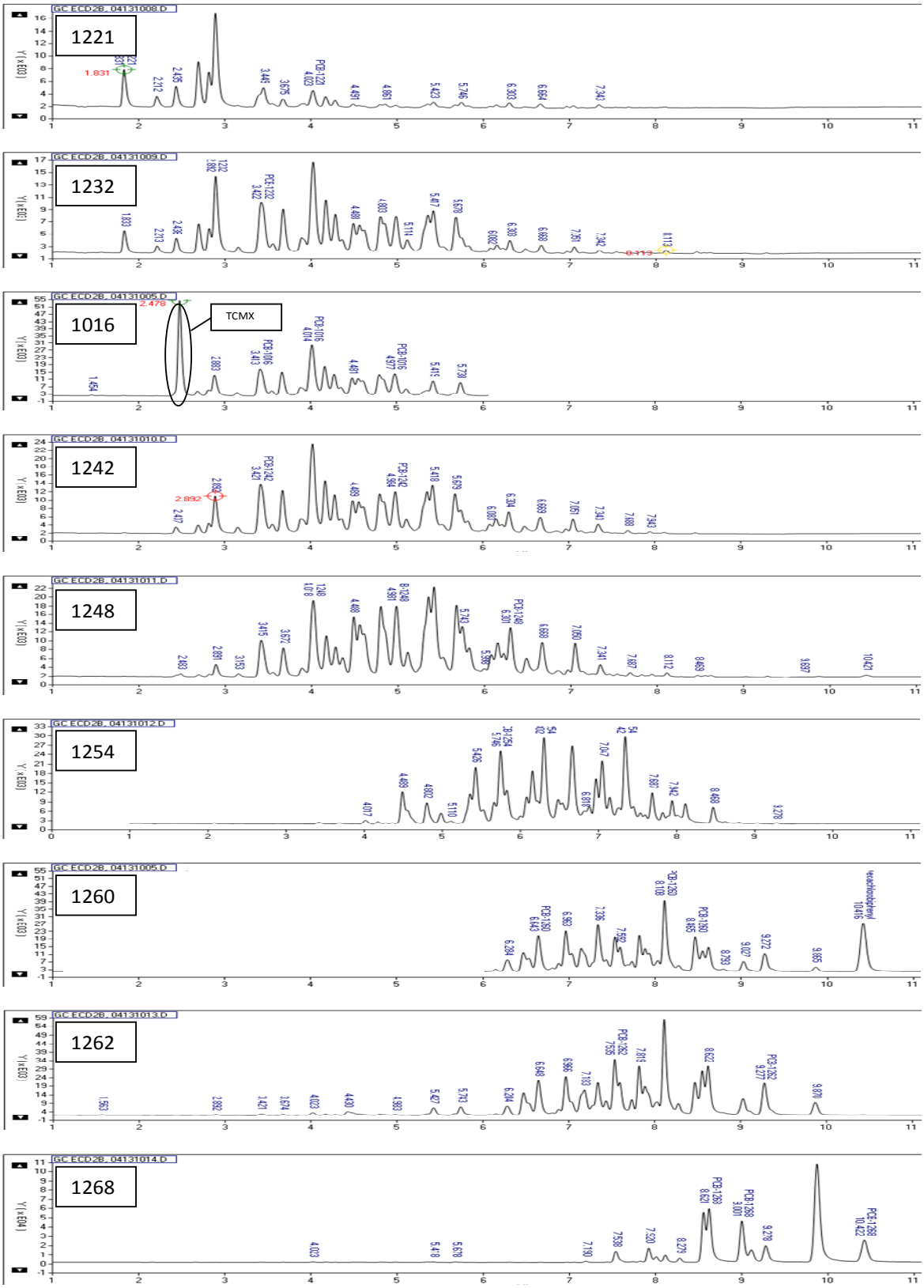


The really strong peak at 9.87 and the lack of much of a pattern between 6.0 and 7.5 minutes are good indicators of 1268.



Attachment 1. Arochlor Identification 101 (Continued)

AROCLOR PATTERNS



Title: Chlorinated Pesticides [Method No. 8081B]

Approvals (Signature/Date):

Dennis Jonsrud — Date: 1-20-10
GC Manager

Adam Alban — Date: 21Jan10
Health & Safety Manager / Coordinator

Karen Kuoppala — Date: 1-21-10
Quality Assurance Manager

Robert C. Hanisch — Date: 1/22/10
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the determination of chlorinated pesticides using the methodology described in EPA SW-846 Method 8081B.

1.2 This SOP is applicable to the gas chromatographic (GC) analysis of extracts of soil and water samples. Table 1 lists the compounds that can be determined by this method and their associated routine reporting limits (RLs).

1.3 This SOP does not include the procedures for extracting soil and water samples. Refer to the following SOPs for sample extraction procedures:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C
DV-OP-0007	Concentration of Organic Extracts, SW846 3510C, 3520C, 3540C, and 3550C
DV-OP-0016	Ultrasonic Extraction of Solid Samples by SW846 3550C
DV-OP-0015	Microwave Extraction of Solid Samples by SW846 3546

1.4 Analytes, Matrix(s), and Reporting Limits

See Table 1 for analytes and reporting limits by matrix.

2.0 Summary of Method

2.1 Sample Preparation

2.1.1 Chlorinated pesticides are extracted from a one-liter water sample with methylene chloride using a separatory funnel (Method 3510C). Detailed instructions are given in SOP DV-OP-0006. The methylene chloride extract is exchanged to hexane as described in SOP DV-OP-0007.

2.1.2 Chlorinated pesticides are extracted from a 30-gram soil subsample into a 50:50 acetone-methylene chloride solution by sonication (Method 3550C) or by microwave extraction (Method 3546). The extract is dried and exchanged to hexane. Detailed instructions are given in SOPs DV-OP-0016 and DV-OP-0015.

2.1.3 SOP DV-OP-0007 provides instructions for the concentration and cleanup of sample extracts. Florisil is used to clean extracts that show color. Sulfur is removed if observed. All extracts are in hexane and the final extract volume is 10 mL.

2.2 Analysis

2.2.1 Samples are analyzed using a gas chromatograph equipped with dual columns and dual electron capture detectors (ECDs).

2.2.2 The instrument is calibrated using external standards. Compounds are identified by their retention time on the columns.

2.2.3 Positive results from the primary column are confirmed with a second, dissimilar column. The laboratory maintains a total of four dissimilar columns for additional confirmation capability.

3.0 Definitions

3.1 Single-Component Pesticides: A pesticide formulation that consists of a single chemical compound. Most of the analytes determined by this procedure are single-compound pesticides.

3.2 Multi-Component Pesticides: A pesticide formulation that consists of more than one chemical compound. Toxaphene and Technical Chlordane are production mixtures of multiple compounds. Toxaphene is manufactured by the chlorination of camphenes, which produces a variety of compounds, not all of which are chromatographically resolved. Technical Chlordane is produced by the chlorination of a mixture of camphenes and pinenes.

3.3 Chlordane: As just described, Technical Chlordane (CAS# 12789-03-6) is a mixture of compounds. Method 8081B, Section 11.6.2 notes that it includes at least 11 major components and 30 minor components, and adds "the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch." The laboratory has found that manufacturing lots of Technical Chlordane produced at different times or at different production facilities have different ratios of the key components. For this reason, it is more common to analyze for the major components of technical Chlordane (α-Chlordane, γ-Chlordane, and heptachlor) instead of analyzing for the total mixture. For the purpose of reporting results under this SOP, the following compounds are reported. Alpha-chlordane (cis-chlordane) CAS # 5103-71-9 and gamma-chlordane (trans-chlordane) CAS # 5103-74-2. The laboratory may also report chlordane (not otherwise specified) or, n.o.s under CAS# 57-74-9.

3.4 The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Denver Quality Assurance Manual (QAM).

4.0 Interferences

4.1 Contamination by carryover can occur when a low concentration sample is analyzed immediately following a high concentration sample. It is the laboratory's policy to reanalyze any samples that follow an unusually concentrated sample and that show detectable levels of the same compounds that appeared in the preceding concentrated sample.

4.2 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3 Sulfur will interfere, and, when observed, is removed using cleanup procedures described in SOP DV-OP-0007.

4.4 Soil and water sample extracts are subject to Florisil cleanup when the extracts have noticeable color or whenever there is clear evidence of interferences in the initial sample chromatograms. Florisil removes low- to medium-molecular weight polar organic interferences from sample extracts. One limitation for this cleanup method is that recoveries for the most polar compounds, endosulfan sulfate and endrin aldehyde in particular, will be lower. Florisil has been observed to remove the compound kepone and is not used where the determination of kepone is required. Instructions for performing Florisil cleanups can be found in SOP DV-OP-0007.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.1.4 The ECD contains a ^{63}Ni radioactive source. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the facility's radioactive material license. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Radiation Safety Officer shall be immediately notified and a letter sent to the Colorado Department of Public Health and Environment.

5.1.5 As a safety precaution, all standards, samples, and extracts are handled in an approved fume hood.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the

method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit [1]	Signs and Symptoms of Exposure
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects are exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 An analytical system complete with a gas chromatograph and dual ECD (Ni-63) detectors is required. A data system capable of measuring peak area and/or height is required.

6.2 An analytical balance capable of weighing to 0.0100g.

6.3 Computer Software and Hardware

6.3.1 Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

6.4 Columns

6.4.1 Primary Column: CLPI, 30 m X 0.32 mm id

6.4.2 Secondary Column: CLPII, 30 m X 0.32 mm id

6.4.3 Additional columns that can be used for confirmation include 30 m X 0.32 mm id DB35-MS or DB-XLB.

6.5 Autosampler vials, crimp-top cap with PTFE-faced septa

6.6 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7.0 Reagents and Standards

Reagents

7.1 Hexane, pesticide grade; each lot tested for purity prior to use per SOP CA-Q-S-001 DV-1.

7.2 Carrier gas, ≥ 99.99999% pure hydrogen or helium

7.3 Make-up gas, ≥ 99.99980% pure nitrogen

Standards

7.4 Standards Verification

All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

7.5 Storage of Stock Standards

7.5.1 Commercial standards are received in flame-sealed ampoules or neat, 100% concentration, solutions. Stock standards are stored refrigerated at ≤ 6 °C. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

7.5.2 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem. Kepone in particular may demonstrate signs of degradation faster than the other compounds, and/or the expiration date. Endosulfan I and II appear to degrade in the presence of methanol. Gamma-BHC appears to degrade in the presence of acetone.

7.6 Calibration Stock Standards

All calibration stock standards are obtained from commercial sources.

NOTE: The availability of the specific commercial standard solutions upon which the following sections are based may change at any time. As a result, it may be necessary to alter the dilution scheme presented herein to accommodate changes in stock standard concentrations. All such changes are documented in the standards preparation records.

7.6.1 Routine Pesticide AB Mix Stock Standard, 1,000 µg/mL

The routine pesticide AB mix stock standard contains all of the "routine" single-component pesticides, as identified in Table 1 with the addition of Hexachlorobenzene at 100 µg/mL Accustandard APP-9-112 and Mirex at 100 µg/mL Accustandard P-066S.

7.6.2 Surrogate B Mix Stock Standard, 200 µg/mL

The surrogate B mix stock standard contains decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX).

7.6.3 Toxaphene Stock, 100 µg/mL

The Toxaphene stock standard contains a specific production mixture of Toxaphene. This mixture does not necessarily match all possible production mixtures that could be found in the environment. This can present problems for Toxaphene quantitation (see Section 12).

7.6.4 Chlordane Stock, 100 µg/mL

The Chlordane stock contains Technical Chlordane (CAS# 12789-03-6).

7.6.5 Appendix IX Calibration Stock

The Appendix IX stock calibration mixture contains the compounds at the concentrations listed in the following table.

Appendix IX Calibration Stock Standard

Compound	Concentration (µg/mL)
2,4-DDD	100
2,4-DDE	100
2,4-DDT	100
Chlorobenzilate	1,000
Chlorpyrifos	500
Diallate	10,000
Dicofol	1,000
Isodrin	500
Kepone	1,000
Mirex	500

7.6.6 Non-Routine Compounds

Other, non-routine compounds not listed in this section may be requested by a client and may be added to this procedure.

7.6.6.1 In these cases, all stock solutions will be obtained from commercial sources and will be verified with a second-source standard as described in Section 7.4 above.

7.6.6.2 Non-routine standards will be stored and treated as described in Section 7.5 above or as specified by the manufacturer.

7.6.6.3 Subsequent dilutions of specially requested compounds will be determined in a manner consistent with the client's recommendations for number of calibration points, inclusion of reporting limit, and concentration range adequate to represent the linearity of the instrument.

7.6.6.4 These specially requested, non-routine compounds either may be added to the dilution scheme used for routine compounds or may be prepared as a separate calibration.

7.6.6.5 All standards preparation for non-routine compounds shall be documented using the same method that is used for routine compounds.

7.7 Intermediate Level Calibration Standards

7.7.1 Routine Pesticide Mix C Intermediate Calibration Standard, 1.0 µg/mLThe intermediate level calibration standard for routine pesticide compounds

including Hexachlorobenzene and Mirex is prepared by diluting the AB (Section 7.6.1) and B (Section 7.6.2) mix stock standards in hexane as follows (all compounds are the same final concentration):

Mix C Intermediate Calibration Standard

Stock AB (mL)	Stock B (mL)	Hexane (mL)	Final Concentration of Each Pesticide (µg/mL)
0.1	0.5	99.4	1.0

7.7.2 Appendix IX Intermediate Calibration Standard

The Appendix IX intermediate level calibration standard is prepared by diluting 0.5 mL of the Appendix IX stock standard (Section 7.6.5) with hexane to a final volume of 50 mL, which results in the following concentrations:

Appendix IX Intermediate Calibration Standard

Compound	Concentration (µg/mL)
2,4-DDD	1.0
2,4-DDE	1.0
2,4-DDT	1.0
Chlorobenzilate	10.
Chlorpyrifos	5.0
Diallate	100.
Dicofol	10.
Isodrin	5.0
Kepone	10.

7.8 Working Level Calibration Standards

7.8.1 Routine Pesticide AB Mix Working Level Calibration Standards

The following volumes of the 1.0 µg/mL Mix C intermediate standard (Section 7.7.1) are diluted to 100 mL with hexane to produce calibration standards at 6 concentration levels, as summarized in the following table:

AB Mix Working Level Calibration Standards

Level	Volume of Mix C Intermediate Std (mL)	Final Concentration (µg/mL)
1	0.4	0.0040
2	1.0	0.010
3	2.5	0.025
4 *	5.0	0.050
5	7.5	0.075
6	10	0.10

* *This level is used as the Continuing Calibration Verification (CCV) standard. As a result, it may be convenient to make a larger volume of this calibration level, by diluting 12.5 mL of the intermediate standard with hexane to a final volume of 250 mL.*

7.8.2 Toxaphene Working Level Calibration Standards

The following volumes of the 100 µg/mL Toxaphene stock standard (Section 7.6.3) are diluted with hexane to the final volumes indicated in the following table:

Toxaphene Working Level Calibration Standards

Level	Volume of Stock Std (mL)	Final Volume (mL)	Final Concentration (µg/mL)
1	0.010	5.0	0.20
2	0.025	5.0	0.50
3	0.05	5.0	1.0
4 *	0.2	10.0	2.0
5	0.25	5.0	5.0
6	0.5	5.0	10.0

* *This level is used as the CCV standard. To make additional volume of this standard, dilute 0.5 mL of the stock with hexane to a final volume of 250 mL.*

7.8.3 Chlordane Working Level Calibration Standards

The following volumes of the 100 µg/mL Chlordane stock standard (Section 7.6.4) are diluted with hexane to the final volume indicated in the following table:

Chlordane Working Level Calibration Standards

Level	Volume of Stock Std (mL)	Final Volume (mL)	Final Concentration (µg/mL)
1	0.005	10.0	0.05
2	0.02	10.0	0.20
3	0.05	10.0	0.50
4*	0.10	10.0	1.0
5	0.20	10.0	2.0

* *This level is used as the CCV standard. To make additional volume of this level, dilute 0.5 mL of the stock standard with hexane to a final volume of 250 mL.*

7.8.4 Appendix IX Working Level Calibration Standards

The following volumes of the Appendix IX intermediate standard (Section 0) are diluted with hexane to a final volume of 1.0 mL. The following table summarizes the final compound concentration ranges for each calibration level. The concentration for each compound at each level is given in Table 3.

Appendix IX Working Level Calibration Standards

Level	Volume of Intermediate Std (mL)	Final Compound Concentration Range (µg/mL)
1	0.005	0.005 - 0.50
2	0.010	0.01 - 1.0
3	0.025	0.025 - 2.5
4 *	0.035	0.05 - 5.0
5	0.050	0.035 - 3.5
6	0.100	0.1 - 10
* *This level is used as the CCV. Because some compounds in this standard are not stable, it is not recommended to make extra volume of the level 4 standard.*		

7.9 Second-Source Standards for Initial Calibration Verification (ICV)

The second-source stock standards are purchased from a vendor different from the one that supplied the stock calibration standards

7.9.1 Routine Pesticide AB Mix ICV Stock Standard, 1,000 µg/mL, (with Mirex at 100 µg/mL)

Commercial standards containing all single-component pesticide compounds are obtained from a vendor different from the one that supplied the calibration stock standard. Typically, the standards are obtained from Ultra Scientific (standard PPM-808C for the AB mix, standard EPA-1125 for Hexachlorobenzene, and standard PST-720S for Mirex).

7.9.2 Appendix IX ICV Stock Standard

Commercial standards are obtained at the same concentrations as shown for the calibration stock standards in Section 7.6.5, but from a different vendor (typically Ultra Scientific standard CUS-7007).

7.9.3 Surrogate ICV Stock Standards, 200 µg/mL

Commercial standards (typically Ultra Scientific standard ISM-320) are obtained containing decachlorobiphenyl (DCBP) and tetrachloro-m-xylene (TCMX).

7.9.4 ICV Intermediate Level Standards, 1.0 µg/mL

The intermediate level calibration standard for routine pesticide compounds is prepared by diluting the AB, Hexachlorobenzene, and Mirex, and surrogate stock standards (Sections 7.9.1 and 7.9.3) with hexane to a final volume of 25 mL as summarized in the table below. All compounds in the intermediate standard are at the same final concentration, i.e., 1.0 µg/mL.

Second-Source ICV Intermediate Standard

Vol of AB Stock (mL)	Vol of Mirex Stock (mL)	Vol of Surrogate Stock (mL)	Final Volume (mL)	Final Conc (µg/mL)
0.025	0.25	0.125	25.0	1.0

7.9.5 Routine Pesticide ICV Working Level Standard, 0.025 µg/mL

The working level ICV standard for the routine pesticide compounds is prepared by diluting the ICV intermediate standard (Section 7.9.4) in hexane follows:

Routine Pesticide Second-Source ICV Working Level Standard

Volume of Intermediate Standard (mL)	Final Volume (mL)	Final Concentration (µg/mL)
2.5	100	0.025

7.9.6 Appendix IX ICV Working Level Standard

The working level ICV standard for the Appendix IX compounds is prepared by diluting 0.025 mL of the second-source Appendix IX stock standard (Section 7.9.2) with hexane to a final volume of 100 mL. The following table lists the final concentration of each pesticide:

Appendix IX ICV Working Level Standard

Pesticide	Final Concentration (µg/mL)
2,4-DDD	0.025
2,4-DDE	0.025
2,4-DDT	0.025
Chlorobenzilate	0.25
Chlorpyrifos	0.125
Diallate	2.5
Dicofol	0.25
Isodrin	0.125
Kepone	0.125

7.10 Continuing Calibration Verification (CCV)Standards

The level 4 AB mix working calibration standard (Section 7.8.1) and the level 4 Appendix IX working calibration standard (Section 0) are used as the CCV standards.

7.11 RL Standard

The lowest concentration calibration standard (i.e., Level 1) is used as the RL standard.

7.12 Laboratory Control Standard (LCS) Spike Solution, 0.5 µg/mL

The working LCS spike solution is prepared by diluting 0.125 mL of the AB mix stock standard (Section 7.6.1) in acetone to a final volume of 250 mL in a volumetric flask, as summarized in the table below.

The LCS for batches of aqueous samples is prepared by adding 1.0 mL of the LCS spike solution to one liter of reagent water. The LCS for batches of soil samples is prepared by adding 1.0 mL of the LCS spiking solution to 30 g of Ottawa sand.

LCS Spiking Solution

Volume of AB Mix Stock (mL)	Conc of AB Mix Stock (µg/mL)	Final Volume (mL)	Final Concentration (µg/mL)
0.125	1000	250	0.5

7.13 Matrix Spike (MS) Spike Solution, 0.5 µg/mL

The working matrix spike solution is the same as the LCS spike solution (Section 7.12). Matrix spikes (MS and MSD) are prepared by adding 1.0 mL of the working spike solution to one liter of an aqueous sample or to a 30-gram soil subsample.

7.14 Toxaphene Spike Solution, 2.0 µg/mL

7.14.1 A Toxaphene stock standard solution at a concentration of 1,000 µg/mL is purchased from commercial sources.

7.14.2 The working Toxaphene spike solution is prepared in a 500 mL volumetric flask by adding 1.0 mL of the stock solution and diluting to volume with acetone.

7.14.3 Aqueous LCSs are prepared by adding 1.0 mL of the Toxaphene spike solution to 1.0 liter of reagent water. Soil LCSs are prepared by adding 1.0 mL of the Toxaphene spike solution to 30 grams of Ottawa sand.

7.14.4 Aqueous MS/MSDs are prepared by adding 1.0 mL of the Toxaphene spike solution to 1.0 liter of the selected aqueous sample. Soil sample MS/MSDs are prepared by adding 1.0 mL of the Toxaphene spike solution to 30 grams of the selected soil subsample.

7.15 Surrogate Spike Solution, 0.2 µg/mL

7.15.1 The surrogate stock solution, containing 200 µg/mL each of decachlorobiphenyl and tetrachloro-m-xylene (TCMX), is purchased form commercial sources.

7.15.2 The working surrogate spike solution is prepared in a 500 mL volumetric flask by adding 0.5 mL of the stock solution and diluting to volume with acetone.

7.15.3 For aqueous sample batches, 1.0 mL of the surrogate spike solution is added to each one-liter sample and QC sample. For soil sample batches, 1.0 mL of the surrogate spike solution is added to each 30-gram soil subsample and QC sample.

7.16 Column Degradation Mix (EVAL B)

7.16.1 The DDT/endrin breakdown stock standard solution is obtained from commercial sources, with endrin at a concentration of 1.0 µg/mL, and 4,4'-DDT at 2.0 µg/mL.

7.16.2 The working EVAL B solution is prepared in a 50 mL volumetric flask, by diluting 1.0 mL of the stock solution in hexane, as summarized in the following table:

Column Degradation Mix (Eval B Std) Spike Solution

Compound	Volume of Stock (mL)	Final Volume (mL)	Final Concentration (µg/mL)
Endrin	1.0	50	0.02
4,4'-DDT			0.04

7.17 Primer Mix

The concentration of the column primer mix is not critical. It generally consists of a mixture of CCV, old ICAL standards, and /or old soil LCS extracts. The primer mix is used to initialize the column and does not affect calibration or quantitation.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples are collected in pre-cleaned, amber glass bottles fitted with a Teflon-lined cap. To achieve routine reporting limits, a full one liter of sample is required. Additional one-liter portions are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes.

8.2 Soil samples are collected in 8-ounce, pre-cleaned, wide-mouth jars with a Teflon-lined lid.

8.3 Samples are stored at 4 ± 2 °C.

8.4 Extracts are refrigerated at ≤ 6 °C.

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	Amber glass	1 Liter	Cool 4 ± 2°C	7 Days	40 Days from extraction	40 CFR Part 136.3
Soils	Glass	30 grams	Cool 4 ± 2°C	14 Days	40 Days from extraction	N/A

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Assurance Program.*

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, *Requirements for Federal Programs.*

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on an instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for batches of aqueous samples consists of 1.0 liter of reagent water, and for batches of soil samples, consists of 30 grams of Ottawa sand, both of which are free of any of the analyte(s) of interest. The method blank is processed and analyzed just as if it were a field sample.

Acceptance Criteria: The result for the method blank must be less than the reporting

limit for the analyte(s) of interest or less than 10% of the analyte concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: If target analytes in the blank exceed the acceptance limits, the source of the contamination must be investigated. All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If the analyte was not detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.5 Laboratory Control Sample (LCS)

At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of reagent water to which the analyte(s) of interest are added at a known concentration. For soil sample batches, the LCS consists of reagent sand to which the analyte(s) of interest are added at a known concentration. See Section 7.12 for the preparation of LCSs. The LCS is carried through the entire analytical procedure just as if it were a sample.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must

be identified and corrective action taken.

Note: Some programs (e.g., South Carolina) do not allow marginal exceedances. Please see the QSAS's in the public folders for the current requirements.

Corrective Action: If LCS recoveries are outside of the established control limits, and the MS/MSD recoveries are also out of control limits then the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Refer to Section 7.13 for preparation of matrix spikes. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

Acceptance Criteria: The recovery results for the MS and MSD must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean. The relative percent difference (RPD) between the MS and MSD must be less than the established RPD limit, which is set at 3 standard deviations above the historical mean. Current control limits are maintained in the LIMS.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e., method blank, LCS, LCSD, MS, and MSD) is spiked with DCB and TCMX surrogate compounds. Refer to Section 7.15 for preparation of the surrogate spike solution.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at ± 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and

acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

For field samples, surrogate recoveries are usually calculated and reported for DCB only. TCMX may also be added, however if two surrogate compounds are analyzed and recoveries calculated, and either surrogate fails to meet acceptance criteria, corrective actions are required. (This also applies to programs that require the use of only one surrogate.)

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control.

10.0 Calibration and Standardization

10.1 TestAmerica Denver gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

10.1.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General).*

10.1.2 Use the ChemStation chromatography data system to set up GC conditions for calibration. See Table 2 for typical operating conditions.

10.1.3 Transfer calibration standard solutions into autosampler vials and load into the GC autosampler. Use the ChemStation software to set up the analytical sequence.

10.1.4 Unprocessed calibration data are transferred to the TARGET DB database for processing. After processing the calibration data, print the calibration report and review it using the calibration review checklist (GC and HPLC Data Review Checklist - ICAL). Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration reports are scanned and stored as Adobe Acrobat files on the Public Drive.

10.2 Column Degradation Evaluation

10.2.1 Each day of before operation any calibration or calibration verification standards are analyzed, the column degradation evaluation mix (EVAL B) must be analyzed. In addition, some programs require injection of the degradation evaluation mix more frequently. The degradation check must be performed

whether or not DDT, endrin, or degradation compounds are designated as target analytes. The purpose of the evaluation is to determine whether instrument/column maintenance is needed. The preparation of this standard is described in Section 7.16.

10.2.2 The results of the analysis of the EVAL B standard solution are used to calculate column degradation in terms of DDT percent breakdown (%B) and Endrin %B as follows:

$$\text{DDT } \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100\% \qquad \textit{Equation 1}$$

Where A_{DDD}, A_{DDE}, and A_{DDT} are the peak responses for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT, respectively, in the EVAL B chromatogram.

$$\text{Endrin } \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100\% \qquad \textit{Equation 2}$$

Where A_{EK}, A_{EA}, and A_E are the peak responses for endrin ketone, endrin aldehyde, and endrin, respectively, in the EVAL B chromatogram.

10.2.3 Acceptance Criteria

The %B for each of these two compounds, DDT and endrin, must not be greater than 15%.

10.2.4 Corrective Action

If the breakdown of DDT and/or endrin exceeds the 15% limit, corrective action must be taken. This action may include any or all of the following:

- Replacing the injection port liner or the glass wool.
- Cutting off a portion of the injection end of the column or guard column.
- Replacing the GC column or guard column
- Replacing the y-splitter.

After taking the appropriate corrective action, the degradation evaluation standard must be reanalyzed and must pass acceptance criteria before conducting any calibration events.

10.3 The laboratory uses six calibration levels (as shown in Table 3) for the single-component pesticides. The lowest point on the calibration curve is at or below the reporting limit (RL). The highest standard defines the highest sample extract concentration that may be reported without dilution. The preparation of the calibration standards is described in Section 7.8.

10.4 All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.5 Calibration for the multi-peak component analytes, Toxaphene and Technical Chlordane, begins with a single-point calibration at or near the RL. If any multi-peak components are found to be present in the samples, a calibration for the multi-component analyte(s) is conducted with a minimum of five calibration levels. The samples are then reanalyzed using the full calibration curve that brackets the quantitation range.

NOTE: Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious mis-injection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

- The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and
- The lowest remaining calibration point is still at or below the project reporting limit; and
- The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and
- The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average calibration factors or linear regressions, or six levels for second-order curve fits.

NOTE: Second order curves are not allowed for South Carolina work.

10.6 External Standard Calibration

External standard calibration involves the comparison of instrument responses (e.g., peak area or peak height) from the target compounds in the sample to the responses of the target compounds in the calibration standards. The ratio of the detector response to the amount or concentration of target analyte in the calibration standard is defined as the calibration factor (CF), as follows:

$$CF = \frac{A_S}{C_S} \qquad \textit{Equation 3}$$

Where:

A_s = Peak area (or height) of the analyte or surrogate in the calibration standard.

C_s = Concentration of the analyte or surrogate, in ng/mL, in the injected calibration standard.

10.7 Establishing the Calibration Function

Calibrations are modeled either as average calibration factors or as calibration curves, using a systematic approach to selecting the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until the calibration acceptance criteria are met.

10.7.1 Linear Calibration Using Average Calibration Factor

Tabulate the peak area response for each target analyte in each calibration level against the concentration injected. For each analyte in each calibration

standard, calculate the calibration factor (CF) as shown in Equation 3 above. The calibration factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

For each target analyte, calculate the average calibration factor as follows:

$$AverageCalibrationFactor = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n} \qquad \textit{Equation 4}$$

Where:

CF_i = Calibration factor for the i^{th} calibration level.

n = The number of calibration levels.

The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{CF}} \times 100\% \qquad \textit{Equation 5}$$

Where SD is the standard deviation of the average CF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)^2}{n-1}} \qquad \textit{Equation 6}$$

10.7.2 Evaluation of the Average Calibration Factor

The calibration relationship can be graphically represented as a line through the origin with a slope equal to the average calibration factor. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Note: The use of grand average (evaluation of the average response over all the compounds) is no longer allowed. Each compound must meet the RSD criteria.

Acceptance Criteria: The RSD must be ≤ 20%.

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.7.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not necessarily pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). A weighted least squares regression may be used if at least three multi-point calibrations have been performed. The weighting used is the reciprocal of the square of the standard deviation. The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = ax + b \qquad \textit{Equation 7}$$

Where:

y = Instrument response (peak area or height).

x = Concentration of the target analyte in the calibration standard.

a = Slope of the line.

b = The y-intercept of the line.

For an external standard calibration, the above equation takes the following form:

$$A_s = aC_s + b \qquad \textit{Equation 8}$$

To calculate the concentration in an unknown sample extract, the regression equation (Equation 6) is solved for concentration, resulting in the following equation, where C_s is now C_e, the concentration of the target analyte in the unknown sample extract.

$$C_e = \frac{A_e - b}{a} \qquad \textit{Equation 9}$$

Where:

A_s = Area of the chromatographic peak for the target analyte in the calibration standard.

A_e = Area of the chromatographic peak for the target analyte in the sample extract.

a = Slope of the line as determined by the least-squares regression.

C_s = Concentration of the target analyte in the calibration standard.

C_e = Concentration of the target analyte in the sample extract.

b = Intercept of the line as determined by the least-squares regression.

10.7.4 Linear Regression Evaluation

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of a weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit, and preferably less than the MDL.

Also examine the residuals, paying particular attention to the residuals at the low end of the curve. If the intercept or the residuals are large, a second-order regression should be considered.

The linear regression must have a correlation coefficient (r) ≥ 0.990. Some programs (e.g., AFCEE, DoD) require a correlation coefficient ≥ 0.995.

Corrective Action: If the correlation coefficient falls below the acceptance limit, linear regression cannot be used and a second-order regression should be attempted.

10.7.5 Non-Linear Calibration

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$y = ax^2 + bx + c \qquad \textit{Equation 10}$$

Where a, b, and c are coefficients determined using a statistical regression technique; y is the instrument response; and x is the concentration of the target analyte in the calibration standard.

10.7.6 Non-Linear Calibration Evaluation

A minimum of six points must be used for a second-order regression fit.

Acceptance Criteria: The coefficient of determination must be ≥ 0.990.

Second-order regressions should be the last option. Note that some programs (e.g., South Carolina) do not allow the use of second-order regressions.

Before selecting a second-order regression calibration model, it is important to ensure the following:

- The absolute value of the intercept is not large relative to the lowest concentrations being reported.
- The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).

- The distribution of concentrations is adequate to characterize the curvature.

Corrective Action: If the coefficient of determination falls below the acceptance limit and the other calibration models are unacceptable, the source of the problem must be investigated and the instrument recalibrated. Third-order regressions are not allowed at TestAmerica Denver.

10.8 Initial Calibration Verification (ICV), 0.025 µg/mL for most compounds

A mid-level standard that is obtained from a source different from that of the calibration standards (second-source standard) is used to verify the initial calibration (see Section 0). The ICV standard is analyzed immediately following the initial calibration (ICAL).

Acceptance Criteria: The result for the target analyte(s) in the ICV standard must be within ± 20% of the expected value(s).

Corrective Action: If this is not achieved, the ICV standard, calibration standards, and instrument operating conditions should be checked. Correct any problems and rerun the ICV standard. If the ICV still fails to meet acceptance criteria, then repeat the ICAL.

10.9 Calibration Verification

10.9.1 12-Hour Calibration Verification

The 12-hour calibration verification sequence consists of, at a minimum, an instrument blank and the mid-level calibration standard. The 12-hour calibration verification sequence must be analyzed within 12 hours of the initial calibration and at least once every 12 hours thereafter when samples are being analyzed.

NOTE: It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.9.2 Continuing Calibration Verification (CCV), 0.05 µg/mL for most compounds

It may be appropriate to analyze a mid-level standard more frequently than every 12 hours. The mid-level calibration standard is analyzed as the continuing calibration verification (CCV) standard (see Section 7). At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCSs, and method blanks. Some programs, specifically drinking water programs, require a CCV after every 10 samples to minimize the number of samples requiring re-injection when QC limits are exceeded. If 12 hours elapse, analyze the 12-hour standard sequence instead.

10.9.3 RL Standard

It may also be appropriate to analyze a standard prepared at or very near the reporting limit (RL) for the method at the end of the analytical sequence, as a minimum (see Section 7.11). This standard can be used to rule out false negatives in client samples in cases where the %D for one or more of the analytes in a bracketing CCV falls below the lower acceptance limit. The results for the RL standard are not evaluated unless the previous CCV fails acceptance criteria.

10.9.4 Acceptance Criteria for Continuing Calibration Verification (CCV)

10.9.4.1 Detected Analytes (≥/= RL)

For any analyte detected at or above the reporting limit (RL) in client samples, the percent difference (%D) for that analyte in the preceding and following CCVs (i.e., bracketing CCVs) or 12-hour calibration, on the column used for quantitation, must be within ± 20% if an average curve fit is used. For other curve fits (i.e. linear or 2nd order) see DV-QA-027P ehich reduces the allowed %D to 15%.

In some cases, the nature of the samples being analyzed may be the cause of the failing %D. When the %D for an analyte falls outside of ± 20% in the CCV, and that analyte is detected in any or all of the associated samples, then those samples must be reanalyzed (at a dilution if column damage is eminent) to prove a matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect if so desired by the client.

Refer to Section 12 for which result to report.

In cases where additional compounds are to be analyzed in conjunction with compounds defined by this method and that are not defined in the scope and application of method 8081B different CCV acceptance criteria may apply. Kepone is not recommended by method 8081B and the CCV acceptance criteria is defined as +/- 53%. Further these additional compounds will not be used in grand mean calculations as discussed in section 10.9.4.2.

The %D is calculated as follows:

$$\%D = \frac{\text{Measured Conc} - \text{Theoretical Conc}}{\text{Theoretical Conc}} \times 100 \qquad \textit{Equation 11}$$

10.9.4.2 Analytes Not Detected (< RL)

For any analyte not detected in client samples, the %D for that analyte in the bracketing CCVs should also be within ± 20% an average curve fit or within 15% for other curve fits. Method 8081B references method 8000 for compounds with curve fits other than an average curve fit and the criteria in table 6 applies to those compounds. See also DV-QA-027P for further evaluation criteria. Any deviation for the calibration criteria outlined in this procedure must be documented in an NCM.

However, the analysis is acceptable if the average of the %D values for all the analytes is within ± 20% and the %D for any individual analyte is within ± 30%. The average %D is calculated by summing all the %D results in the calibration and dividing by the number of analytes. If an average %D is used and the %D for any individual analytes falls outside of ± 30%, then additional evaluation is needed as summarized in Table 6.

10.10 Retention Time Windows

Retention time (RT) windows must be determined for all analytes.

10.10.1 Determine new RT windows each time a new column is installed or annually, whichever is most frequent.

10.10.2 Make an injection of all analytes of interest each day over a 72-hour period.

10.10.3 Calculate the mean and standard deviation for the three RTs for each analyte as follows:

$$\text{Mean RT} = \overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RT_i - \overline{RT}\right)^2}{n-1}}$$

Equations 12 & 13

Where:

RT_i = Retention time for the i^{th} injection.

n = Number of injections (typically 3).

SD = Standard deviation.

NOTE: For the multi-component analytes, Toxaphene and Technical Chlordane, the mean and standard deviation must be calculated for each of the 3 to 6 major peaks used for sample calculations.

10.10.4 Set the width of the RT window for each analyte at ± 3 standard deviations of the mean RT for that analyte.

10.10.5 The center of the RT window for an analyte is the RT for that analyte from the last of the three standards measured for the 72-hour study.

10.10.6 The center of the window for each analyte is updated with the RT from the level 4 standard of the ICAL, or the CCV at the beginning of the analytical sequence. The width of each window remains the same until new windows are generated following the installation of a new column, or in response to an RT failure. The RT window width may be expanded if the RT drift observed in the ICAL is greater than the established window. The expanded window is noted on the ICAL checklist.

10.10.7 If the RT window as calculated above is less than ± 0.01 minute, use ± 0.01 minute as the RT window. This allows for slight variations in retention times caused by sample matrix.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Preparation

11.3.1 Sample preparation for aqueous samples is described in SOP DV-OP-0006.

11.3.2 Sample preparation for solid samples is described in SOPs DV-OP-0016 and DV-OP-0015.

11.3.3 Cleanup and concentration of sample extracts are described in SOP DV-OP-0007.

11.3.4 The final extract volume in hexane is 10 mL.

11.3.5 Use hexane to dilute sample extracts, if necessary.

11.4 Gas Chromatography

Chromatographic conditions for this method are presented in Table 2. Use the ChemStation interface to establish instrument operating conditions for the GC. Raw data obtained by the ChemStation software is transferred to the TARGET DB database for further processing. The data analysis method, including peak processing and integration parameters, calibration, RT windows, and compound identification parameters, is set up in the TARGET DB software.

11.5 Sample Introduction

All extracts and standards are allowed to warm to room temperature before injection. An autosampler is used to introduce samples into the chromatographic system by direct injection of 1 or 2 µL of the sample extract. Samples, standards, and QC samples must be introduced using the same procedure. Use the ChemStation interface to set up and run the analytical sequence. Sample injection and analysis are automated and may proceed unattended.

11.6 Analytical Sequence

An analytical sequence starts with a minimum five-level initial calibration (ICAL) or a daily calibration verification. Refer to Table 3 for the calibration levels used.

11.6.1 Prior to analyzing any calibration or calibration verification standards, the column degradation evaluation standard is injected and the results are evaluated as described in Section 10.2.

11.6.2 The daily calibration verification includes analysis of the 12-hour calibration sequence (Section 10.9.1) and updating the retention time windows (see Section 10.10).

11.6.3 If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration verification.

11.6.4 The following is a typical analytical sequence:

- Primer
- Hexane blank
- Eval B Std (column degradation evaluation)
- Daily initial CCVs

- LCS
- Method Blank
- 10 samples
- CCVs
- Followed by cycles of 10 samples and CCVs as needed
- Closing CCV

11.7 Daily Retention Time Windows

The center of the retention time (RT) windows determined in Section 10.10 are adjusted to the RT of each analyte as determined in the 12-hour calibration verification. The centers of the RT windows must be updated at the beginning of each analytical sequence and with each 12-hour calibration, but not for any other calibration verification standards.

11.8 Upon completion of the analytical sequence, transfer the raw chromatography data to the TARGET DB database for further processing.

11.8.1 Review chromatograms online and determine whether manual data manipulations are necessary.

11.8.2 All manual integrations must be justified and documented. See DV-QA-011P requirements for manual integration.

11.8.3 Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature.

11.8.4 Alternatively, the manual integration may be processed manually. In the latter case, print both the both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration.

11.9 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD.

11.9.1 Perform a level 1 data review and document the review on the data review checklist (GC and HPLC Data Review Checklist).

11.9.2 Submit the data package and review checklist to the peer reviewer for the level 2 review. The data review process is explained in SOP DV-QA-0020.

12.0 Calculations / Data Reduction

12.1 Qualitative Identification

12.1.1 Tentative identification of an analyte occurs when a peak is found on the primary column within the RT window for that analyte, at a concentration above the reporting limit, or above the MDL if qualified data (J flags) are to be reported. Identification is confirmed if a peak is also present in the RT window for that analyte on the second (confirmatory) column and if the analyte concentration is greater than the MDL. When confirmation is made using a

second column, the analysis on the second column must meet all of the QC criteria for continuing calibration verification and RTs.

12.1.2 The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2 Dual-Column Quantitation and Reporting

12.2.1 Each sample is analyzed on two different columns at the same time. The laboratory designates a primary column based on optimal separation of the compounds of interest and other desirable chromatographic characteristics. The result from the primary column is normally reported. The result from the secondary (confirmatory) column is reported if any of the following is true:

- There is obvious chromatographic interference on the primary column.
- The difference between the result for the primary column and the result for the secondary column is > 40% and chromatographic interference is evident.

12.2.2 Dual Column Results With > 40% RPD

12.2.2.1 If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, the higher of the two results is reported unless there is obvious interference documented on the chromatogram.

12.2.2.2 If there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

12.2.2.3 If there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.

12.2.2.4 The RPD between two results is calculated using the following equation:

$$\%RPD = \frac{\left|R_1 - R_2\right|}{\frac{1}{2}\left(R_1 + R_2\right)} \times 100\% \qquad \textit{Equation 14}$$

Where R_1 is the result for the first column and R_2 is the result for the second column.

12.3 Multi-Component Analytes (Toxaphene and Technical Chlordane)

12.3.1 Qualitative Identification

Retention time windows are also used for identification of multi-component analytes, but the "fingerprint" produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the

compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3.2 Quantitation of Toxaphene

12.3.2.1 While Toxaphene contains a large number of compounds that produce well resolved peaks in a GC/ECD chromatogram, it also contains many other components that are not chromatographically resolved. The unresolved complex mixture results in a "hump" in the chromatogram that is characteristic of the Toxaphene mixture of compounds. The resolved peaks are important for the identification of the mixture, and the area of the unresolved complex mixture contributes a significant portion of the area of the total response.

12.3.2.2 To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the RTs of the first and last eluting Toxaphene components in the standard. In order to use the total area approach, the pattern in the sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present in the sample. Otherwise, the sample concentration may be significantly underestimated.

12.3.2.3 Toxaphene may also be quantitated on the basis of 4 to 6 major peaks. Using a subset of 4 to 6 peaks for quantitation provides results that agree well with the total peak approach and may avoid difficulties when interferences with Toxaphene peaks are present in the early portion of the chromatogram from compounds such as DDT.

12.3.2.4 When Toxaphene is determined using the 4 to 6 peaks approach, care must be taken to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms.

12.3.2.5 The chosen peaks must be within the established retention time. If there is an interference that affects the accuracy of results, the analyst may use as few as 4 major peaks. The same peaks that are used for sample quantitation must be used for calibration.

12.3.2.6 The heights or areas of the chosen peaks should be summed together to determine the Toxaphene concentration.

12.3.2.7 Second column confirmation of multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

NOTE: USACE projects require the use of second-column confirmation of multi-component analytes unless the project work plans (SOW, SAP, QAPP, etc.) specify single-column analysis.

12.3.3 Quantitation of Technical Chlordane

12.3.3.1 Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. Cis-Chlordane (or α-Chlordane) and trans-Chlordane (or γ-

Chlordane) are the two most prevalent major components of Technical Chlordane. However, the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch.

12.3.3.2 When the GC pattern of the sample resembles that of Technical Chlordane, Chlordane may be quantitated by comparing the total area of the Chlordane chromatogram using 3 to 5 major peaks or the total area. If the Heptachlor epoxide peak is relatively small, include it as part of the total Chlordane area for calculation. If Heptachlor and/or Heptachlor epoxide are much out of proportion, calculate these separately and subtract their areas from the total area to give a corrected Chlordane area.

NOTE: Octachlor epoxide, a metabolite of Chlordane, can easily be mistaken for Heptachlor epoxide on a nonpolar GC column.

12.3.3.3 To measure the total area of the Chlordane chromatogram, construct the baseline of Technical Chlordane in each calibration chromatogram between the RTs of the first and last eluting Technical Chlordane components. Use this area and the mass or concentration of Technical Chlordane in each calibration standard to establish the calibration function (Section 10.7). Construct a similar baseline in the sample chromatogram, measure the area, and use the calibration function to calculate the concentration in the sample extract.

12.3.3.4 When the GC pattern of Chlordane in a sample differs considerably from that of the Technical Chlordane standard, it may not be practical to relate a sample chromatogram back to the Technical Chlordane standard chromatogram. In these cases, all identifiable Chlordane components may be summed and reported as "Chlordane (not otherwise specified, CAS number 57-74-9)."

12.3.3.5 A third option for quantitating Technical Chlordane is to quantitate the peaks for α-Chlordane, γ-Chlordane, and Heptachlor separately against the appropriate reference materials, and report these individual components under their respective CAS numbers.

NOTE: CHD Flag section 12.6.2

12.3.3.6 Second column confirmation of multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

NOTE: USACE projects require the use of second-column confirmation of multi-component analytes unless the project work plans (SOW, SAP, QAPP, etc.) specify single-column analysis.

12.4 Surrogate recovery results are calculated and reported for DCB. TCMX may also be added, however if the two surrogate compounds are analyzed, and recoveries are calculated, and either surrogate fails to meet control limits, corrective actions are required (this also applies to programs that require the use of only one surrogate). See section 9.7 for further details.

12.5 Calibration Range and Sample Dilutions

12.5.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for the analyte(s) that were found to be over the calibration range in the high sample, they must be reanalyzed to rule out carryover, unless other objective evidence indicates that the detection is not the result of carryover. Such evidence may include an observation where carryover was not observed when samples or blanks were analyzed after another sample with similar high compound recovery or when the detection in the sample with suspected carryover is much higher than the expected amount of carryover (i.e. the sample's concentration may be similar to or higher than the concentration found in the previous sample). It may also be necessary to dilute samples because of matrix interferences.

12.5.2 If the initial diluted run has no hits or hits below 20% if the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.5.3 Guidance for Dilutions Due to Matrix Interference

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

12.5.4 Reporting Dilutions

Some programs (e.g., South Carolina and AFCEE) and some projects require reporting of multiple dilutions (check special requirements in LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported.

12.6 Interferences Observed in Samples

12.6.1 Dual column analysis does not entirely eliminate interfering compounds. Complex samples with high background levels of interfering organic compounds can produce false positive and/or false negative results. The analyst must use appropriate judgment to take action as the situation warrants.

12.6.2 Suspected Negative Interferences

If peak detection is prevented by interferences, further cleanup should be attempted (see SOP DV-OP-0007). Elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

If the individual isomers of chlordane are identified, but there is no pattern for the confirmation of "Technical Chlordane", and the project has ONLY technical chlordane requested, the results for technical chlordane should be qualified ("CLD") by the analyst to indicate the presence of the chlordane isomers.

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Title: Acid Digestion of Aqueous Samples for Metals Analysis by ICP

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1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the acid digestion of aqueous samples by SW-846 Method 3005A, or SW-846 Method 3010A prior to the determination of the concentration of individual metallic elements by inductively coupled plasma atomic emission spectroscopy (ICP). These methods include digestions for total, total recoverable, dissolved, and potentially dissolved analytes (see definitions in Section 3).

1.2 This SOP is applicable to ground water, surface water, domestic and industrial wastewater, TCLP leachates, and other aqueous media. This SOP is not applicable to oils or other liquids that are not miscible in water.

NOTE: Samples that are found to be immiscible with water, e.g., contain or consist of oil or other immiscible organic solvents, are subcontracted to other labs that have the capability of handling such samples. If during the preparation process it is discovered that the sample is immiscible with water or is biphasic, then the analyst notifies the Group Leader and Project Manager, who can subcontract the samples to a laboratory with the capability to handle the oil matrix.

1.3 The following table summarizes the applicability of the various digestion methods referenced in this SOP. All sample digestates are analyzed by ICP in accordance with SOP DV-MT-0012.

3005A	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP	Preparation of surface and ground water samples for total recoverable or dissolved metals for analysis by ICP.	10.9
3010A	Acid Digestion of Aqueous Samples and Extracts for Total Metals Analysis by ICP	Preparation of aqueous samples, EP and mobility procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP.	10.6

1.4 Sample digestion requirements are established by the laboratory Project Manager before samples are received, and the LIMS codes applied to samples indicate which digestion is to be used for each sample.

1.5 This procedure can be used for all of the elements listed in Table 1. Additional elements may be analyzed using the digestion methods in this SOP provided that the method performance criteria specified in Section 12 and the QC acceptance criteria specified in Section 9 of this SOP and the ICP determinative SOP, DV-MT-0012 are met.

1.6 All samples require digestion prior to analysis, with the possible exception of "direct analysis" of dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples. This must be determined by

the laboratory Project Manger before projects start, and is communicated to the analysts through special instructions in LIMS.

2.0 Summary of Method

2.1 Method 3005A, Total Recoverable, Dissolved Metals or Potentially Dissolved Metals

A representative portion of sample is heated with diluted nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to volume.

2.2 Method 3010A Total Metals

A representative portion of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary), and brought up to volume.

3.0 Definitions

3.1 Dissolved Analyte: The concentration of analyte in an aqueous sample that will pass through a 0.45-µm membrane filter prior to acidification (sample is acidified after filtration).

3.2 Potentially Dissolved Metals: The concentration of elements in solution after acidifying the sample with nitric acid to pH < 2, holding at room temperature for 8 to 96 hours, and then filtering through a 0.45-µm membrane filter. This definition is based on the Colorado surface water regulations.

3.3 Total Recoverable Analyte: The concentration of analyte determined by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s).

3.4 Total Metals: The concentration of elements in an unfiltered sample subject to a more rigorous nitric acid / hydrochloric acid digestion than is used for total recoverable metals

4.0 Interferences

4.1 Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, and atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not miscible with acids. If physical interferences are present, they should be documented in the final report case narrative.

4.3 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented in the final report case narrative.

4.4 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.5 Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample. Samples containing more than 1 mg/L of silver are redigested at a reduced sample volume and reanalyzed to produce more accurate results.

4.6 Specific analytical interferences are discussed in the ICP determinative method. See SOP DV-MT-0012.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 Samples that contain high concentrations of carbonates or organic material, or samples that are at elevated pH can react violently when acids are added.

5.3.3 Care must be taken when handling the digestion tubes. The tubes may become very hot during the digestion procedure. Allow the tubes to cool before attempting to touch the sample digestate.

5.4 Primary Materials Used

5.4.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Material Safety Data Sheet (MSDS) for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit(2)	Signs and Symptoms of Exposure
Stock Standard Solutions	Oxidizer Corrosive Poison	5 mg/m^3 as HNO_3	Toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Nitric Acid (HNO_3)	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid (HCl)	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Digestion block, with adjustable heating, capable of maintaining a sample temperature of 90 - 95 °C.

6.1.2 Thermometer that covers a temperature range of at least 80 - 110 °C, in increments of 1 °C.

6.1.3 Liquid-filled thermometers must have a tag indicating that the accuracy was checked by the QA group within the last 12 months.

6.1.4 Digital thermometers must have a tag showing that they were checked within the last three months.

6.1.5 See SOP DV-QA-0001 for details of the calibration procedure.

6.1.6 Centrifugation equipment (when desired method of removing particulates is centrifugation).

6.1.7 Calibrated mechanical pipettes with pipette tips or Class A glass volumetric pipettes. Pipette calibration is checked in accordance with SOP DV-QA-0008.

6.2 Supplies

6.2.1 Disposable digestion tubes, with volume accuracy verified to ± 3% gravimetrically prior to use.

6.2.2 Watch glasses, ribbed or equivalent, or disposable digestion tube covers.

6.2.3 Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters, No. 6973-2504, for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.

6.2.4 Syringes or equivalent filtration apparatus.

6.2.5 Graduated cylinder or equivalent, capable of measuring 50 mL to ± 3% accuracy.

6.2.6 Re-pipettors or suitable reagent dispensers.

6.2.7 Class A volumetric flasks.

6.2.8 pH indicator strips (pH range 0 - 6).

6.2.9 Plastic digestate storage bottles.

7.0 Standards and Reagents

7.1 Standards must be NIST traceable, where available. Standards are verified against a second-source standard before they are put into use (the only exception is standards purchased directly from NIST), which is described in SOP DV-QA-0015.

7.2 Stock standards are purchased as custom TAL multi-element mixes or as single-element solutions. Standards are logged into the TAL Denver Standards Log database and are assigned unique identification numbers that can be used to

access traceability information. The Standards Log identification numbers are recorded on the metals prep bench sheet

7.3 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. These plastic bottles may be stored in a glass jar.

7.4 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.5 Standards containing silver must be protected from light using either a cardboard box or amber containers.

7.6 Shelf-Life

7.6.1 Stock standards, standards as received from the vendor, expire on the date assigned by the vendor. If no date is assigned by the vendor, then one-year expiration will be assigned by the laboratory.

7.6.2 Intermediate concentration standards or working standards may be used for up to three months. The expiration date cannot be later than the date assigned to the stock standard.

7.6.3 Any suspect standards are re-verified, and replaced if re-verification fails.

7.7 Laboratory Control Sample (LCS) Spike Stock Standards

The LCS spike stock standard are custom-made standards purchased from Inorganic Ventures. The standards are designated TALDEN-SPK-2A (“ICP2) and TALDEN-SPK-3A (“ICP1”) and contain the following elements at ready-to-use concentrations:

LCS Spike Stock Standards "ICP 1 & 2"

Elements in LCS Spike	Concentration in ppm (µg/mL)
Ca, K, Mg, Na	5,000
P, Si	1,000
Zr, Sb	500
Al, Ba, Bi, Se, Tl, U, Sn	200
Fe, Sr, Li, B, Mo, Ti, As, Th	100
Co, Mn, Ni, Pb, V, Zn, Sb, Zr	50
Cu	25
Cr	20
Cd	10
Ag, Be	5

7.8 TCLP ICP Spike Stock Standard

The TCLP spike stock standard is purchased from commercial sources. The stock is a custom-made standard purchased at ready-to-use concentrations, as follows:

TCLP ICP Spike Stock Standard

Elements in TCLP Spike	Concentration in ppm (µg/mL)
Ba	1,000
Cr, Pb	500
As	300
Cu, Zn	200
Ag, Cd, Se	100

7.9 TCLP Mercury Spike Solution

TCLP leachate matrix spike samples are spiked for both ICP elements and mercury at the time of sample preparation but before preservation. The mercury spike standard is prepared as the mercury calibration working standard solution at a concentration of 10 mg/L by the mercury analyst as described in SOP DV-MT-0015 (Section 7.13).

7.10 Reagent water must be produced by a Millipore de-ionized system or equivalent and must achieve the performance specifications for ASTM Type II water, i.e., conductivity < 1.0 µmhos/cm; resistivity > 1.0 megohms-cm; silica < 3.0 µg/L. In addition, the reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOP, DV-MT-0012.

7.11 Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.12 Hydrochloric acid (HCl), concentrated, trace metal grade or better.

8.0 Sample Collection, Preservation, Shipment and Storage

Preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	HDPE	500 mL	HNO_3, pH < 2	180 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12 for more details initial demonstrations of capability, and analyst training and qualification.

9.3 Preparation Batch

A preparation batch is a group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, a matrix spike (MS), and a matrix spike duplicate (MSD). In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify samples for the MS/MSD pair, then the batch may contain multiple MS/MSD pairs to accommodate client requests. Clients may also request a duplicate LCS (LCSD). In cases where the client has not provided sufficient

sample to prepare an MS and MSD, an LCS and LCSD will be prepared instead.

9.4 Sample Count

Laboratory-generated QC samples (method blanks, LCSs) are not included in the sample count for determining the size of a preparation batch. The MS and MSD are usually not included in the sample count.

9.5 Method Blank (MB)

9.5.1 The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. When samples are filtered in the laboratory for determination of dissolved metals, then the blank is filtered using a filter of the same type that was used for the samples. The performance of the filtration process must be acknowledged on the Supplemental Metals Prep Sheet.

9.5.2 TCLP method blanks are prepared by taking 50 mL of TCLP leachate fluid (see SOP DV-IP-0012) through the appropriate procedure as described in Section 10.

9.5.3 One method blank must be processed with each preparation batch. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false-positive data. Method blank results are evaluated by the ICP analysts as described in SOP DV-MT-0012.

9.5.4 Acceptance Criteria

The method blank should not contain any analyte of interest at or above the reporting limit (RL) or at or above 10% of the measured concentration of that analyte in associated samples, whichever is higher. In other words, the sample result must be a minimum of 10 times higher than the blank contamination level.

9.5.5 Corrective Action

If the method blank does not meet the acceptance criteria, the blank and all associated samples in the batch must be re-digested and reanalyzed.

9.6 Laboratory Control Sample (LCS)

9.6.1 One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. When samples are filtered in the laboratory for determination of dissolved metals, then the LCS is filtered using a filter of the same type that was used for the samples. The performance of the filtration process must be acknowledged on the Supplemental Metals Prep Sheet.

9.6.2 An LCS for a batch of aqueous samples is prepared by adding 0.5 mL of each of the LCS spike stock standards, "ICP 1 & 2", (Section 7.7) to 50 mL of reagent water. This produces the final concentrations shown in Table 1.

9.6.3 An LCS for a TCLP batch is prepared by adding 0.5 mL of each of the LCS spike stock standards, "ICP 1 & 2", (Section 7.7) plus 0.5 mL of the TCLP Stock Standard (Section 7.8) to 50 mL of the TCLP leachate solution (see SOP DV-IP-0012). This produces the final concentrations shown in Table 2.

9.6.4 The LCS is used to monitor the accuracy of the analytical process. LCS results are evaluated by the ICP analyst, as described in SOP DV-MT-0012. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.6.5 Acceptance Criteria

LCS recovery control limits are set at ± 3 standard deviations about the historical mean. These limits must not be wider than 85 - 115 % recovery for Method 200.7 or 80 - 120 % for Method 6010. The control limits are maintained in the LIMS system.

9.6.6 Corrective Action

If the LCS % recovery falls outside of the control limits for any analyte, that analyte is judged to be out of control. All associated samples must be reprocessed for analysis. One possible exception is a recovery for a given element above the upper control limit with no detection for the same element in the samples. This latter case must be explained in the case narrative.

9.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.7.1 A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Normally, one MS/MSD pair is prepared each preparation batch. Samples identified as field blanks, equipment blanks, or rinse blanks cannot be used for MS/MSD analysis.

9.7.2 Some programs (e.g., South Carolina and North Carolina) require that MS/MSD pairs are run at a 10% frequency. Also, some clients may require unspiked duplicate samples in place of or in addition to an MS/MSD pair. Check special project instructions in the Client Requirements Checklist of LIMS before starting the batch.

9.7.3 If insufficient sample is available to process an MS/MSD pair, then a second LCS must be processed and an NCM generated. The LCS pair

is then evaluated according to the MS/MSD criteria.

9.7.4 The purpose of analyzing matrix spike samples is to assess the effect of the sample matrix on the accuracy and precision of the analysis. MS/MSD results are evaluated by the ICP analysts as described in SOP DV-MT-0012. If the MS/MSD results fail to meet control limits, while the LCS results were in control, that indicates that something about the sample is interfering with the analysis.

9.7.5 Matrix spikes for aqueous sample batches are prepared by adding 0.5 mL of each of the LCS spike stock standards, "ICP 1 & 2", (Section 7.7) to a digestion tube containing 50 mL of the selected sample. The final spike concentrations are shown in Table 1.

9.7.6 Matrix spikes for TCLP batches are prepared by adding 0.5 mL of the TCLP Stock Standard (Section 7.8) plus 0.5 mL of each of the LCS spike stock standards, "ICP 1 & 2", (Section 7.7) to 50 mL of the parent TCLP aliquot. A second aliquot is spiked for mercury analysis at the same time by adding 1.5 mL of the 100 mg/L Hg standard to 30ml of parent sample. The matrix spike samples are then preserved with HNO_3 to pH < 2. The final spike concentrations are shown in Table 2.

NOTE: The TCLP matrix spike must be added prior to preservation of the leachate.

9.7.7 Acceptance Criteria

The recovery for each analyte must fall within established limits. The relative percent difference (RPD) between the MS and MSD must be less than or equal to the established RPD limit. If any analyte recovery or relative percent difference (RPD) between the MS and MSD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS.

9.7.8 Corrective Action

If the recovery of the LCS also failed acceptance criteria, then corrective action must be taken. Corrective action will include re-preparation and reanalysis of the batch. If MS results fail to meet control limits, but the LCS results are within limits, then samples do not require re-preparation and reanalysis, unless the results indicate that a spiking error may have occurred.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for

potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

10.3.1 Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating sample preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous, but it appears to be more like a waste (biphasic, oil, sludge-like, organic liquid, lots of sediment, etc.), then contact the project manager and the laboratory group leader for further instructions. It may be necessary to subcontract these samples to a laboratory with the capability to digest organic matrices.

NOTE: TAL Denver has not implemented digestion methods for water-immiscible organic matrices, e.g., oils. Samples that are known to be incompatible with TAL Denver digestion techniques are typically subcontracted to other laboratories.

10.3.2 All samples are to be checked out of sample control with the chain of custody documentation filled out completely. Select the sample(s) designated for either total or dissolved metals.

10.3.3 Proper sample identification is extremely important in any preparation procedure. Labeling of beakers, digestion tubes, and bottles must be done in a manner to ensure connection with the proper sample.

10.3.4 If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab, data review, and reporting groups.

10.3.5 Guidelines are provided in Appendix 1 on procedures to minimize contamination of samples and standards.

10.4 Aqueous Sample Preparation Setup

The following setup procedure must be followed for all aqueous samples prior to performing the specific digestion procedure. The sample preparation procedure for Method 3005A and 3010A detailed in the following sections is also summarized in Figures 1 and 2 of this SOP and in work instruction WI-DV-016.

10.4.1 Sample pH is verified during sample receipt. When a sample is received with improper/insufficient preservation, the sample is delivered with notification of the deficiency.

10.4.1.1 Measure the sample pH with pH paper using a separate

aliquot of sample. This can be done using disposable bulbs.

10.4.1.2 If the pH>2 for a sample requiring acidic preservation, record the pH in the Metals Prep Log and record the anomaly using in a NCM. Samples cannot be digested for 24 hours after preservation. Samples must be kept with a sample receiving Rush form during the 24 hour wait period.

10.4.1.3 All water sample pH's must be verified and documented before digestion.

10.4.1.4 Add 1-2 mL of conc. HNO_3 to the sample. Replace the lid and mix the sample.

10.4.1.5 Recheck the pH of the sample. If the pH<2, record the volume of acid added in the Metals Prep Log. If the pH>2, repeat Section 10.4.1.3 until pH<2. Record the volume of HNO_3 added.

10.4.1.6 Allow the sample to sit for 8-16 hours following acidification.

10.4.1.7 After 8-16 hours, recheck the pH of the sample. If the pH<2, proceed with the appropriate digestion procedure. Note the date/time of this pH recheck in the Metals Prep Log.

10.4.1.8 If after 8-16 hours the pH>2, repeat steps 10.4.1.3 through 10.4.1.6 until the pH remains <2 following the 8-16 hour period.

Note: Acid must be added at least 24 hours before digestion.

10.4.1.9 Leachates or portions of leachates for metallic analyte deteminations must be acidified with nitric acid to a pH less than 2 unless precipitation occurs. Test a small portion of sample to see if precipitation occurs. If a precipitation forms do not acidify the leachate and analyzed as soon a possible. Leachates may be digested as soon as they are acidified.

10.4.2 Select the unfiltered fraction for a total or total recoverable analysis or the filtered fraction for a dissolved analysis. If requested by the client, select the filtered fraction for a total dissolved analysis. For TCLP and SPLP, select the proper sample leachates.

NOTE: Samples requiring dissolved metals determination are either filtered and preserved in the field or are filtered and preserved by the laboratory as soon as possible after receiving the samples. When filtered in the laboratory, the filtration and preservation are recorded in the Laboratory Sample Filtration and Preservation Logbook, including the preservative type and lot number. Filter acceptability is demonstrated by using filters

of the same type to filter samples and batch QC samples when preparation batches include samples that were filtered in the laboratory. The results of the analysis of the batch QC samples are used to demonstrate that the filtration process neither adds nor subtracts target analytes from samples. The performance of the filtration process is recorded in LIMS.

10.4.3 Mix the sample by shaking the container.

10.4.4 Measure and transfer 50 mL of the sample into a digestion tube. When using calibrated digestion tubes, pour the sample into the tube to the 50-mL mark. Unless specifically required for a project, all samples are measured by volume and not by weight. Record the volume and units on the preparation bench sheet in LIMS. Also, record the lot number of the digestion tubes used in LIMS.

10.4.5 Mix the sample by shaking the container and then measure two extra aliquots of the sample that is selected for the MS/MSD analysis. Spike each aliquot as described in Section 9.7. Refer to Section 9.7.6 for specific instructions for spiking the selected TCLP sample. Record the standards and pipette identifications in LIMS.

10.4.6 Measure and transfer 50 mL of reagent water into a digestion tube for the method blank. If a determination of dissolved metals is requested (LIMS 3005A), use filtered reagent water for the method blank. For TCLP sample batches, use an aliquot of the TCLP leachate solution for the blank. See Section 9.5 for a detailed description of the method blank.

10.4.7 Measure and transfer 50 mL of reagent water into a digestion tube for the LCS and add the spiking solutions as described in Section 9.6.2. For TCLP sample batches, use the TCLP leachate fluid for preparing the LCS (Section 9.6.3). Record the standards and pipette identifications in LIMS. If determination of dissolved metals is requested and one or more samples were filtered in the laboratory, then filter the LCS using a filter of the same type that was used to filter the sample(s).

10.4.8 If the analysis is for total recoverable, dissolved metals, or potentially dissolved metals, continue on with Section 10.5. If the analysis is for total metals, skip Section 10.5 and go to Section 10.6.

10.5 Total Recoverable, Dissolved, or Potentially Dissolved Digestion for Waters by 3005A

10.5.1 Add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl to the sample in the digestion tube.

10.5.2 Heat at 90 - 95 °C until the volume is reduced to between 15 and 20 mL. Record the digestion block temperature on the Supplemental Metals Prep Sheet. Record the start and stop times and the digestion block temperature in LIMS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

10.5.3 Allow the digestion tube to cool in a fume hood.

10.5.4 Wash down the digestion tube walls and watch glass (or digestion tube cover) with reagent water.

10.5.5 If insoluble materials are present, filter the sample.

10.5.6 Add 1.5mL of concentrated HNO_3 to the digestate.

10.5.7 Revolume to 50 mL with reagent water. Cap and shake to mix.

NOTES: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Instead of filtering, the samples can be diluted and mixed (step 11.9.7) and then centrifuged or allowed to settle overnight to remove insoluble material from the supernatant solution.

10.5.8 The sample is now ready for analysis.

10.6 Total Metals Digestion for Waters or TCLP Leachates by 3010A

10.6.1 Add 1.5 mL of concentrated HNO_3 to the sample in the digestion tube.

10.6.2 Heat at 90-95 °C until volume is reduced to 10 ± 5 mL. Record the digestion block temperature in LIMS. Record the start and stop times and the digestion block temperature in LIMS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

10.6.3 Allow the digestion tube to cool in a fume hood.

10.6.4 Add another 1.5 mL portion of concentrated HNO_3 and cover the sample

10.6.5 Continue refluxing until the digestion is complete.

NOTE: Digestion is complete when the digestate is light in color or does not change in appearance. For most samples the addition of two nitric acid aliquots is sufficient, additional aliquots of nitric acid may be added if necessary.

10.6.6 Evaporate to a low volume of 5 to 10 mL. If the sample does go to dryness, the digestion must be started over using a fresh portion of sample.

10.6.7 Allow the digestion tube to cool in a fume hood.

10.6.8 Add 2.5 mL of concentrated HCl.

10.6.9 Cover and reflux for an additional 15 minutes to dissolve any precipitate or residue.

10.6.10 Wash down the digestion tube walls and watch glass (or digestion tube cover) with reagent water.

10.6.11 Adjust to 50 mL final volume with reagent water. This must be done volumetrically, and not using a balance.

10.6.12 If insoluble materials are present, the sample will be filtered at the instrument by the analyst.

NOTES: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Instead of filtering, the samples can be diluted and mixed (step 10.10.14) and then centrifuged or allowed to settle overnight to remove insoluble material from the supernatant solution.

10.6.13 The sample is now ready for analysis.

10.7 Calibration

10.7.1 The digestion block temperature must be maintained between 90 and 95 °C. The temperature must be monitored continuously while in use and must be recorded in LIMS. The temperature must be monitored by measuring the temperature of reagent water contained in a capped digestion tube that is placed in each digestion block. The thermometer used and the start and end times for all temperature cycles are recorded in LIMS

10.7.2 The thermometer is calibrated in accordance with SOP DV-QA-0001, Thermometer Calibration.

11.0 Calculations / Data Reduction

11.1 This SOP does not produce any analytical data. See the determinative method SOP, DV-MT-0012, for data analysis and applicable calculations.

11.2 Documentation

11.2.1 The laboratory LIMS system stores all of the preparation information.

11.2.2 The LIMS system documentation includes:

11.2.2.1 Batch number, job and sample numbers, preparation date, and analyst name;

11.2.2.2 Matrix and prep type;

11.2.2.3 Initial sample volume and final volume;

11.2.2.4 Reagent manufacturer and lot number;

11.2.2.5 Digestion tube lot information;

11.2.2.6 Standard identification number for each standard used;

11.2.2.7 Calibrated measuring equipment used (thermometers, balances, pipettes, etc.).

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy DV-QA-005P. An MDL verification is performed once a year to satisfy NELAC 2003 requirement. For DoD and AFCEE projects, an MDL verification is performed quarterly. MDLs are stored in the LIMS.

12.1.2 The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

An initial demonstration of capability for each method must be performed prior to analyzing samples.

12.2.1 For the standard analyte list, the initial demonstration consists of the preparation and analysis of a QC check sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study.

12.2.2 Four aliquots of the QC check sample are analyzed with the same procedures used to analyze samples, including sample preparation.

12.2.3 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples.

12.2.4 For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is successful analysis of an extracted standard at the reporting limit and a single point calibration.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

13.1 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, “Waste Management and Pollution Prevention”, of the Environmental Health and Safety Manual, and HS-001, “Waste Management Program.”

14.2 The following waste streams are produce when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards: Contact Waste Coordinator

14.2.2 Acidic waste from sample digests: Waste Stream J.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.1.2 Method 3010A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

16.0 Method Modifications

16.1 Modifications Specific to MCAWW Methods

It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846 methods and therefore were combined under the scope of this SOP as described in Section 10.0. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness versus an exact volume).

16.2 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common laboratory contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

16.3 The referenced methods use 100 mL of sample for digestion. This SOP uses a 50 mL aliquot, with a proportional reduction in digestion reagents. This change is made to allow better control of temperature and potential sample contamination with the use of the digestion block. It is also considered one of the laboratory's hazardous waste reduction initiatives.

16.4 The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document states "flexibility to alter digestion volumes is addressed and 'allowed' by the table (3-1) and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..." EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."

17.0 Attachments

Figure 1. Total Recoverable, Dissolved, or Potentially Dissolved Digestion (SOP Section 10.5)
Figure 2. Total Metals Digestion for Waters or TCLP Leachates (SOP Section 10.6
Table 1. Matrix Spike and Aqueous Laboratory Control Sample Levels
Table 2. TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels
Appendix 1. Contamination Control Guidelines

18.0 Revision History

- Revision 4.7, dated 18 July 2012

- Annual review
- Updated Section 9.1, 10.1 and 10.2 to reflect current practice
- Updated Section 9.7.6 on spiking TCLP aliquots
- Added section 10.4.1.9 for TCLP preservation
- Removed Appendix 2. Added reference to work instruction in Section 10.4
- Updated Figures 1 and 2 to reflect current practice.
- Formatting and editorial changes throughout

- Revision 4.6, dated 24 August 2011
  - Added recommendation to use disposable bulbs for pH checking in section 10.8.1.
  - Added requirement to store samples with a Rush form after preserving in section 10.8.1.2.

- Revision 4.5, dated 31 January 2011
  - Change note in section 10.8.1.8 to be 24 hours before preparation.

- Revision 4.4, dated 01 September 2010
  - Annual Technical Review.
  - Section 7.6 prep spike standard updated to TALDEN-SPK-3A, Bi added.
  - Added to section 7.8 that TCLP's are spiked prior to preservation.
  - Removed all references to Quantims ID's.
  - All references to supplemental metals prep sheets were removed from Section 10.
  - Updated Section 11.2 to reference the LIMS for all documentation.
  - Added Bismuth to Table 1
  - Removed example prep sheets (Appendix 2)
  - Removed prep codes from old LIMS in Appendix 3.

- Revision 4.3, dated 24 August 2009
  - Section 16.1 was updated to reference section 10 and not section 11.

- Revision 4.2, dated 19 June 2008
  - Basic Annual Review.

- Revision 4.1, dated 13 June 2008
  - Added requirement to wait 24 hours after the addition of acid to samples.

- Revision 4, dated 18 March 2008
  - Integration for TestAmerica and STL operations.

Changes From Previous Version of the SOP

- Updated formatting and added table of contents.
- Revised Section 1 to better reflect the scope of the SOP. Included a note explaining that samples that are not miscible with aqueous acids cannot be prepared using this SOP.
- Specified type of filters used.
- Revised Section 9.1 to include a reference to Policy QA-024 for QC requirements specific to federal programs.
- Expanded Sections 9.5, 9.6, and 937 to provide more detailed information for the method blank, LCS, and MS/MSD, respectively.
- Updated Sections 10 and 11 to reflect current laboratory practice.
- Added Appendix 1 to provide guidelines for contamination control.
- Added Appendix 2 to show Supplemental Metals Prep Sheet.
- Added Appendix 3 Example Work Instruction

Figure 1.

Flow Chart for Total Recoverable, Dissolved, or Potentially Dissolved Digestion by SW-846 3005A (SOP Section 10.5)

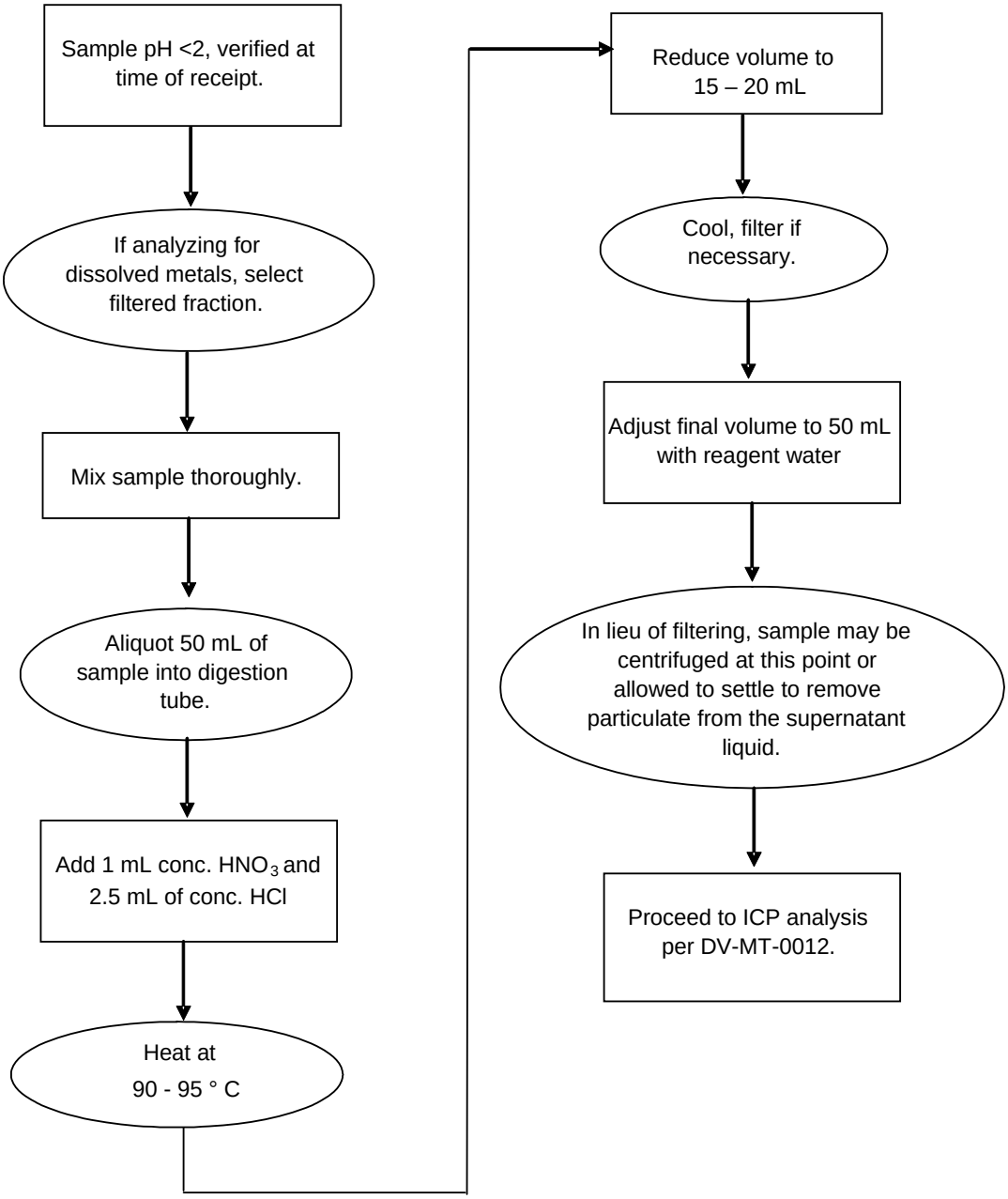

Figure 2.

Flow Chart for Total Metals Digestion for Waters or TCLP Leachates by SW-846 3010A (SOP Section 10.6)

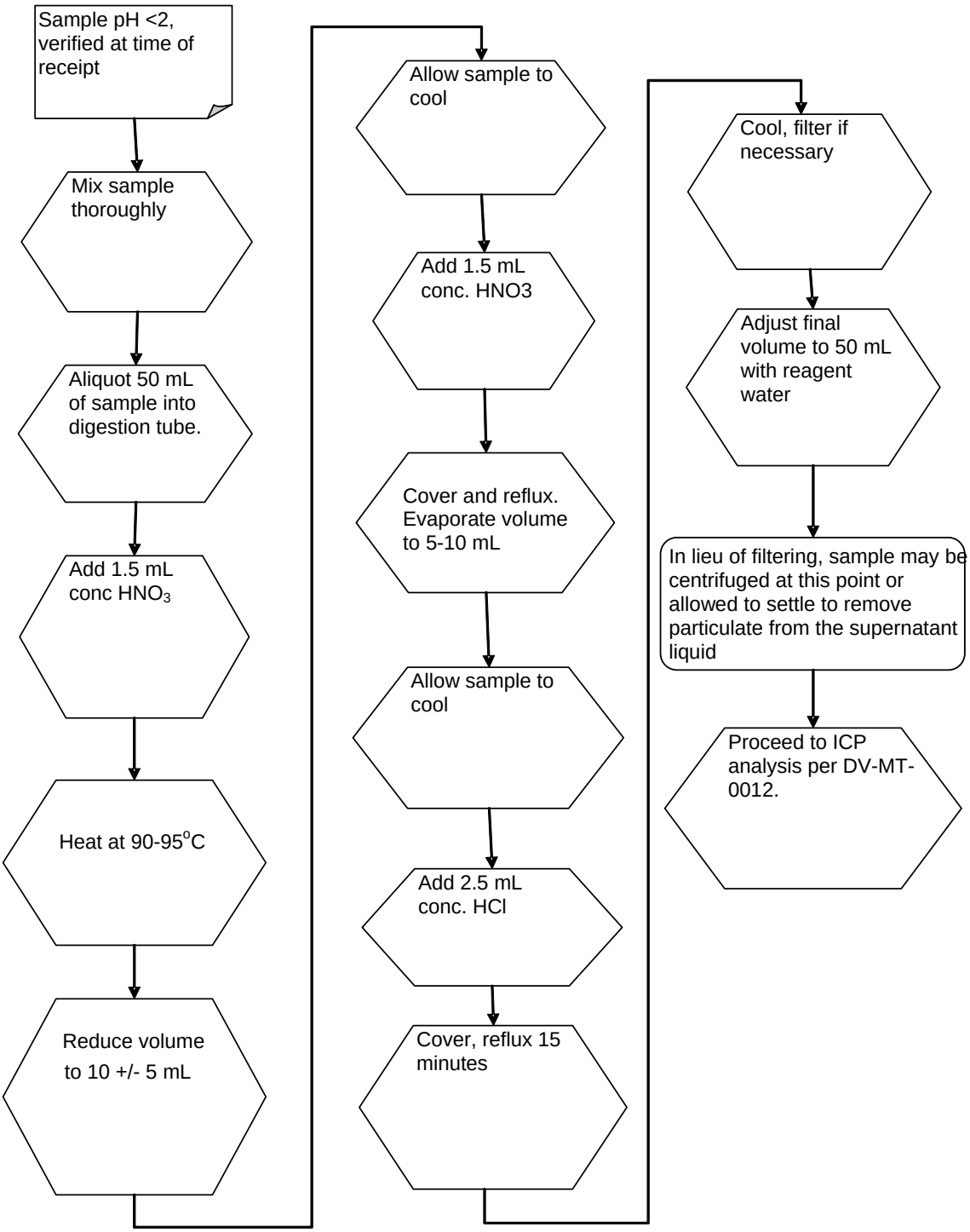

Table 1.

Matrix Spike and Aqueous Laboratory Control Sample Levels

Element	LCS Concentration (ug/L)	Matrix Spike Concentration (ug/L)
Aluminum	2,000	2,000
Antimony	5,000	5,000
Arsenic	2,000	2,000
Barium	2,000	2,000
Beryllium	50	50
Bismuth	2,000	2,000
Boron	1,000	1,000
Cadmium	50	50
Calcium	50,000	50,000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1,000	1,000
Lead	500	500
Lithium	1,000	1,000
Magnesium	50,000	50,000
Manganese	500	500
Molybdenum	1,000	1,000
Nickel	500	500
Phosphorous	10,000	10,000
Potassium	50,000	50,000
Selenium	2,000	2,000
Silicon	10,000	10,000
Si (as SiO2)	21,400	21,400
Silver	50	50
Sodium	50,000	50,000
Strontium	1,000	1,000
Thallium	2,000	2,000
Tin	2,000	2,000
Titanium	1,000	1,000
Uranium	2,000	2,000
Vanadium	500	500
Zinc	500	500
Zirconium	5,000	5,000

Table 2.

TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels

Element	RL (mg/L)	Regulatory Limit (mg/L)	Spike Level (mg/L)
Arsenic	0.1	5,000	5.0
Barium	1.0	100,000	12.
Cadmium	0.05	1,000	1.05
Chromium	1.0	5,000	5.2
Lead	0.03	5,000	5.5
Selenium	0.05	1,000	3.0
Silver	0.1	5,000	1.05

Appendix 1.

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) [Method No(s). SW846 1311 and 1312]

Approvals (Signature/Date):

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Susan Decker — Date
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Karen Kuoppala — Date
Quality Assurance Manager

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Robert C. Hanisch — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW-846 Method 1311. The Toxicity Characteristic (TC) of a sample is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a sample. The TC is one of four criteria in 40 CFR Part 261 to determine whether a sample is classified as a hazardous waste. The other three are corrosivity, reactivity and ignitability. The TC Rule utilizes the TCLP method to generate the leachate under controlled conditions that were designed to simulate leaching through a landfill. EPA's "worst case" waste disposal model assumes mismanaged wastes will be exposed to leaching by the acidic fluids generated in municipal landfills. The EPA's model also assumes the landfill fluids will dominate the acid/base characteristics of the waste. The TCLP procedure directs the testing laboratory to use a more acidic leaching fluid if the sample is an alkaline waste, again in keeping with the model's assumption that the acid fluids will dominate leaching chemistry over time.

1.2 The specific list of TC analytes and regulatory limits may be found in Attachment 1.

NOTE: The list in Attachment 1 does not include the December 1994 EPA rule for Universal Treatment Standards for Land Disposal Restrictions. Those requirements include 216 specific metallic and organic compounds and, in some cases, lower detection limit requirements (see 40 CFR 268.40). TCLP leachates are part of the new Universal Treatment Standards, but the conventional analytical methods will not necessarily meet the new regulatory limits. Consult with the client and with TestAmerica Laboratories Technical Specialists before establishing the instrumental methods for these regulations.

1.3 This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) which was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW-846 Method 1312. The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in Attachment 1. With the exception of the use of a modified extraction fluid, the SPLP and TCLP protocols are essentially equivalent. Where slight differences may exist between the SPLP and TCLP they are distinguished within this SOP.

1.4 The procedure is applicable to liquid, solid, and multiphase wastes.

1.5 The results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled.

1.6 The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits.

1.7 If a total analysis of the waste demonstrates that individual analytes are not present in the waste or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the procedure need not be run. If the total analysis results indicate that TCLP is not required, the decision to cease TCLP analysis should be remanded to the client.

1.8 If an analysis of any one of the liquid fractions of the leachate indicates that a regulated compound is present at such a high concentration that, even after accounting for dilution from the other fractions of the leachate, the concentration would be equal to or above the regulatory level for that compound, then the waste is hazardous and it may not be necessary to analyze the remaining fractions of the leachate. However, the remaining analyses should not be terminated without the approval of the client.

2.0 Summary of Method

2.1 For liquid samples that contain less than 0.5% dry solid material, the sample, after filtration through 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP leachate and reagent water is used as the blank fluid.

2.2 For samples containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solids and stored for later analysis. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is leached with an amount of leach fluid equal to 20 times the weight of the solid phase. For TCLP, the leach fluid employed for the leaching of non-volatile analytes is a function of the alkalinity of the solid phase of the sample. For SPLP, the leach fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. Two leachates may be generated: a) one for analysis of non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals and b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents. Following leaching, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8 μm fiber filter.

2.3 If the initial liquid phase of the sample (the filtrate) is miscible with the leachate, then they are combined, prepared, and analyzed together. If not miscible, the filtrate and leachate are analyzed separately and the results can be mathematically combined to yield a volume-weighted average concentration.

3.0 Definitions

3.1 Leachate: The TCLP solution generated after solids are tumbled with leaching fluid.

3.2 Filtrate: The liquid fraction of a sample that passes through a 0.6 to 0.8 μm fiber filter.

3.3 Final Leachate: The final solution generated from this procedure - either a leachate or a leachate combined with filtrate.

3.4 Leach Batch: A Leach Batch as a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same leaching procedure, reagents, and blank fluid type within the same time period. A minimum of one TCLP leach blank (LB) will be prepared with each TCLP leachate batch.

3.5 Percent Wet Solids: The fraction of a sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

4.0 Interferences

4.1 Oily samples may present unusual filtration and drying problems. For example, the oily sample may pre-maturely clog the filter used in the percent wet solids determination, causing a high-biased result for percent wet solids. Also, oils may contaminate the ZHEs and filtration apparatus. Therefore it is the laboratory's procedure to analyze all oil samples for total analysis. See section 1.7. For oily samples requiring metals analysis, a

suitable sub-lab will be procured. For oily wastes requiring semi-volatile organic analysis, the sample should be logged into the LIMS as a solid matrix for waste dilution extraction. For oily samples requiring volatile organic analysis, the sample should be logged into the LIMS as an aqueous matrix for analysis.

4.2 Samples containing free organic liquids (i.e., those with separable non-aqueous liquid phases) will be assumed to be 100% liquid and totals analysis will be performed to determine if the sample exceeds TCLP limits. See Section 0 on how these samples will be logged into the LIMS.

4.3 Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing leach blanks as described in the Section 9.4 and the individual determinative SOPs.

4.4 Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.

4.5 Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.

4.6 Over exposure of the sample to the environment will result in the loss of volatile components.

4.7 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements

5.2.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2.2 Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized.

CAUTION: Do not open equipment that has been pressurized until it has returned to ambient pressure.

5.2.3 A rotary agitation apparatus is used in this procedure. Certain samples may break the glass jars used in the procedure. For these samples, extra caution, including plastic or polyethylene over-wraps of the glass jar, may be necessary.

5.2.4 Secure tumbler and extraction apparatus before starting rotary agitation apparatus.

5.2.5 During sample rotation, pressure may build up inside the bottle. Periodic venting of the bottle will relieve pressure.

5.2.6 Due to the potential for ignition and/or flammability, do not attempt to dry non-aqueous liquid samples in an oven.

5.2.7 Do not attempt to manually stop a rotating piece of equipment. Keep all hanging objects, such as ties, hair, necklaces, etc., away from rotating equipment. Guards must be used when the apparatus is rotating to prevent loose clothing or limbs from getting caught.

5.2.8 Glass vials can break when the caps are being tightened. Cut resistant gloves should be worn whenever caps are being tightened.

5.2.9 When cleaning ZHE's a methanol rinse is used to remove any residual volatile compounds. After the rinse, the ZHE is put in an oven as a final cleaning procedure. It is very important that after the rinse the ZHE is allowed to dry for two hours in a fume hood before it is put in the oven. If this is not done then Methanol vapor will acuminate in the oven resulting in a hazard. This hazard can cause a fire, explosion, or methanol exposure to the face and/or eyes when the door to the oven is opened.

5.3 Primary Materials Used

The following is a list of materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not contain all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagent and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Nitric Acid, HNO_3	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 mg/m3 (Ceiling)	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and with greater exposures, it can cause burns that may result in permanent impairment of vision, even blindness.
Acetic Acid, Glacial	Corrosive Poison Flammable Liquid and Vapor	10 ppm (TWA)	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur. Can cause serious damage to skin, including redness, pain, and burns. Contact with eyes may cause severe damage followed by loss of sight.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Leach Vessels

6.1.1 For volatile analytes - zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Millipore YT3009OHW or equivalent (see Attachment 6).

6.1.2 For metals - either borosilicate glass jars (1 gallon, with Teflon lid inserts) or 2 L HDPE (Nalgene® or equivalent) bottles may be used.

6.1.3 For non-volatile organics - borosilicate glass jars must be used.

6.2 Vacuum Filtration Apparatus - Capable of 0 - 50 psi. For the filtering of leachates for metal analysis only as the apparatus is constructed of plastics. Cleaned by disassembling completely, washing with warm soapy water, rinsing with hot tap water, rinsing with DI water, and allowing to dry.

6.3 Stainless Steel Pressure Filtration Apparatus – 142 mm diameter. Capable of 0 - 50 psi. (See Attachment 7). For the filtering of leachates for semi-volatile organics and metals. For the percent wet solids determination. Cleaned by disassembling completely, washing with warm soapy water, rinsing with hot tap water, rinsing with DI water, rinsing with methanol, and allowing to dry.

6.4 Acid Washed, Low Metal, Borosilicate Glass Fiber Filters - 0.6 - 0.8 µm (Ahlstrom Grade 26). Certified for low metal content. 14.2 cm in diameter for pressure filter use. 4.7 cm in diameter for vacuum filter use. Glass fiber filters are fragile and should be handled with care.

6.5 Glass Fiber Filter Paper – 90 mm in diameter. For use in the ZHE.

6.6 Rotary Agitation Apparatus - Multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent (see Attachment 6). The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm. The RPM is checked annually.

6.7 Gas-Tight Syringes - 100mL capacity, Luer Lock Hamilton 0158330 or equivalent

6.8 Top Loading Balance - Capable of 0g – 4000g ± 0.01g. The balance accuracy is verified each day of use in accordance with SOP DV-QA-0014.

6.9 pH Meter and Probe - Capable of reading to the nearest 0.01 unit, and with automatic temperature compensation. Calibrated daily.

6.9.1 Always use fresh aliquots of the pH buffers

6.9.2 Always keep the probe immersed in pH electrode storage solution when not in use.

6.9.3 Rinse the probe with DI water and then immerse it in pH 4 buffer solution.

6.9.4 Press the "CAL" button on the pH meter and the light by "1" will come on.

6.9.5 Wait until the "Ready" light comes on. If the meter does not read 4.00 adjust the reading to 4.00 and then press ENTER. If the meter reads exactly 4.00, press the up arrow to change the reading to 4.01, and then press the down arrow to return the reading to 4.00 and then press ENTER.

6.9.6 Wait until the light by "2" comes on and then remove the probe from the pH 4 buffer solution and rinse it with DI water. Immerse the probe in the pH 10 buffer solution.

6.9.7 Wait until the "Ready" light comes on. If the meter does not read 10.00 adjust the reading to 10.00 and then press ENTER. If the meter reads 10.00, press the up arrow to change the reading to 10.01, and then press the down arrow to return the reading to 10.00 and then press ENTER.

6.9.8 Wait until the light by "SAMPLE" comes on and then remove the probe from the pH 10 buffer solution and rinse it with DI water. Immerse the probe in the pH 7 buffer solution.

6.9.9 Wait until the "Ready" light comes on. Record the reading in the pH meter calibration logbook. The meter is in control if it reads between 6.95 and 7.05.

6.9.10 If the electrode does not calibrate, check to make sure the electrode is completely filled with solution. If it is not, fill the electrode with reference electrode solution and try again. If this does not correct the problem, completely drain the solution out of the electrode and refill the electrode with fresh solution. If the probe still does not calibrate, the probe needs to be replaced.

6.10 Magnetic Stirrer/Hotplate and Stirring Bars – For use in the leach fluid determination.

6.11 VOA Vials – 20 mL, with caps and septa. For the storage of leachates for volatile organic compounds analysis.

6.12 Glass Jars - 1/2 to 1 gallon, with Teflon lid-inserts. For the storage of leachates for sem-volatile organic compounds analysis.

6.13 Nalgene Plastic Bottles – 250mL to 1 L. For the storage of leachates for metals analysis.

6.14 Pipette - Calibration checked daily per SOP DV-QA-0008.

6.15 Miscellaneous laboratory glassware and equipment.

6.16 Computer Software and Hardware

6.16.1 Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

6.17 Computer Software and Hardware

Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent Water – TestAmerica Denver has three ELGA Analytical water purification systems. The water coming from the ELGA system should be 17-18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026. Either water from the ELGA system or bottled HPLC grade water may be used in this procedure.

7.1.1 When water samples are logged for ZHE leaching, the leach blank is created using reagent water. For this application, the water must first be purged with nitrogen to remove any volatile compounds.

7.2 Hydrochloric Acid, 1 N - For use in leach fluid determination. Add approximately 800mL of reagent water to a 1 liter Class A graduated cylinder. Using a 100mL Class A graduated cylinder, measure out 83mL of concentrated reagent grade HCl and carefully add the acid to the reagent water. Dilute to 1 liter with reagent water. Transfer to a 1 liter glass bottle, cap and shake to mix well.

7.3 69%-70% Trace Grade Nitric Acid - For the preservation of final leachates prior to metals analysis. Purchased ready to use.

7.4 Sodium Hydroxide, 1 N - For use in TCLP Fluid #1. Using a Class A graduated cylinder add 2700mL of reagent water to a clean glass bottle. Using a Class A graduated cylinder, carefully add 300mL of 10 N NaOH. Cap and shake gently to mix well.

7.5 Glacial Acetic Acid – For use in TCLP Fluid #1 and #2. Concentrated, reagent grade liquid (HOAc).

7.6 pH Calibration Solutions - Buffered to a pH of 4, 7, and 10. Commercially available. Fresh buffer solution must be used each day of analysis.

7.7 TCLP Leaching Fluids

General Comments

The pH of both solutions listed below will be monitored daily before use by mixing fluid well and test with a calibrated pH meter.

The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid must be discarded and fresh extraction fluid prepared.

TCLP Fluid #1: For every liter of reagent water, carefully add 5.7 mL glacial acetic acid and 64.3 mL of 1 N NaOH. Cap and shake to mix well. When correctly prepared, the pH of this solution is 4.93 ± 0.05.

TCLP Fluid #2: For every liter of reagent water, carefully add 5.7 mL glacial acetic acid. When correctly prepared, the pH of this solution is 2.88 ± 0.05.

7.8 60/40 Sulfuric Acid / Nitric Acid - (60/40 weight percent mixture H2SO4/HNO3) For use in SPLP fluids. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.

7.9 SPLP Leaching Fluids

SPLP solutions are unbuffered. The pH of SPLP fluids will be checked daily prior to use. Mix well and check with a calibrated pH meter. If not within specifications, the fluid may be discarded and fresh fluid prepared or the fluid must be adjusted using additional acid or reagent water to achieve proper pH.

SPLP Fluid #1: This fluid is used for soils from a site that is east of the Mississippi River. Add approximately 60/40 weight percent mixture of sulfuric and nitric acids to approximately 20 liters of reagent water until the pH is 4.20 ± 0.05. Test with a calibrated pH meter. If the pH is not 4.20 ± 0.05, either add more acid or dilute by adding more reagent water.

SPLP Fluid #2: This fluid is used for soils from a site that is west of the Mississippi River. Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 5.00 ± 0.05. Test with a calibrated pH meter. If the pH is not 5.00 ± 0.05, either add more acid or dilute by adding more reagent water.

SPLP Fluid #3: This fluid is reagent water and is used for leaching of volatiles. Additionally, any cyanide-containing waste or soil is leached with fluid #3 because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas. This fluid is also used as the blank fluid for SPLP water samples.

7.10 Metals Spike Standards

7.10.1 TCLP Spike – Purchased ready to use in 2% nitric acid at the concentrations listed in Attachment 2.

7.10.2 ICP SPK 2A – Purchased ready to use in 2% nitric acid at the concentrations listed in Attachment 3.

7.10.3 ICP SPK 3A – Purchased ready to use in 2% nitric acid at the concentrations listed in Attachment 4.

7.10.4 Hg Daily Spk – Prepared in 1% nitric acid at the concentration listed in Attachment 5.

7.11 Methanol and methylene chloride - used to aid in cleaning oil contaminated equipment.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples being analyzed for non-volatile organic compounds should be collected and stored in glass containers with Teflon lid liners. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation.

8.2 Samples being analyzed for metals only can be collected in either glass or polyethylene containers.

8.3 When the waste is to be evaluated for volatile analytes, care should be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes. Water samples should be collected in Teflon lined septum capped vials. Soil samples should be collected in Teflon line 4 oz jars. Both water and soils should be collected with minimal headspace and stored at 4 ± 2 o C). Samples should be opened only immediately prior to leaching. A second container should be supplied for the percent solids determination.

8.4 Samples should be refrigerated to 4 ± 2°C unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

8.5 The physical state or states of the waste and the analytes of concern determine the minimum TCLP sample collection size. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing.

8.5.1 For multi-phasic samples containing between 0.5% and 10% solids, several kilograms of sample are required to complete the analyses.

8.5.2 The general minimal requirements when the samples are 100% solids include: 1 - 32 oz jar for semi-volatile organic analysis and metals, and 1 - 4 oz jar for volatile organic analysis. Low-density sample materials, such as rags or vegetation, will require larger volumes of sample.

8.5.3 For liquid samples (less than 0.5% solids), minimum requirements are 2 - 32 oz jars for semi-volatile organic analysis and metals, and 2 - 8 oz jars for volatile organic analysis. If volatile organic analysis is the only requested parameter, 2 separate jars are required.

8.5.4 If matrix spike or duplicate control samples are requested, additional sample volume is required.

8.5.5 If sufficient sample volumes were not received, analyses cannot be started and the project manager should be notified as soon as possible.

8.6 Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. If precipitation occurs upon addition of nitric acid, then no more acid shall be added and the leachate shall be analyzed as soon as possible.

8.7 All leachates for semivolatile organic analysis should be stored under refrigeration (4 ± 2 °C) until analyzed.

8.8 Leachates for volatile analysis must be stored under refrigeration (4 ± 2 °C) in VOA vials filled to eliminate all headspace.

8.9 Samples are subject to appropriate treatment within the following time periods:

HOLDING TIMES (DAYS)				
PARAMETER	**COLLECTION TO START OF LEACH**	**END OF TCLP TUMBLE TO PREPARATION**	**START OF TCLP LEACH OR SEMIVOLATILE PREP EXTRACTION TO ANALYSIS**	**TOTAL ELAPSED TIME**
Volatiles	14	N/A	14	28
Semi-Volatiles	14	7	40	61
Mercury	28	N/A	28	56
Other Metals	180	N/A	180	360

NOTE: The hold is the same for water and solids.

NOTE: The initial holding time is measured from date of collection to date TCLP leach started. (This should be the TCLP leach date in LIMS.) Semi-volatile method prep holding time is measured from the day leaching is complete to the start of method extraction. Subsequent analysis holding times are measured from the date extraction (TCLP or method prep) starts. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding holding times is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory limit. The Total Elapsed Time is to be used as guidance. If preps are initiated at the last possible moment of a holding time, the elapsed times may be exceeded.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments and special instructions to determine specific QC requirements that apply.

The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in DV-QA-003P, Quality Assurance Program.

Specific QC requirements for Federal programs, e.g., Department of Defense (DoD) Department of Energy (DoE), AFCEE etc., are desribed in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batching Samples - Groups of samples with visibly different bulk matrices (e.g., petroleum sludge and soil samples) must be batched separately for QC testing purposes. Samples that are 100% solids are batched separately from samples that are less than 0.5% solids.

9.3 A Leach Batch as a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same leaching procedure, reagents, and blank fluid type within the same time period. A minimum of one TCLP leach blank (Method Blank) will be prepared with each TCLP leachate batch.

9.4 TCLP Leach Blanks - A minimum of one blank (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch of samples leached that day in a particular vessel type. The leach blanks are generated in the same way as the samples (i.e., blanks will be tumbled and filtered with the samples). Leach fluid is tumbled with the samples in the same type of leach vessel (see Section 6.1) and filtered using the same filtration apparatus (see Section 6.2 and 6.3). Zero Headspace Extraction vessels are uniquely numbered. Each time a new batch is set up the blank should be rotated randomly to a different vessel to ensure all vessels are periodically checked. A vessel cannot be used in the leaching of more than 20 samples before it is used for the leaching of a blank. This is documented in the ZHE logbook.

9.5 Laboratory Control Sample (LCS) - A LCS is required with each batch of 20 or fewer samples. The LCS shall be created at the time of the preparative digestion or extraction by spiking an aliquot of the appropriate leach fluid used for that batch. Consult the

individual analysis SOPs for additional LCS guidance (i.e., spike amounts, spike levels, recovery criteria, etc.).

9.6 Matrix Spike (MS/MSD) - Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences. An MS/MSD pair is required with each batch.

9.7 MS/MSD samples will be spiked after final leachate generation at the time of preparative digestion or extraction. Spikes are not to be added prior to the TCLP leaching. For metals, matrix spikes are to be added before preservation with nitric acid.

9.8 Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.9 Corrective Actions

9.10 Consult the individual analysis SOPs for corrective action for blanks, LCSs, and MS/MSDs

10.0 Procedure

- One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

- Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

NOTE: The worksheets referred to in this SOP can be found in G:\OrgPrep\Read\TCLP Worksheets.

NOTE: See Attachment 12 for instructions on how to create batches in the LIMS system "TALS".

10.1 WORKSHEET 1, SECTION A SAMPLE DESCRIPTION – Enter data on Worksheet 1.

10.1.1 Preliminary TCLP evaluations (percent solids, particle size, selection of leach fluid, and fluid/leachate compatibility) are required to be done using a minimum of a 100 gram aliquot of sample. This aliquot may also undergo the actual TCLP or SPLP extraction for non-volatiles ONLY IF it has NOT been oven dried. If the solid portion is oven dried, a separate aliquot must be used for the actual leaching procedure.

10.1.2 Determine the total volume of leachate (solid phase leachate + liquid filtrate) that needs to be generated for analysis according to Table 2 below. Note that the volumes listed in Table 2 are the minimum volume required for one extraction and analysis. If possible, extra volume should be prepared for re-extractions and re-analysis. Additional volume for MS/MSD analysis should be provided for at least one sample per leach batch for every requested analysis. The samples will be leached at a 20X dilution (i.e. 100g of solids will generate 2000mL of leachate).

Table 2. Minimum Required Leachate Volume

Analysis	Required Leachate Volume for TCLP (mL)	Required Leachate Volume for SPLP (mL)
Volatiles	20 (3 x 20mL vials are normally supplied to provide volume for screening and re-analysis)	40 (3 x 40mL vials are normally supplied to provide volume for screening and re-analysis)
Semivolatiles	200	1000
Pesticides	100	1000
Herbicides	100	1000
Metals	100	100

10.1.3 Record the number of phases observed in the sample. It is common that when more than one container of multi-phasic materials is received from the field, each container will show different amounts of each phase.

10.1.4 If the sample has multiple phases and is received in more than one bottle, then the contents of each bottle should be combined in a single larger container prior to processing the sample further. However, the aliquot for volatile analysis should not be combined because that would expose the sample to headspace.

10.1.5 LINE A.1 - Record the visible presence of a solid material heavier than water. If the sample contains more than one solid phase (e.g., wood and sediment mixed with water), describe the different phases in an NCM.

10.1.6 LINE A.2 - Record the number of liquid phases observed in the sample according to apparent density. It may be impossible to distinguish apparent density if only one liquid phase is observed and there is no indication on the COC form. If this is the case, a small drop of the liquid can be added to a small amount of water to test the relative density.

NOTE: If the sample contains an oil layer, the client should be consulted. See Section 0 for guidance. Oily samples will be assumed to be 100% liquid and analysis for total concentrations of contaminants will be performed.

10.1.7 If the sample will obviously yield no free liquid when subjected to pressure filtration (i.e., it is 100% solid), then proceed to Section 10.3 (Leach Fluid Determination) for semi-volatile and metals analysis and proceed to Section 10.7 (ZHE Leaching Procedure) for volatile analysis. If only one jar was received, the ZHE procedure (Section 10.7) should be completed before proceeding to Section 10.3 for semi-volatile and metals analysis.

10.2 WORKSHEET 1, SECTION B – PERCENT SOLID PHASE

10.2.1 Percent Solids and ZHE Extractions - The ZHE filtration apparatus cannot accurately determine percent solids less than 5%. If an extraction is to be performed solely for volatile organic compounds and the percent solids

concentration is apparently greater than 5%, proceed to Section 10.7 (ZHE Extraction Procedure: Volatile Constituents). Otherwise, continue with the steps in this section. The aliquot of sample used here cannot be used again for the ZHE extraction.

10.2.2 Determine Type of Filtration Apparatus Needed –

- If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (<10%) or a liquid containing highly granular solids, either vacuum filtration or pressure filtration may be used
- If the sample is viscous (sludge or has high solids content), use pressure filtration.

10.2.3 LINE B.1 - Weight of Filter. Measure and record this value before loading the filter into the filter holder. Assemble the filtration apparatus. Use care when handling the 0.6 to 0.8 μm filter so as not to bend the filter or to contaminate it with trace amounts of oil from your hands.

10.2.4 LINE B.3.b – Tare Weight of filtration collection bottle. Select an appropriate container to collect the filtrate into. Weigh the empty container as the tare weight of the filtrate. A plastic bottle can be used if only metals analysis is requested, but a glass container should be used if any organic analyses are requested.

10.2.5 LINES B.2.a, B.2.b, and B.2.c - Weight of Subsample for Percent Solids Determination. Homogenize the sample. Weigh the full sample container and document this as the gross weight (Line B.2.a). Transfer the entire contents to the filtration apparatus, or if there is limited sample volume, care must be taken to transfer a representative sub sample by creating a well-mixed slurry before adding the sample to the filtration apparatus. Weigh the empty sample container with any residual sample and document this as the tare weight (Line B.2.b). The worksheet will then calculate the net weight of the sample used for the percent solids determination in Line B.2.c. If net weight is less than 100 g, an NCM should be written as the percent solids determination should be performed on an aliquot of at least 100 g.

10.2.6 Slowly apply gentle pressure or vacuum of 10 psi to the filtration apparatus. Allow the sample to filter until no additional liquid has passed through the filter during a 2-minute period.

10.2.7 Increase the pressure in 10-psi increments until a maximum of 50 psi is reached. Stop the filtration when no additional filtrate is generated within a 2-minute period. This may require many hours to complete. The sample should not be filtered for more than 24 hours to avoid evaporation of the filtrate and thus miscalculation of the percent wet solids. If the sample filtration is not complete in 24 hours, then the client should be contacted.

NOTE: Some samples will contain liquid material that does not filter. Do not attempt to filter the sample again by exchanging filters. Viscous liquids or solids that do not pass through the filter are classified as a solid.

10.2.8 LINE B.3.a – Gross Weight of Filtrate. Remove the filtrate collection bottle, weigh and record the gross weight.

10.2.9 LINE B.3.c – Net Weight of Filtrate. The worksheet will calculate the net weight of the filtrate.

10.2.10 LINE B.4 – Total Weight of Wet Solids. The worksheet will calculate the total weight of wet solids by subtracting the net weight of the filtrate (Line B.3.c) from the net weight of the subsample (Line B.2.c)

10.2.11 LINE B.5 – Weight Percent of Wet Solids. The worksheet will calculate the percentage of wet solids in the sample based on weight by dividing the Total Weight of Wet Solids (Line B.4) by the Net Weight of the Subsample (Line B.2.c) and multiplying by 100.

10.2.12 LINE B.3.d – Density of Filtrate. Determine the density of the aqueous phase of the filtrate using a calibrated pipette to measure the mass of 1 mL.

10.2.13 LINE B.7 - The worksheet will then calculate the volume of the aqueous phase of the filtrate.

10.2.14 LINE B.8 - If the filtrate is multi-phasic, pour the filtrate into a graduated cylinder. Measure and record the volume of the non-aqueous organic phase. If more than one organic phase is observed, enter "See Below" and provide a description at the bottom of Worksheet 1.

10.2.15 Retain the filtrate for use in Section 10.3.3. If the sample is logged for metals analysis only, the filtrate can be stored in a plastic container at room temperature. If the sample is logged for any organic analyses, then the filtrate must be stored refrigerated in a glass container. If the sample is logged for analysis of VOCs and a separate container was not received, then a small portion of this filtrate must be stored refrigerated in a VOA vial with no headspace and an NCM written.

10.2.16 If the Weight Percent of Wet Solids in Line B.5 is greater than 5.0%, and semi-volatile and metals analyses are required, proceed to section 10.3. If the Weight Percent of Wet Solids in Line B.5 is greater than 5.0% and volatile analysis is required, proceed to Section 10.7.3.

10.2.17 If the Weight Percent of Wet Solids in Line B.5 is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is equivalent to the final leachate. If the sample is logged for method 8260B, refer to Section 10.7.1 (ZHE leaching of 100% Liquid Samples) to generate leachate and blanks for volatile analysis. If the sample is logged for semi-volatiles and metals analysis, generate a leach blank by passing reagent water through a clean filtration apparatus similar to the apparatus used in the percent solids determination of the sample. Deliver the leachates and the associated blank to the appropriate departments along with all completed documentation.

10.2.18 If the Weight Percent of Wet Solids in Line B.5 is greater than or equal to 0.5% but less than 5.0% and it is noticed that a small amount of the aqueous filtrate is entrained in the wetting of the filter, proceed to Section 10.2.19 to complete the percent solids measurement on a dry-weight basis. If it is apparent to the analyst that the sample contains a significant amount of solids (>0.5%), the analyst can proceed to Section 10.2.19 to complete the percent solids measurement on a dry-weight basis to confirm this, or can proceed to Section 10.3

(Particle Size Reduction for Fluid Determination) for semi-volatile and metals analysis and Section 10.7.3 (ZHE Leaching of Samples Less than 100%, but greater than 0.5% Solids)

NOTE: If obviously oily (non-aqueous) material is entrained on the filter, do not dry the filter but instead proceed to Section 10.3 (Particle Size Reduction for Fluid Selection). Document in an NCM that the percent wet solids result is most likely biased high due to oily material trapped on the filter and that percent dry solids could not be performed.

10.2.19 LINE B.6 – Weight Percent of Dry Solids

NOTE: These steps are required only if it is noticed that a small amount of the filtrate is entrained in the wetting of the filter and the percent wet solids in Line B.5 is ≥ 0.5% and < 5.0 %.

- Remove the filter with the wet solids from the filtration apparatus. Take care to remove the entire filter. Often the filter will adhere to the apparatus.
- Dry the filter and solid phase at 100 ± 20 ° C. Record the temperature of the oven on the Worksheet 1. Allow the filter to dry in the oven for at least 10 minutes.
- Remove the filter from the oven and allow to cool.
- Weigh and record the gross dry weight (Line B.6.a). If it is less than 0.5%, then go to Section 10.2.17. If it is greater than 0.5%, repeat the drying step.
- Weigh and record the second gross dry weight (Line B.6.b). If the two weighings do not agree within 1%, perform additional drying and weighing until successive weights agree within 1%. Record the last two successive weights as Weight 1 and Weight 2 on Lines B.6.1 and B.6.2
- The Worksheet will calculate the Weight percent of Dry Solids in Line B.6.c using the equation in Section 11.5
- If the Weight Percent of Dry Solids is ≥ 0.5% and the sample will be extracted for non-volatile constituents, proceed to Section 10.3 (Particle Size Reduction for Fluid Selection) using a fresh wet portion of sample.
- If the Weight Percent of Dry Solids result is ≥ 0.5% and the sample will be extracted for volatile constituents, proceed to Section 10.7.3 (ZHE Extraction Procedure).
- If the Weight Percent of Dry Solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is the TCLP leachate. Go to Section 10.2.17.

10.3 WORKSHEET 2, SECTION C and D– LEACH FLUID DETERMINATION

- The sub-sample used for fluid selection must not have been subjected to the oven drying in Section 10.2.19.
- If the solid content is greater than or equal to 0.5% and if the sample is being analyzed for metals or non-volatile organic compounds, the type of leaching solution must be determined.
- Follow times, temperature, and particle size specified in this section as closely as possible. If reaction time between the acid solution and solid waste is too short or too long, the procedure may produce false pH readings.
- For SPLP, refer to Section 7.9 for fluid selection. The client must specify matrix type. Check special instructions, LIMS method, or the PM to determine if the sample is from east or west of the Mississippi River. Document on Line D.3, D.4, or D.5 the fluid type used and then proceed to Section 10.3.3 (Fluid Compatibility)

10.3.1 LINE C.1 – Particle Size Reduction for Fluid Determination

- The sub-sample used for fluid determination must consist of particles less than 1 mm in diameter (versus the less than 1 cm requirement for the material used in the actual leach). The method requires smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full leaching. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.
- Surface Area Exclusion – Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram. Weigh the particle, and estimate the surface area based on three dimensions assuming a cuboid shape. If the surface area is less than or equal to 3.1 cm^2 per gram, enter "No" on Line C.1 and prepare an NCM documenting the surface area per gram of sample.
- If the sample contains particles greater than 1 mm in diameter, crush, cut, or grind the solids to the required size. Enter "Yes" in Line C.1.
- Consult your supervisor and project manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick)

10.3.2 Determination of Appropriate Leach Fluid

- Calibrate the pH meter with fresh aliquots of buffer solution in accordance with the manual. See Section 6.9
- LINE C.2 – Calibrate a balance per DV-QA-0014 and record the balance ID.
- LINE C.3 - Weigh out a 5.0g ± 0.1g sub sample (less than 1mm particle size) of the solid phase into a 150mL beaker. This sub sample cannot have been subjected to the oven drying in Section 10.2.19
- LINE C.4 – Using a Class A graduated cylinder, add 96.5 ± 1.0mL of reagent water to the sub sample. Stir for 5 minutes on a stirrer set to 500rpm
- LINE C.5 – Measure and record the sample pH.
- If the pH is less than or equal to 5.0, use TCLP Fluid #1. Place an "X" in LINE D.1 and proceed to Section 10.3.3 (Fluid Compatibility)
- LINE C.6 - If the pH is greater than 5.0, add 3.5mL of 1 N HCl, using a calibrated pipette. Put a "X" on line C.6 and record the HCl Lot# and the Pipette ID in Lines C.6.a and C.6.b.
- LINE C.7 - Then heat at 50 ° C for 10 minutes while stirring. The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.
- LINE C.8 – Remove the sample from the hot plate. Measure and record the pH of the mixture.
- LINE D.1 and LINE D.2 – If the pH is less than or equal to 5.0, use Fluid #1 and enter an "X" on Line D.1. If the pH is greater than 5.0, use Fluid #2 and enter a "X" on Line D.2.

10.3.3 Determination of Filtrate/Leach Fluid Compatibility

- Skip this Section if the sample did not yield an initial filtrate from Section 10.2
- Place 5mL of the appropriate leaching fluid (determined in the previous step) into a 25mL vial. Add 5mL of the initial filtrate, cap and shake.
- LINE D.6

- If the phases are miscible, the initial filtrate and solid phase leachate will be physically recombined upon completion of the leachate generation. Enter a "X".
- If the phases are not miscible, enter "NO". The initial filtrate and the solid phase leachate will be prepared and analyzed separately and the results mathematically combined. See Section 11.12.

10.4 WORKSHEET 3, SECTION E– DETERMINATION OF SAMPLE SIZE FOR BOTTLE LEACH PROCEDURE

10.4.1 The aliquot used in the Preliminary Evaluation may be used for this procedure ONLY if it was not oven dried. If the sample is 100% solid or the preliminary aliquot was not oven dried proceed directly to Section 10.4.2 (Particle Size Reduction for Leaching). If the aliquot from the Preliminary Evaluation was oven dried then, using a fresh aliquot of sample, filter the sample to obtain wet solids and filtrate as described in Sections 10.2.2 through Section 10.2.15. The percent wet solids calculations may need to be repeated in order to correct for sub-sampling error. Then using this new aliquot, proceed to Section 10.4.2

10.4.2 LINE E.1 – Particle Size Reduction for Leaching

- Evaluate the solid portion of the sample for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the sample for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e, capable of passing though a 9.5 mm, 0.375 inch standard sieve). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram. (See Section 10.3.1)
- Consult your supervisor or manager when dealing with unusual sample matrices (e.g. wood, cloth, metal, brick). Scissors or tin snips may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks or other solids amenable to grinding can be reduced using a jaw crusher. Document in an NCM how unusual samples were handled. Note that size reduction to fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.

10.4.3 LINE E.2 -Calibration check a top-loading balance per DV-QA-0014. Document the Balance ID on Line E.2.

10.4.4 LINE E.3 - Weigh at least 100g of wet solids into an appropriate leach vessel. . See Section 6.1 for appropriate leach vessels. Document the weight of the sample to the nearest 1% on Line E.3. A minimum sample size of 100g is required. If there is insufficient sample, a NCM is needed. If full suite TCLP is requested, use 150g to generate sufficient leachate. Refer to Table 2 in Section 10.1.2

10.5 WORKSHEET 3, SECTION F– DETERMINATION OF AMOUNT OF LEACH FLUID FOR BOTTLE LEACH PROCEDURE

10.5.1 LINES F.1 through F.4 – Lot number of Leach fluid.

- Refer to Lines D.1 through D.5 for the correct leach fluid to use. Document the Lot number of the leach fluid used in Lines F.1 through F.4.

10.5.2 LINE F.5 – pH of Leach fluid.

- Record the pH of the Leach fluid. Check to make sure the pH of the fluid is still within the specifications in Section 7.7 and Section 7.9. If the pH of the buffered TCLP fluids is not within specifications, discard the fluid and make fresh fluid. If the pH of the un-buffered SPLP fluid is not within specifications, either discard the fluid or adjust the pH by adding more acid or more water. See Section 7.9

10.5.3 LINE F.6 – Volume of Leach Fluid

- The worksheet will calculate the volume of leach fluid to add to each sample based on the weight of the sample in Line E.3 using the formula in Section 11.7

- Prepare method blanks by filling similar leach vessels with the same leach fluid used for the samples.

10.6 WORKSHEET 3, SECTION G– RECORD OF BOTTLE LEACH

10.6.1 LINE G.1 – Check the rotator logbook to make sure the rotator has been checked in the past year to be rotating at 28-32 rpm. If the check has not been performed in the past year, count how many revolutions the rotator completes in one minute and document the check in the rotator log book. If the rotator does not rotate at a rate between 28-32 rpm, tag out the rotator until the rotator can be repaired.

10.6.2 LINE G.2 and G.3 - Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and rotate end-over-end for 16-20 hours. Record the leach start date and time on Line G.2. As agitation continues, pressure may build up within the bottle for some types of samples. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented fume hood to relieve any built-up pressure. Due to the higher acidity of TCLP Leach Fluid #2, it is more common for these samples to generate excess pressure. Record the leach stop date and time on Line G.3.

10.6.3 LINE G.4 – Temperature of Leach. The temperature of the room should be 23 ± 2°C. A data-logging device records the room temperature. After the leach has been stopped, record the maximum temperature in Line G.4.a and the minimum temperature in Line G.4.b.

10.6.4 LINE G.9 - Filter the leachate using vacuum or pressure filtration. For final filtration of the leachate, the glass fiber filter may be changed, if necessary to facilitate filtration. The entire leachate need not be filtered; however sufficient volume should be filtered to support the required analyses plus extra volume in case of re-extraction, re-digestion and MS/MSD. If needed, the leachate can be centrifuged to help facilitate filtration. Record the date and time the filtration is completed on G.9

10.6.5 LINE G.5 – pH of Leachate. Record the pH of the leachate.

10.6.6 If the sample contained no initial filtrate, (i.e the sample was 100% solids) the filtered leachate is defined as the final TCLP leachate. Proceed to Section 10.6.10

10.6.7 LINE G.6 Volume of Leachate. If the sample had an initial filtrate from Section 10.2, then measure the volume of leachate recovered so the leachate and the filtrate can be combined in the correct ratio. If the leachate contains an oil phase,

it must be separated and its volume recorded on Line G.6.a. The oil and the filtered leachate must be analyzed separately. If requested, the results can be mathematically re-combined. See Section 11.11 and Section 11.12.

10.6.8 LINE G.7 – Volume of initial filtrate for recombination. The worksheet will use the equation in Section 11.8 to calculate how much of the initial filtrate should be combined with the volume of leachate in Line G.6. Consult Line D.6 to determine if the initial filtrate is compatible to the leachate. If they are compatible, they are to be combined in the correct proportions and mixed well. The combined solution is defined as the TCLP leachate. If the initial filtrate and the leachate are not compatible, they are to be prepared and analyzed separately and the results mathematically combined. See Section 11.11 and Section 11.12. The leachate and the filtrate will have to be logged as separate samples in LIMS.

10.6.9 LINE G.8 – Volume of combined initial filtrate and leachate. The worksheet will calculate the volume of the combined filtrate and leachate using the equation in Section 0.

10.6.10 Leachates for organic analyses should be stored in glass containers at 4°C ± 2°C.

10.6.11 Leachates for metals analysis should be stored in poly bottles.

10.6.12 Prepare a MS/MSD sub-sample for metals testing following the steps below.

- Using a Class A graduated cylinder measure out 50mL of leachate in a bottle labeled for the MS and 50mL of leachate in a bottle labeled for the MSD.
- Add 0.5mL of the TCLP Spike described in Section 7.10.1.
- Add 0.5mL of the Prep Spike 2A described in Section 7.10.2
- Add 0.5mL of the Prep Spike 3A described in Section 7.10.3
- If mercury is requested, measure out additional 20mL aliquots for the MS and the MSD. Add 1mL of the mercury spike described in Section 7.10.4

10.6.13 Immediately preserve all leachates for metals by adding 1mL of nitric acid at a time until pH of 2 has been achieved. If after 5mL of acid has been added and the pH is still not 2, do not add more acid, but document final pH in an NCM. If a precipitate starts to form, immediately stop adding acid and document in an NCM.

10.7 WORKSHEET 4, ZHE PROCEDURE

- Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g. metals, pesticides, etc.).
- Due to the shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses

are minimized. Charge the ZHE with sample only once and do not open the divice until the final leachate has been collected. Do not allow the waste, the initial liquid phase, or the leachate to be exposed to the atmosphere any longer than necessary.

- The ZHE cannot accurately determine percent solids <5%. Go to Section 10.2 if it is apparent that the sample is less than 5% solids. If the sample is apparently greater than 5% solids, but less than 100% solids, go to Section 10.7.3. If the sample is 100% solids, go to Section 10.7.3. If the sample is 100% liquid, proceed to Section 10.7.1

10.7.1 ZHE Leaching of 100% Liquid Samples

10.7.1.1 This procedure is to be used for samples determined to be 100% liquid per Section 10.2.

10.7.1.2 Place o-rings and wiper seals on the ZHE piston. Moisten the o-rings with nitrogen-purged reagent water and place the piston in the ZHE body. Adjust the ZHE piston in the ZHE body to the appropriate height in order to contain the sample. At least 80mL of sample should be used. If the piston is 2cm below the top of the cylinder, this will be enough volume for 80mL.

10.7.1.3 Assemble the top flange and test the inlet/outlet valve to make sure it opens and closes easily. If the valve is sticking, run DI water through the valve and work the o-ring back and forth until it loosens up. This will prevent the o-rings from tearing and will prevent the valve from leaking and reduce the frequency of o-ring replacement.

10.7.1.4 LINE H.2. - Place the sample in the ZHE body. Place the ZHE body on the ZHE base. Place the top flange on top of the ZHE body and secure tightly. Record the ZHE used on Line H.2

10.7.1.5 With the inlet/outlet valve closed, pressurize the ZHE until you hear the piston move upwards.

10.7.1.6 LINE I.6 - Slowly open the inlet/outlet valve to release any headspace. Once liquid appears through the inlet/outlet valve, close the valve and attach a clean gas-tight syringe. Slowly open the valve and collect the filtrate. This filtrate is the final leachate. After all leachate has been collected, remove the syringe from the ZHE and document the filtration completion date and time on Line I.6

10.7.1.7 Transfer the leachate from the syringe to 20mL voa vials. Care should be taken not to leave any headspace in the vials. The entire leachate need not be transferred, but three 20mL vials should be filled to allow for re-analysis and screening.

10.7.1.8 Generate a leach blank using reagent water in the same manner as above. Document in the ZHE logbook which ZHEs were used for samples and which ZHEs were used for method blanks. A ZHE cannot be used for a sample if it has not been used as a method blank in the past 20 uses.

10.7.2 ZHE Leaching of 100% Solid Samples

10.7.2.1 Consult Worksheet 1 and examine the sample. If the sample appears to be different from the preliminary information found on the worksheet, consult your supervisor. If the preliminary evaluations indicate the need for particle size reduction, crush, cut, or grind the sample so that all particles are less than 1 cm in size as measured with a ruler. (Do not sieve the sample). Size reduction is not required if the sample surface

area is greater than or equal to 3.1 cm2 per gram. Weigh the particle, and estimate the surface area based on three dimensions assuming a cubiod shape. If particle size reduction was necessary, document this on Worksheet 4 as an observation.

NOTE: To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4 °C) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate.

10.7.2.2 Place o-rings and wiper seals on the ZHE piston. Moisten the o-rings with nitrogen-purged water and place the piston in the ZHE body. Adjust the ZHE piston in the ZHE body to the appropriate height in order to contain the required sample. At least 5 grams of sample will be needed.

10.7.2.3 LINE H.1 – Calibration check the balance per DV-QA-0014 and record the balance ID.

10.7.2.4 LINE H.2 – Record the ID of the ZHE

10.7.2.5 LINE H.3 - Place the assembled ZHE piston and body on the balance and tare. Place 5 g ± 0.1 g of the sample in the ZHE body. Record the mass on Line H.3. If less than 5 g is used an NCM should be written to document the deviation from the procedure.

10.7.2.6 Place the ZHE body on the ZHE base.

10.7.2.7 Assemble the top flange and test the inlet/outlet valve to make sure it opens and closes easily. If the valve is sticking, run nitrogen-purged water through the valve and work the o-ring back and forth until it loosens up. This will prevent the o-rings from tearing. It will also prevent the valve from leaking and reduce the frequency of o-ring replacement. Place the assembled top flange on top of the body, secure tightly. See Attachment 6.

10.7.2.8 Close the liquid inlet/outlet valve. Pressurize the ZHE until you hear the seals set.

10.7.2.9 Slowly open the liquid inlet/outlet valve to release all headspace. Then depressurize the ZHE using the pressure release valve.

10.7.2.10 LINE H.7 – The worksheet will calculate the required volume of leach fluid to add to the ZHE in Line H.7 using the formula in Section 11.7 Load a clean gas-tight syringe with TCLP Fluid #1 or SPLP Fluid #3 depending on the analysis requested. Adjust the volume of the fluid in the syringe to the volume in Line H.7, which is 20 times the mass of the wet solids in the ZHE body. (e.g. If 5 g of wet solids were used, then 100 mL of fluid would be required). Attach the syringe to the inlet/outlet valve of the ZHE. Press down on the syringe plunger, forcing the fluid into the ZHE. While still pressing on the plunger, close the inlet/outlet valve. Remove the syringe. Observe the valve opening for any leaks. If it is leaking, the valve o-rings will need to be replaced.

10.7.2.11 LINES H.7a, H.7.b, and H.7.c – Record the lot number and the pH of the fluid used.

10.7.2.12 LINE I.1.a and LINE I.1.b - Pressurize the ZHE to at least 15 psi. Record the on I.1.a. Let the ZHE sit for at least 15 minutes. Check to

make sure gauge still reads 15 psi. Record this check on Line I.1.b. Check the inlet/outlet valve for signs of leakage. If the ZHE shows signs of leakage or the pressure gauge indicates leakage, then the ZHE will be removed from service and repaired. Start the procedure over using either a new ZHE or the repaired ZHE and a fresh aliquot of sample. All repairs and maintenance performed on ZHEs are documented in the ZHE log book. If the ZHE has held pressure and there is no sign of leakage from the inlet/outlet valve then proceed on.

10.7.2.13 If the pressure gauge indicates a leak, place the ZHE in a bucket of water and watch for air bubbles. If bubbles are coming from the o-ring at the bottom of the cylinder, clean or replace the o-ring and wipe any contamination from the o-ring grooves. If bubbles are coming from the base pressure relief valve, try seating the valve with your finger or mark the base as having a leaky valve and set aside for repair.

10.7.2.14 Generate a leach blank by assembling and loading a ZHE with the same leach fluid used for the samples. Record in the ZHE logbook which ZHEs were used for the leaching of samples and which ZHEs were used for the leaching of blanks. A ZHE cannot be used for the leaching of a sample if it has not been used for the leaching of a blank in the past 20 leaches.

10.7.2.15 LINE I.2 and LINE I.3- Secure the ZHE in a rotatary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. Record the start time and the end time on Lines I.2 and I.3

10.7.2.16 LINE I.4.a and LINE I.4.b – A data-logging device records the room temperature. The maximum and minimum temperature during the leach is recorded.

10.7.2.17 LINE I.5 - Remove the ZHE from the rotary agitator and check that the ZHE is still under pressure. Do this by quickly opening and closing the pressure release valve and listening for the release of gas. If the ZHE is not under pressure, then the procedure must be repeated using a fresh aliquot of sample and the ZHE should be taken out of service for maintenance and repair.

10.7.2.18 LINE I.6 – Attach a clean gas- Attach a clean gas-tight syringe to the inlet/outlet valve. The plunger of the syringe should be completely compressed before being attached to the ZHE. Slowly open the inlet/outlet valve and allow the leachate to enter the syringe. If necessary the ZHE can be pressurized to facilitate the collection of the leachate, but care should be taken not to cause effervescence. After all leachate has been collected, remove the syringe from the ZHE and document the volume of leachate recovered. If less than 80% of the initial fluid added to the ZHE is recovered as leachate, write an NCM to document this. The sample may need to be re-leached.

10.7.2.19 LINE I.6.a - If the leachate is bi-phasic record the volume of the non-aqueous phase on Line I.6.a. Document in an NCM. The oil phase may need to be analyzed separately and results mathematically re-combined.

10.7.2.20 Transfer the leachate from the syringe to three 20 mL voa vials. Care should be taken not to leave any headspace in the vials. The entire leachate need not be transferred.

10.7.2.21 LINE I.8 - Record the filtration completion date and time.

10.7.2.22 Label all leachates and deliver the leachates and associated blank to the GC/MS Volatiles department along with all completed documentation. The leachates should be stored at 4 ± 2 °C.

10.7.3 ZHE Leaching of Samples Less than 100%, but greater then 0.5% Solids

10.7.3.1 Consult Worksheet 1 and examine the sample. If the sample appears to be different from the preliminary information found on the worksheet, consult your supervisor. If the preliminary evaluations indicate the need for particle size reduction, crush, cut, or grind the sample so that all particles are less than 1 cm in size as measured with a ruler. (Do not sieve the sample). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm2 per gram. Weigh the particle, and estimate the surface area based on three dimensions assuming a cubiod shape. If particle size reduction was necessary, document this on Worksheet 4 as an observation.

NOTE: To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4 °C) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate.

10.7.3.2 Place o-rings and wiper seals on the ZHE piston. Moisten the o-rings with nitrogen-purged water and place the piston in the ZHE body. Adjust the ZHE piston in the ZHE body to the appropriate height in order to contain the required sample.

10.7.3.3 LINE H.1 – Calibration check the balance per DV-QA-0014 and record the balance ID.

10.7.3.4 LINE H.2 – Record the ID of the ZHE

10.7.3.5 LINE H.3 - Place the assembled ZHE piston and body on the balance and tare. If possible, transfer the entire contents of the sample container to the ZHE body. Use the equation in Section 11.10 to estimate how much sample to place into the ZHE. Record the mass. **NOTE:** The ZHE has a maximum capacity of 500mL. Therefore you cannot load more than 25g of solids into the ZHE or else you will not be able to add the appropriate volume of leach fluid.

10.7.3.6 Place the ZHE body on the ZHE base.

10.7.3.7 Assemble the top flange and test the inlet/outlet valve to make sure it opens and closes easily. If the valve is sticking, run nitrogen-purged water through the valve and work the o-ring back and forth until it loosens up. This will prevent the o-rings from tearing. It will prevent the valve from leaking and reduce the frequency of o-ring replacement. Place the assembled top flange on top of the body, secure tightly. See Attachment 6.

10.7.3.8 LINE H.4.b - Weigh 1 to 2 empty gas-tight syringes. Record their combined weight as the tare weight on Line H.4.b. More syringes may be needed if the sample contains a low percent solids value. See Line B.5.

10.7.3.9 Close the liquid inlet/outlet valve. Pressurize the ZHE until you hear the seals set.

10.7.3.10 Slowly open the liquid inlet/outlet valve to release all headspace. Once liquid starts to come out of the valve, immediately close the valve and attach one of the tared syringes.

10.7.3.11 Open the valve again and collect the filtrate. Once syringe is filled, close the valve and attach an additional tared syringe and repeat until no more filtrate is collected. Increase the pressure of the ZHE 10 psi at a time up to 50 psi until no more filtrate emerges from the ZHE after 2 minutes.

10.7.3.12 LINE H.4.a - Weigh the full syringes and record their combined weight as the gross weight.

10.7.3.13 LINE H.4.c – The worksheet will then calculate the net weight of the filtrate using the equation in Section 11.2

10.7.3.14 LINE H.5 – Record the volume of the filtrate by reading the graduations on the syringe(s).

10.7.3.15 Transfer the filtrate into vials with no headspace. Label and store the filtrate refrigerated 4 ± 2°C.

10.7.3.16 LINE H.6 – The worksheet will then calculate the total grams of wet solids remaining in the ZHE using the formula in Section 11.3

10.7.3.17 LINE H.8- The worksheet will then calculate the percent wet solids using the formula in Section 11.4

10.7.3.18 Follow steps in Section 10.7.2.10 through 10.7.2.21

10.7.3.19 If the initial filtrate from Section 10.7.3.15 is miscible with the leachate (as determined in Section 10.3.3), the leachate and the initial filtrate are directly recombined in the correct proportions.

10.7.3.19.1 For samples containing greater than 5% wet solids, the percent wet solids value from the ZHE filtration process should be used to determine the volume of filtrate to re-combine with the leachate. Therefore use the value in Line I.7.b. This approach is required since the percent solids value determined using the pressure filter may differ from the percent solids value determined using the ZHE due to sample variability or differences in the filtration apparatus.

10.7.3.19.2 For samples containing less than 5% wet solids, the percent wet solids value from the pressure or vacuum filtration process should be used to determine the volume of the filtrate to re-combine with the leachate. Therefore use the value in Line I.7.a. This approach is required because the ZHE is not appropriate to determine the percent solids of a sample if the percent solids are less than 5%.

10.7.3.20 If the individual phases are NOT compatible, they are to be collected, prepped and analyzed separately. If the individual phases are analyzed separately, the results can be mathematically recombined by using the recombination calculation in Section 11.12.

10.7.3.21 Label all leachates and deliver the leachates and associated blank to the GC/MS Volatiles department along with all completed documentation. The leachates should be stored at 4 ± 2 °C.

11.0 Calculations and Data Reduction

11.1 Weight of Subsample (Line B.2.c)

(Net Weight, B.2.c) = (Gross Weight, B.2.a) – (Tare Weight, B.2.b)

11.2 Weight of Filtrate (Line B.3.c) or (Line H.4.c)

(Net Weight, B.3.c) = (Gross Weight, B.3.a) – (Tare Weight, B.3.b)

(Net Weight, H.4.c) = (Gross Weight, H.4.a) – (Tare Weight, H.4.b)

11.3 Total Weight of Wet Solids (Line B.4) or (Line H.6)

(Wet Solids, B.4) = (Weight of Subsample, B.2.c) – (Weight of Filtrate, B.3.c)

(Wet Solids, H.6) = (Weight of Subsample, H.3) – (Weight of Filtrate, H.4.c)

11.4 Weight Percent Wet Solids (Line B.5) or (Line H.8)

(% Wet Solids, B.5) = 100 x (Wet Solids, B.4) / (Weight of Subsample, B.2.c)

(% Wet Solids, H.8) = 100 x (Wet Solids, H.6) / (Weight of Subsample, H.3)

11.5 Weight Percent Dry Solids (Line B.6.c)

$$\text{Weight percent dry solids, B.6.c)} = 100 \times \frac{\text{(Gross dry weight2, B.6.b.)} - \text{(Weight of filter, B.1.)}}{\text{(Weight of subsample, B.2.c.)}}$$

11.6 Volume of Aqueous Filtrate (Line B.7)

(Vol. of Filtrate B.7) = (Weight of Filtrate, B.3.c) / (Density of Filtrate, B.3.d)

11.7 Volume of Fluid for Bottle Leach (Line F.6) or ZHE Leach (H.7)

(Vol. Fluid, F.6) = (Weight of Wet Solids, E.3) x 20

(Vol. Fluid, H.7) = (Weight of Wet Solids, H.6) x 20

11.8 Volume of Initial Filtrate to recombine with Leachate (Line G.7), (Line I.7.a) or (Line I.7.b)

$$(\text{Vol. of Inital Filtrate for Recombination, G.7}) = \frac{(\text{Solids Leachated, E.3})}{(\text{Tot. Wet Solids, B.4})} \times \frac{(\text{LeachateRecovered, G.6})}{(\text{FluidAdded, F.6})} \times (\text{InitialFiltrat, B.7})$$

$$(\text{Vol. of Inital Filtrate for Recombination, I.7.a}) = \frac{(\text{Wet Solids in ZHE, H.6})}{(\text{Tot. Wet Solids, B.4})} \times \frac{(\text{LeachateRecovered, I.6})}{(\text{FluidAdded, H.7})} \times (\text{InitialFiltrat, B.7})$$

$$(\text{Vol. of Inital Filtrate for Recombination, I.7.b}) = \frac{(\text{Weight of Filtrate, H.4.c})}{(\text{Fluid Added, H.7})} \times (\text{Volume of Leachate Recovered, I.6})$$

11.9 Combined initial filtrate and leachate (Line G.8)

(Combined Filtrate & Leachate, G.8) = (Vol of Leachate, G.6)+(Vol of Filtrate, G.7)

11.10 Weight of Sample to Charge to ZHE

(Weight of Sample) = 100 X [20g/ (%wet solids, B.5)]

11.11 Reporting Conventions for Multi-phase Leachates:

11.11.1 If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.

11.11.2 If both phases are "ND," not detected, the recombined result is "ND," and the reporting limit is calculated from the reporting limit for each phase.

11.11.3 If one phase is "ND" and the other phase has a positive result, use the reporting limit for the "ND" phase and the positive value for the other phase to calculate the combined result. The combined reporting limit is based on the reporting limit for both phases. If the combined result is less than the combined reporting limit, then supply a footnote to indicate that "a positive result was detected below the calculated detection limit."

11.11.4 Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

11.11.5 For limits and significant figures, consult the appropriate analytical methods

11.12 Mathematical recombination of analytical results:

$$\text{Final Analyte Concentration} = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

V_1 = total volume of the initial filtrate phase (L).

C_1 = analyte concentration in initial filtrate phase (mg/L).

V_2 = volume of the theoretical solid phase leachate (L).

C_2 = analyte concentration in solid phase leachate (mg/L).

12.0 Training Requirements

12.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

13.1 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, "Waste Management and Pollution Prevention", of the Enviornmental Health and Safety Manual, and DV-HS-001P, "Waste Management Plan."

14.2 The following waste streams are produce when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Solid waste (post extraction) – Excess Solid Samples - Waste Stream S
- Aqueous waste (post extraction) - Aqueous Waste from TCLP - Waste Stream T
- Buffer 4 - Aqueous Waste from TCLP - Waste Stream T
- Buffers 7 and 10 - Aqueous Waste from TCLP - Waste Stream T
- Methanol waste - Flammable Solvent - Waste Stream C
- Methylene chloride waste - Waste Stream B

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References

15.1 Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update I.

15.2 Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, November 1992, SW-846 Proposed Update II.

15.3 Related Documents

15.3.1 Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990.

15.3.2 Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990.

15.4 Technical Background Document and Response to Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April, 1989.

16.0 Method Modifications

Item	Method	Modification
1	SW846 1311	Section 7.1 of the source method states that the sample aliquot used for the preliminary evaluation "...may not actually undergo TCLP extraction." Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 further indicates that if the sample was subjected to filtration (i.e., < 100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). This SOP states that samples which have been subjected to the oven drying step may not be used for TCLP extraction because solid phase degradation may result upon heating.
3	SW846 1311	Percent Solids Determination. Section 7.1.2 of the source method indicates that "if the percent wet solids is ≥ 0.5% and it is noticed that a small amount of the filtrate is entrained in wetting of the filter" that the filter should be oven dried to determine percent dry solids ". Drying of oil or organic matrices can both be hazardous and inappropriate. Additionally, it may be impossible to achieve a constant weight when performing this step. Due to safety concerns, this SOP states that if obviously oily or heavy organic matrices are entrained on the filter, the filter is not oven dried.
4	SW846 1311	Section 7.2.13 of the source method provides no guidance as to how to determine filtrate and leach fluid compatibility. Therefore, this SOP has incorporated a miscibility test into the Preliminary Determinations section.
5	SW846 1311	Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that Fluid #1 must be used if the pH is less than or equal to 5.0.

6	SW846 1311	Section 8.2 of the source method states "A matrix spike shall be performed for each waste type..." and "A minimum of one matrix spike must be analyzed for each analytical batch." Further, Section 8.2.3 of the source method also states "The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist." The TestAmerica Laboratory Quality Manual is designed to address the performance monitoring of analytical methodology through the LCS program. A minimum of one MS and MSD will be prepared for each TCLP leachate batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results have immediate bearing only on the specific sample spiked and not all samples in the batch.

17.0 Attachments

Attachment 1: Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)
Attachment 2: Metals TCLP Spike
Attachment 3: Metals ICP SPK 2A
Attachment 4: Metals ICP SPK 3A
Attachment 5: Metals Hg Daily Spk
Attachment 6: Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)
Attachment 7: Pressure Filtration Device
Attachment 8: TCLP Worksheet No. 1: Sample Description
Attachment 9: TCLP Worksheet No. 2: Selection of Leach Fluid
Attachment 10: TCLP/SPLP Worksheet No. 3: Bottle Leach Procedure
Attachment 11: TCLP Worksheet No. 4: ZHE Leach
Attachment 12: Instructions for Batching in LIMS

18.0 Revision History

- Revision 3, dated 12, March 2010
    - Added instructions to Section 5 to ensure guards are used on any rotating machinery to prevent injury.
    - Added details to Section 6.9 and through the procedure to more closely document the proper procedure for pH probe calibration, including instructions to use fresh calibration fluid every day.
    - Added Section 6.16 Computer Software and Hardware
    - Revised Section 7 and Section 10 to include the use of nitrogen-purged water for the leach blanks for water volatile samples.
    - Corrected Section 7.4 to properly document the preparation of 1N Sodium Hydroxide.

- Reformatted Section 10 of the procedure to more closely follow and reference the worksheets.
- Reformatted the Worksheets to allow the analyst to document up to 10 samples on a single page. Combined TCLP Worksheet #2 and SPLP Worksheet #2.
- Added guidance in Section 10.7 on how to lubricate the o-rings in the valve of the ZHE to prevent leakage. Also added information on how to detect leaks in the ZHE apparatus.
- Removed what was labeled as Attachment 7: US EPA Memoradum #35. Clarification was received from Methods Information Communications Exchange Service that this memo was not meant to be used for policy or rulemaking purposes. It is however still the laboratory's procedure to analyze all oils for total analysis to prevent contamination of the filtering apparatus.
- Added instructions for batching in TALS – TestAmerica LIMS System.

- Revision 2, dated 13 March 2008
  - Updated formatting.
  - Added criteria that requires extraction fluid pH to be re-prepped if the pH does not meet criteria.

- Revision 1, dated 21 November 2006
  - Company name changed from STL Denver to TestAmerica Denver.
  - Clarified and standardized language throughout the SOP to consistently refer to samples instead of wastes, and leachates instead of extracts.
  - Added clarification throughout the SOP to help analysts in the reading and understanding of the procedure.
  - Section 1.9 – This section was eliminated. This section described how to evaluate results from a bottle leach for volatile organic analysis. It is not standard operating procedure for TestAmerica Denver to analyze a bottle leachate for volatiles. It is the laboratory's standard procedure to generate a leachate for volatile analysis using a Zero Headspace Extractor.
  - Section 3– Added "Leach Batch", "Filtrate", and "Final Leachate" to the list of definitions.
  - Sections 4.1 ,4.2, and 11.5.1.3– Added clarification on how non-aqueous oily samples will be processed.
  - Section 5.2.8– Added this section to identify a safety hazard.
  - Section 6 – Removed Tedlar bags from the list of equipment and supplies as these are not used at TestAmerica Denver
  - Sections 6.1.1, 6.2, and 6.3 – Added information on how these items are cleaned.

- Sections 7.2.1, 7.4, 7.7.2, 7.7.3, 7.9.2, and 7.9.3 – Added clarification on how these reagents are made up in the laboratory.
- Section 7 – Deleted reference to 50% Nitric Acid and 1 N Nitric Acid as these reagents are not currently used. Added 69%-70% Trace Grade Nitric Acid.
- Section 9.3 – Added the requirement that all leach blanks will be rotated in extraction vessels similar to the extraction vessels used in the leaching of the samples.
- Section 9.5 and 9.6 – Changed these sections to reference analytical SOPs for corrective actions for QC outliers.
- Section 11 – This section was divided into the current Sections 11, 12, and 13 in order to make the SOP easier to read.
- Section 10.2.4 – Table 2 was corrected and edited to include the minimum leachate volume for SPLP analysis.
- Section 10.2.5 – Sample Description. This section was revised to document the current procedure for handling multi-phasic samples that are received in more than one container. The old procedure called for the analyst to make a relative measurement of each phase in order to take a representative aliquot for percent solids determination. This new procedure calls for the analyst to combine all sample into one container and perform the percent solids determination on the entire sample. This eliminates a possible error.
- Section 10.2.6.7 – This density check of the filtrate was added in order to help the analyst identify non-aqueous filtrates and to provide our clients with more observations and information about their samples.
- Throughout the SOP guidance is given to the analysts on how method blanks will be generated.
- The worksheets are now EXCEL spreadsheets in which the calculations are performed using formulas in locked cells. This was done to eliminate the possibility of a mathematical error. Many additional details and sample observations were added to the worksheets as well in order to improve the quality and of the raw data.
- Section 10.4.6.15 gives direction for the recombination of filtrates and leachates from a ZHE. The old procedure directed the analyst to use the percent wet solids determined by pressure filtration in the calculation of the appropriate volume of filtrate and leachate to combine. This new section directs the analyst to use percent solids determined from the ZHE filtration when a sample is greater than 5% solid.
- Flow Charts were revised to add details and more closely match the procedure.
- Figure 4 was added.
- Tables 4, 5, 6, and 7 were added.

Attachment 1.
Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)

Contaminant	mg/L
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresols	200.0
m-Cresols	200.0
p-Cresols	200.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor (& epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

Attachment 2
Metals TCLP Spike

Component	Concentration (ug/mL)
Silver	100
Arsenic	300
Barium	1000
Cadmium	100
Chromium	500
Copper	200
Lead	500
Selenium	100
Zinc	200

Attachment 3.
Metals ICP SPK 2A

Component	Concentration (ug/mL)
Boron	100
Molybdenum	100
Antimony	50
Silicon	1000
Tin	200
Titanium	100
Zirconium	50

Attachment 4.
Metals ICP SPK 3A

Component	Concentration (ug/mL)
Silver	5
Aluminum	200
Arsenic	100
Barium	200
Beryllium	5
Calcium	5000
Cadmium	10
Cobalt	50
Chromium	20
Copper	25
Iron	100
Potassium	5000
Lithium	100
Magnesium	5000
Manganese	50
Sodium	5000
Nickel	50
Phosphorus	1000
Lead	50
Selenium	200
Strontium	100
Thorium	100
Thallium	200
Uranium	200
Vanadium	50
Zinc	50
Bismuth	200

Attachment 5.

Metals Hg Daily Spk

Component	Concentration (mg/L)
Mercury	0.1

Attachment 6.
Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

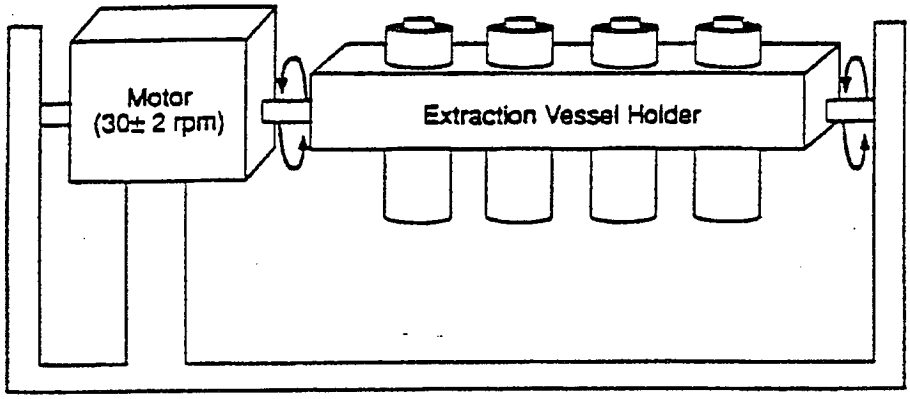
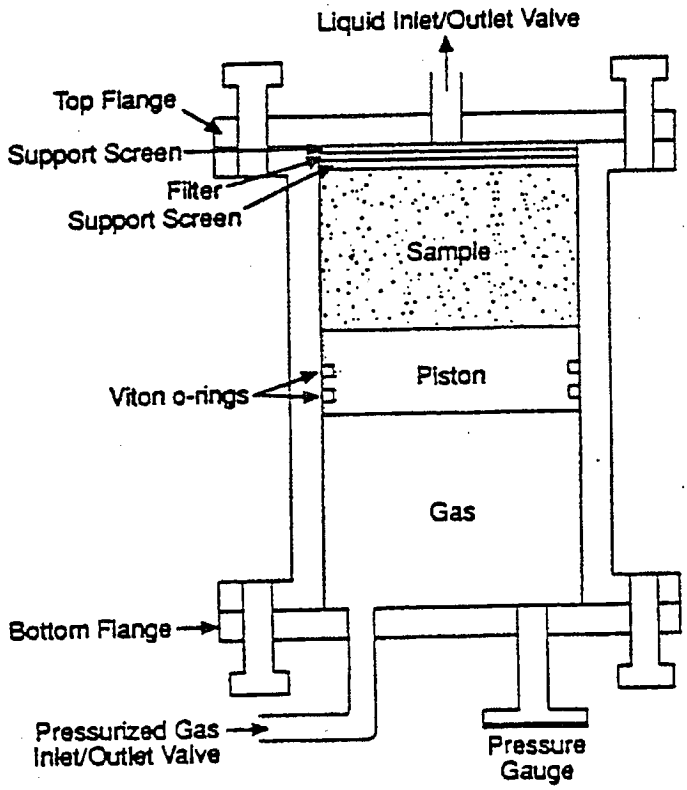
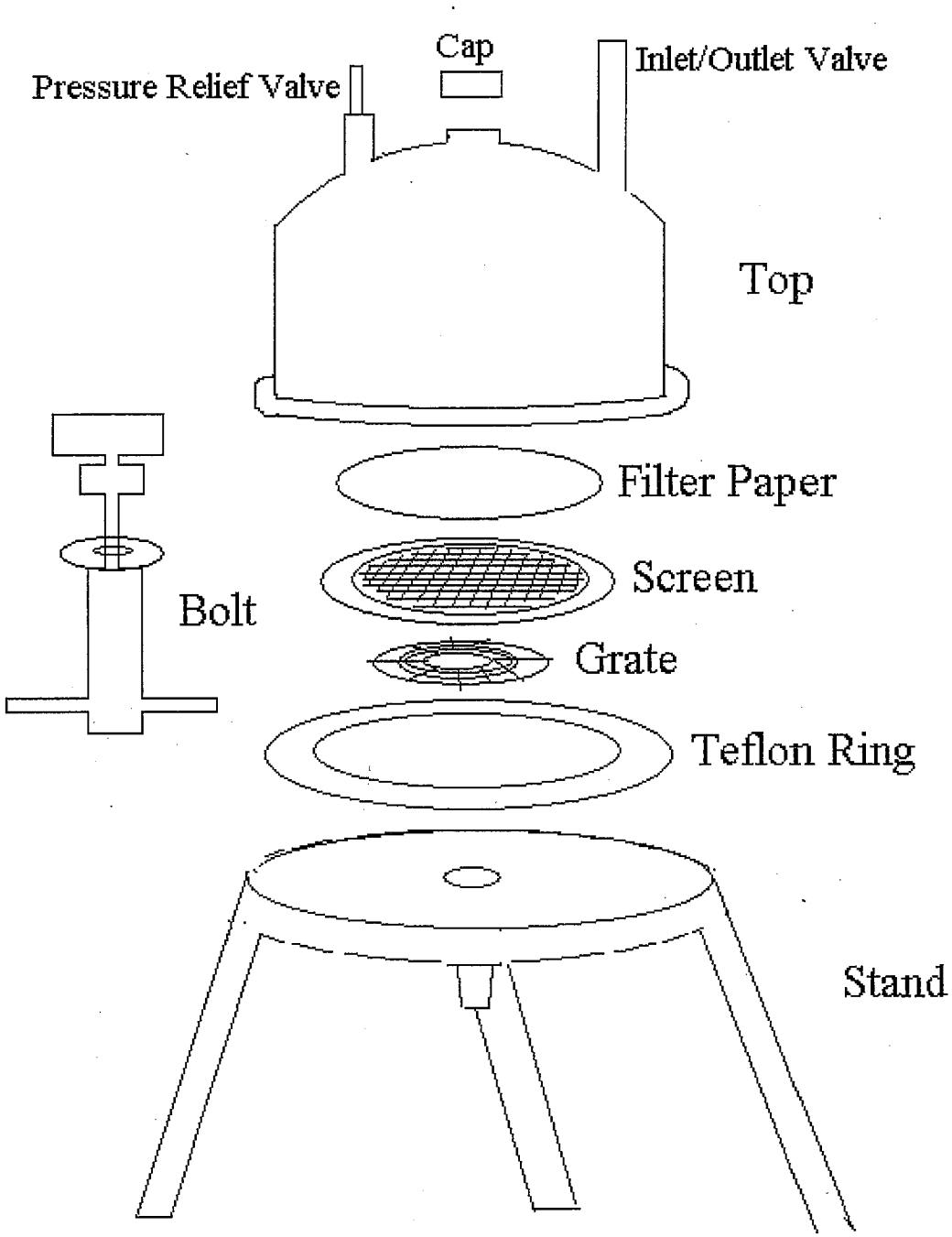


Figure 1. Rotary Agitation Apparatus



Attachment 7
Pressure Filtration Device



Attachment 8
Worksheet No. 1

Attachment 8
Worksheet No. 1
TCLP

Analyst:

Date:

DV-IP-0012

TCLP/SPLP Worksheet No. 1
Sample Description

TestAmerica
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Laboratory Sample No.																	
Field Sample No.																	
A. Sample Description																	
Number of phases																	
1. Solid																	
2. Liquid																	
a. lighter than water																	
b. water																	
c. heavier than water																	
B. Percent Solid Phase																	
Balance ID																	
1. Weight of filter (g)																	
2. Weight of subsample																	
a. gross weight (g)																	
b. tare weight (g)																	
c. net weight (g)																	
d. density of filtrate (g/mL)																	
4. Total weight wet solids (g)																	
5. Weight percent solids (wet) (%)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
6. Weight percent solids (dry)																	
Oven Temp (°C)																	
a. gross dry weight 1 (g)																	
b. gross dry weight 2 (g)																	
c. percent dry solids (%)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
7. Vol. of initial aqueous filtrate (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
8. Vol. of initial organic filtrate (mL)																	

Comments:

(Net Weight of Subsample, B.2.a) = (gross weight, B.2.a) - (tare weight, B.2.b)

(Net Weight of Filtrate, B.3.a) = (gross weight, B.3.a) - (tare weight, B.3.b)

(Total weight wet solids, B.4) = (Weight subsample, B.2.a) - (Weight filtrate, B.3.c)

(%Weight percent wet solids, B.5.)= 100 X (Total weight wet solids, B.4.)/(%Weight of subsample, B.2.c.)

(Weight percent dry solids, B.6.c.) = 100 X [Gross dry weight(B.6.b.)] - [Weight of filter, B.1.] / (Weight of subsample, B.2.c.)

(Vol of initial filtrate, B.7)= (%Weight of filtrate, B.3.c) / (Density of filtrate, B.3.d)

Attachment 9
TCLP Worksheet No. 2

Analyst: 0

DV-IP-0012
TCLP Worksheet No. 2
Selection of Leach Fluid

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Laboratory Sample No.										
Field Sample No.										
C. Leach Fluid Determination - Does not apply to determination of volatile organic components or SPLP.										
1. Particle size reduction? (<1mm) Yes/No										
2. Balance ID										
3. Sample weight, 5.0 +/- 0.1g										
4. Volume of water, X if 96.5 +/- 1.0 mL added										
5. Initial pH (after 5 min. mixing time)										
6. If pH > 5.0, then add 3.5 mL 1N HCl and mark "X"										
a. HCL Lot# used										
b. Pipette ID										
7. X if heated and held at 50 C for ten minutes										
8. Secondary pH (at room temp.)										
D. Selection of Leach Fluid										
1. X if pH from C.5. or C.8. is < 5.0, use Leach Fluid #1										
2. X if pH from C.8. is > 5.0, use Leach Fluid #2										
3. SPLP Fluid 1: Soils- East of the Mississippi River; Wastewates; or Wastewaters										
4. SPLP Fluid 2: Soils- West of Mississippi River										
5. SPLP Fluid 3: If VOCs or Cyanide containing wastes.										
6. X if filtrate and fluid are miscible										

COMMENTS:

Attachment 10
TCLP Worksheet No. 3

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Analyst: 0

DV-IP-0012
TCLP/SPLP Worksheet No. 3
Bottle Leach Procedure for Metals and Semi-Volatile Organic Components

Laboratory Sample No.																								
Field Sample No.																								
E. Determination of Sample Size																								
1. Particle size reduction? Yes/no																								
2. Balance ID																								
3. Weight of wet solids after filtration (g)																								
F. Determination of Amount of Leach Fluid																								
1. TCLP Fluid 1 Lot #																								
2. TCLP Fluid 2 Lot #																								
3. SPLP 1 (East) Lot #																								
4. SPLP 2 (West) Lot #																								
5. pH of leach fluid																								
6. Vol of Fluid = wet solids x 20 (mL)																								
G. Record of Leach - leach period is 16 to 20 hours																								
1. Rotator has been checked in the past year to be rotating at 28-32 RPM?																								
2. Leach start date and time																								
3. Leach stop date and time																								
4. Room temperature																								
a. Temp Min (°C)																								
b. Temp Max (°C)																								
5. pH of leachate																								
6. Volume of leachate (mL)																								
a. Oil recovered from leachate (mL)																								
7. Volume of initial filtrate for recombination (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
8. Combined initial filtrate + leachate (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
9. Date and time filtration finished																								

(Volume of Fluid, F:4) = (Weight of wet solids, E:3) x 20 (Vol. of Initial Filtrate for Recombination, G:7) = (Solids Leachate, E:7) x (Leachate Volume, G:6) / (Tot. Wet Solids, F:4) (Fluid Added, F:5) (Initial Fil trat, B:7)

(Vol. of Combined Filtrate and Leachate, G:8) = (Vol of Leachate, G:6) + (Vol of Filtrate, G:7)

COMMENTS:

Attachment 11
TCLP Worksheet No. 4

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Analyst: 0

DV-IP-0012
TCLP/SPLP Worksheet No. 4
ZHE Leach

Laboratory Sample No.										
Field Sample No.										
H. Determination of Amount of Leach Fluid										
1. Balance ID										
2. ZHE vessel number										
3. Weight of material added to ZHE (g)	5									
4. Weight of filtrate in syringe										
a. gross weight (g)										
b. tare weight (g)										
c. net weight (g)	0	0	0	0	0	0	0	0	0	0
5. Volume of filtrate in syringe (mL)										
6. Wet solids in ZHE (g)	5									
7. Weight of fluid to add (g)	100	0	0	0	0	0	0	0	0	0
a. TCLP Fluid 1 Lot #										
b. SPLP Fluid 3 Lot #										
c. pH of Blank Fluid										
8. Percent Wet Solids (%)	100	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
I. Record of ZHE Leach - the Leach period is 16 to 20 hours										
1. Leak Check										
a. Reading #1 (psi)										
b. Reading #2 (psi)										
2. Leach start date & time										
3. Leach stop date & time										
4. Room temperature										
a. Min (°C)										
b. Max (°C)										
5. **X** if still under positive pressure after leaching										
6. Volume of leachate recovered (mL)										
a. Volume of oil recovered after leaching										
7. Vol. of initial aqueous filtrate for recombination										
a. Calcualated from Worksheet 1 (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
b. Calculated from Worksheet 4 (mL)	0.0	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
8. Filtration completed date & time										

(Net Weight of Filtrate, H.4.c)= (Gross weight, H.4.a.) - (Tare weight, H.4.b.)

(Wet Solids in ZHE, H.6) = (Weight of material added to ZHE, H.3.) - (net weight of filtrate, H.4.c.)

(Weight of Fluid to add, H.7.)= (Wet Solids in ZHE, H.6) X 20

(Percent Wet Solids, H.8.)= 100 X [(Wet Solids in ZHE, H.6) / (Weight of material added to ZHE, H.3)]

(Vol of Filtrate for recombination, I.7.b)= (Vol of Leachate Recvd, I.6) X (Weight of Filtrate, H.4.c) / (Vol Fluid Added, H.7.)

(Vol of Filtrate for recombination, I.7.a)= [(Wet Solids in ZHE, H.6.) / (Tot Wet Solids, B.4)] X [(Vol Leachate Recvd, I.6) / (Vol Fluid Add, H.7)] X Vol Filtrate, B.7)

Comments:

Attachment 12

How to Batch TCLP and SPLP:

1311_T (Organics) **1311T_Hg** (Mercury) **1311T_M** (Metals)	**1312_E** (Organics) **1312_E_Hg** (Mercury) **1312_E_M** (Metals)	**1312_W** (Organic) **1312_W_Hg** (Mercury) **1312_W_M** (Metals)	**1311_Z** (ZHE)

Overview

The pre-prep methods listed above are specific to the analytes requested, but it is not necessary to batch them all separately. Above, the methods are placed in boxes to indicate which methods can be batched together, with one exception: *SPLP 8260s will be logged with 1312_E or 1312_W, which is the same leach method used for organic bottle preps so not all 1312_E can be batched together and not all 1312_W can be batched together.*

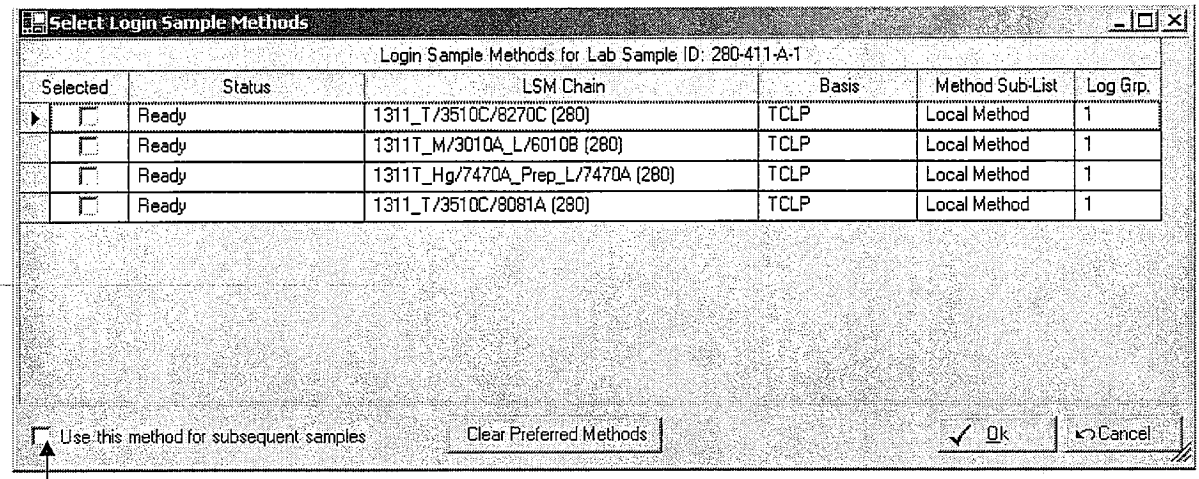
If one sample is logged in for TCLP 8270C, TCLP 8081B, TCLP 8260, TCLP 6010B, and TCLP 7470A, the sample will show up on the Organic Extractions backlog 5 times for TCLP, (once for each analytical method), and twice for 3510C.

Record Status	Status	A-Status	HT Expires	Rush	Method	A-Method	Job Number	Lab Sample ID	Container Matrix
Active	Ready	Active	1/23/2010 11:59	☐	1311_T	8081A	280-J411-1	280-411-1	Solid
Active	Ready	Active	1/23/2010 11:59 PM	☐	1311_T	8270C	280-J411-1	280-411-1	Solid
Active	Ready	Active	1/23/2010 11:59	☐	1311_Z	8260B	280-J411-1	280-411-1	Solid
Active	Ready	Active	2/6/2010 11:59	☐	1311T_Hg	7470A	280-J411-1	280-411-1	Solid
Active	Ready	Active	7/8/2010 11:59	☐	1311T_M	6010B	280-J411-1	280-411-1	Solid
Active	Wait	Active	1/23/2010 11:59	☐	3510C	8081A	280-J411-1	280-411-1	TCLP Leach
Active	Wait	Active	1/23/2010 11:59	☐	3510C	8270C	280-J411-1	280-411-1	TCLP Leach

For the sample above, we would leach the sample in a glass bottle for the organics and metals and we would also do a ZHE leach. Therefore there will be 2 leach batches.

Simple Steps

1. Run the OP - Extractions Not Batched backlog. This backlog is sorted by Extraction Method so all of the TCLP and SPLP methods should be grouped together at the top. You can select only the TCLP and SPLP methods by clicking on them while holding the Control or Shift key. Then check the Selected Only box at the top and hit print.
2. Pull the samples from the walk-in cooler and take custody of the samples.
3. Use the TCLP spreadsheets in EXCEL to determine blank fluid for each sample. Once the leach fluid has been determined, you will know what samples can be batched together. You can not put samples with different leach fluids in the same batch.
4. Open Analyst Desktop and select Create Batch from Scratch
5. Select any leach method that is logged on the sample.
6. Your Batch Notes will appear, but we are not going to use the Batch Notes. Instead all of our data will be recorded in the TCLP spreadsheets in EXCEL.
7. Scan your samples into the batch. If your samples are logged in for more than one of the leach methods, a window will appear called "Select Login Sample Methods".



8. Select all LSM Chains that include the leach preps. If all of the samples that you want to batch together are logged in for the same methods, you can click the box in the lower left-hand corner that says "Use this method for subsequent samples". Then click "OK".
   NOTE: Be careful when clicking the "Use this method for subsequent samples" box. If you click this and the subsequent samples have more methods than the ones listed in the LSM box, they will not be included in the batch. You can check this in Step 10 below and fix it there if there is something wrong.
9. If your batch is for TCLP Fluid #1 or SPLP East Fluid, create a "LB" for the Leach Blank. If your batch is for TCLP Fluid #2 or SPLP_West Fluid, create a "LB2" for the Leach Blank. If your batch is for water TCLP samples, water SPLP samples, or SPLP ZHE samples, then you are using reagent water as your blank fluid and create a "LB3" for your Leach Blank There will be no other QC here at this point unless a client has requested MS/MSD on a sample. If that is the case, add the MS/MSD to the leach batch, but it does not get spiked before the leach.
10. Go to the Sample List tab. Here you will see that if the sample was logged in for more than one method chain, the sample will be listed here multiple times – one for each method chain. It is a good idea to check your backlog against the Sample List tab to make sure that all of your method chains that were listed on the backlog are in the batch. If they are not, right click on the sample and click on "Select LSM" to add the missing tests into the batch.
11. Go to the Worksheet tab. We won't be using the fields here to record our data because the calculations are not locked. We will use the TCLP spreadsheet instead. **But we will have to enter the Leach Fluid type or else our spreadsheets will not get into the raw data. Scroll all the way over to the right and enter "T1", "T2" "T3", "SE", "SW" or "S3".**
12. We will use a different status to indicate where the samples are.
    a. A status of "Batched" means the blank fluid determination is done.
    b. A status of "1st Level Review" means the samples are tumbling.
    c. A status of "2nd Level Review" means that the samples have completed the leachate and have been filtered.
13. Save the TCLP worksheet in EXCEL. Then print it to pdf and save it in the same directory as the EXCEL file. **When you print it to pdf, be sure to select "Entire Workbook" so that all worksheets will be in the pdf.**
14. Go into the TALS batch and click on the documents button. Attach the pdf to the TALS batch.

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Title: Acid digestion of Aqueous Samples for Analysis by ICP- MS [SW-846 3005A, 3020A, 3050B and EPA Method 200.8.]

Approvals (Signature/Date):

Doug Gomer 8/30/10 — DG 8/30/10 ~~9/27/10~~
Doug Gomer — Date
Metals Supervisor

Adam W. Alban 29 Aug 10
Adam Alban — Date
Health & Safety Manager / Coordinator

Karen Kuoppala 8-30-10
Karen Kuoppala — Date
Quality Assurance Manager

Robert C Hanisch 8/30/10
Robert C. Hanisch — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This procedure describes the preparation of aqueous samples for the analysis of metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) using EPA Method 200.8, and SW-846 Methods 3005A, 3020A, and 3050B.

1.2 Aqueous samples also include aqueous equipment rinse blanks for soil sampling. In some cases, where the associated soil samples require the SW-846 Method 3050B, Section 7.5, optional treatment to improve solubility and recovery of Sb, Ag, and Sn. The client may require that the aqueous equipment blank receive the same treatment. Refer to section 10.13 for this prep.

1.3 The applicability of each of these preparation protocols to specific analytes is detailed in Table I. Additional elements may be analyzed following digestion by these protocols, provided that the method performance criteria specified in Section 12.0 of this SOP are met.

1.4 This SOP provides procedures applicable to the preparation of dissolved, total recoverable, potentially dissolved, and total metallic elements in ground water, aqueous samples, aqueous sludges, aqueous wastes, aqueous air sampling media, and leachates/extracts. This SOP is not applicable to samples that contain or consist of oil or other immiscible organic solvents.

NOTE: Samples that are known to be immiscible with water, e.g., contain or consist of oil or other immiscible organic solvents, are subcontracted to other labs that have the capability of handling such samples. If during the preparation process it is discovered that the sample is immiscible with water or is biphasic, then the analyst notifies the Group Leader and Project Manager, who can subcontract the samples to a laboratory with the capability to handle the oil matrix.

1.5 SW-846 Method 3005A is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP-MS.

1.6 EPA Method 200.8 Section 11.2 is used to prepare surface water, and domestic and industrial waste samples for total recoverable and dissolved metals.

1.7 SW-846 Method 3020A is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP-MS. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

1.8 The following table lists the sample preparation methods that are covered in this SOP and the specific section of this SOP for each preparation method. Prepared samples are analyzed by inductively-coupled plasma-mass spectrometry (ICP-MS).

PREPARATION METHOD	SOP SECTION	DETERMINATIVE METHOD	SOP #
Method 6020 – 3020A	10.10	ICP-MS	DV-MT-0002
Method 6020 – 3005A	10.11	ICP-MS	DV-MT-0002
Method 200.8 – 200.8 Total Rec.	10.12	ICP-MS	DV-MT-0002
Method 200.8 – Dissolved	10.12	ICP-MS	DV-MT-0002
Method 3050B-Special Sb Prep	10.13	ICP-MS	DV-MT-0002

2.0 Summary of Method

2.1 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals.

This preparation method is used for total recoverable and dissolved metals analysis by ICP-MS method 6020. A representative aliquot of sample is heated with nitric acid and substantially reduced in volume. The digestate is diluted to volume and then filtered (if necessary).

2.2 Method 3020A, Acid Digestion of Aqueous Samples and Extracts for Total Metals.

This preparation method is used for total metals analysis by ICP-MS method 6020. A representative aliquot of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. The digestate is diluted to volume and then filtered (if necessary).

2.3 Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry

This preparation method is used for metals analysis by ICP-MS method 200.8. A representative aliquot of sample is refluxed with nitric and hydrochloric acids. The digestate is diluted to volume and then filtered (if necessary).

3.0 Definitions

Additional definitions of terms used in this SOP may be found in the glossary of the QAM.

- Dissolved Metals: The concentration of metals determined in a sample after the sample is filtered through a 0.45-µm membrane (Method 3005A). (The sample is acidified after filtration).
- Total Metals: The concentration of metals determined in an unfiltered sample following digestion (Method 3020A).
- Total Recoverable Metals: The concentration of metals determined in an unfiltered sample following treatment with hot, dilute mineral acid (Method 200.8)-(Method 3005 A).
- Potentially Dissolved Metals: An acidified sample is filtered between 8- 96 hours following acidification and the filtrate is digested using Method 3005A.

4.0 Interferences

4.1 There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include the following: metallic or metal-containing labware (e.g., latex gloves coated with talc, which contains high levels of zinc), containers,

impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix C for additional contamination control guidelines.

4.3 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not soluble in acids. If physical interferences are present, they should be documented.

4.4 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.5 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared.

4.6 Specific analytical interferences are discussed in the ICP-MS determinative method SOP, i.e., DV-MT-0002.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, and this document.

5.1 Specific Safety Concerns

5.1.1 Samples that contain high concentrations of carbonates or organic material, or samples that are at elevated pH can react violently when acids are added.

5.1.2 The digestion solution must be cooled sufficiently before adding hydrogen peroxide (H2O2) to avoid a reaction and possible violent effervescence, or boiling over of the digestion solution.

5.1.3 Care must be taken when handling the digestion tubes. The tubes may become very hot during the digestion procedure. Allow the tubes to cool before attempting to touch the sample digestate.

5.1.4 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm (TWA)	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

- Digestion block, with adjustable heating, capable of maintaining a sample temperature of 85 - 95 °C.
- Thermometer that covers a temperature range of at least 80 - 110 °C.
- Centrifugation equipment (if desired method of removing particulate material is centrifugation).

6.2 Supplies

- Disposable digestion tubes, with volume accuracy verified to ± 3% gravimetrically prior to use.
- Watch glasses, ribbed or equivalent, or disposable digestion tube covers.
- Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters, No. 6973-2504, for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add or subtract analytes.
- Syringes or equivalent filtration apparatus.
- Graduated cylinder or equivalent capable of measuring 50 mL to ±3% accuracy.
- Repipetors or suitable reagent dispensers.
- Calibrated automatic pipettes with pipette tips or Class A glass volumetric pipettes.
- Class A volumetric flasks.
- pH indicator strips (pH range 0 - 6).
- Plastic digestate storage bottles.

7.0 Reagents and Standards

7.1 Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative method SOP, DV-MT-0002.

7.2 Laboratory control sample (LCS), and matrix spike and matrix spike duplicate (MS/MSD) spike solutions are purchased as custom TAL Denver solutions. Standards are logged into the Standards Log database and are assigned unique identification numbers that can be used to access traceability information. The Standards Log identification numbers are recorded on the metals prep bench sheet.

7.2.1 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. These plastic bottles may be stored in a glass jar.

7.2.2 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.2.3 See Table II for the list of spiking levels. A volume of 0.1 mL of working spike solution is added to the 50-mL final sample volume.

7.3 Nitric Acid (HNO_3), concentrated, trace-metal grade or better.

NOTE: When preparing diluted acids, always add acid to water. If the water is added to the acid, the sudden increase in temperature may cause splashing.

7.4 Nitric Acid, 1:1

Dilute concentrated HNO_3 with an equal volume of reagent water.

7.5 30% Hydrogen Peroxide (H2O2), ultra pure grade.

7.6 Hydrochloric Acid (HCl), concentrated, trace metal grade or better.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE Or Glass	500 mLs	HNO_3, pH < 2;	180 Days	40 CFR Part 136.3
Soils	Glass	4 oz	Cool 4 ± 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TAL Denver policy DV-QA-003P, Quality Control Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TAL Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Table IV provides a summary of quality control requirements, including type, frequency, acceptance criteria, and corrective action.

9.3 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12.2 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.4 Preparation Batch

A preparation batch is a group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, a matrix spike (MS), and a matrix spike duplicate (MSD). In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify samples for the MS/MSD pair, then the batch may contain multiple MS/MSD pairs to accommodate client requests. Clients may also request a duplicate LCS (LCSD). In cases where the client has not provided sufficient sample to prepare an MS and MSD, an LCS and LCSD will be prepared instead.

9.5 Sample Count

Laboratory-generated QC samples (method blanks, LCSs) are not included in the sample count for determining the size of a preparation batch. The MS and MSD are usually not included in the sample count.

NOTE: For samples prepared under any AFCEE QAPP, all MSs and MSDs are included in the sample count.

9.6 Method Blank (MB)

The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. When samples are filtered in the laboratory for determination of dissolved metals, then the blank is filtered using a filter of the same type that was used for the samples. The performance of the filtration process must be acknowledged on the Supplemental Metals Prep Sheet. One method blank must be processed with each preparation batch. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false-positive data.

Acceptance Criteria:

The method blank should not contain any analyte of interest at or above the reporting limit (RL) or at or above 10% of the measured concentration of that analyte in associated samples, whichever is higher. In other words, the sample result must be a minimum of 10 times higher than the blank contamination level. An exception is made for common laboratory contaminants (see section 16.1.1).

Corrective Action:

If the method blank does not meet the criteria, the blank and all associated samples in the batch must be re-digested and reanalyzed.

9.7 Laboratory Control Sample (LCS)

One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. When samples are filtered in the laboratory for determination of dissolved metals, then the LCS is filtered using a filter of the same type that was used for the samples. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

Acceptance Criteria:

LCS recovery control limits are set at ± 3 standard deviations about the historical mean. These limits must not be wider than 85 - 115 % recovery for Method 200.8, or 80 - 120 % for Method 6020. The control limits are maintained in the LIMS system.

Corrective Action:

If the LCS % recovery falls outside of the control limits for any analyte, that analyte is judged to be out of control. All associated samples must be reprocessed for analysis. One possible exception is a recovery for a given element above the upper control limit with no detection for the same element in the samples. This latter case must be explained in the case narrative.

9.8 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. One MS/MSD pair must be processed for each preparation batch. Some client programs require a 10 % MS/MSD analysis frequency. If insufficient sample is available to process an MS/MSD pair, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria.

The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks, equipment blanks, or rinse blanks cannot be used for MS/MSD analysis.

Acceptance Criteria:

The recovery for each analyte must fall within established limits. The relative percent difference (RPD) between the MS and MSD must be less than or equal to the established RPD limit. If any analyte recovery or relative percent difference (RPD) between the MS and MSD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS.

Corrective Action:

If the recovery of the LCS is outside limits, then corrective action must be taken. Corrective action will include re-preparation and reanalysis of the batch. If MS results fail to meet control limits, but the LCS results are within limits, then samples do not require re-preparation and reanalysis unless the results indicate that a spiking error may have occurred.

9.9 Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede the SOP requirements. Quality Assurance Summaries (QASs) are developed to address these requirements.

10.0 Procedure

Sample Preparation

10.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file.

10.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.3 All samples are to be checked out of Sample Control with the chain of custody documentation filled out completely.

10.4 Proper sample identification is extremely important in any preparation procedure. Labeling of beakers, digestion tubes, and bottles must be done in a manner to ensure connection with the proper sample.

10.5 Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating sample preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous, but it appears to be more like a waste (biphasic, oil, sludge-like, organic liquid, lots of sediment, etc.), then contact the project manager and the laboratory group leader for further instructions. It may be necessary to subcontract these samples to a laboratory with the capability to digest organic matrices.

NOTE: TAL Denver has not implemented digestion methods for water-immiscible organic matrices, e.g., oils. Samples that are known to be incompatible with TAL Denver digestion techniques are typically subcontracted to other laboratories.

10.6 If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab, data review, and reporting groups.

10.7 Guidelines are provided in Appendix C on procedures to minimize contamination of samples and standards.

10.8 Aqueous Sample Preparation Setup

The following setup procedure must be followed for all aqueous samples prior to performing the specific digestion procedure:

10.8.1 If the sample pH has already been verified and documented at sample receipt, the following steps may be omitted.

10.8.1.1 Measure the sample pH with pH paper using a separate aliquot of sample.

10.8.1.2 If the pH>2 for a sample requiring acidic preservation, record the pH in the Metals Prep Log and record the anomaly using Clouseau.

10.8.1.3 Add 1-2 mLs of conc. HNO_3 to the sample. Replace the lid and mix the sample.

10.8.1.4 Recheck the pH of the sample. If the pH<2, record the volume of acid added in the Metals Prep Log. If the pH>2, repeat 10.8.1.3 until pH<2. Record the volume of HNO_3 added.

10.8.1.5 Allow the sample to sit for 8-16 hours following acidification.

10.8.1.6 After 8-16 hours, recheck the pH of the sample. If the pH<2, proceed with the appropriate digestion procedure. Note the date/time of this pH recheck in the Metals Prep Log.

10.8.1.7 If after 8-16 hours the pH>2, repeat steps 10.8.1.3 through 10.8.1.6 until the pH remains <2 following the 8-16 hour period.

Note: Acid must be added at least 24 hours before analysis.

10.8.2 Select the unfiltered fraction for a total or total recoverable analysis or the filtered fraction for a dissolved analysis. For SPLP select the proper sample leachates.

NOTE: Samples requiring dissolved metals determination are either filtered and preserved in the field or are filtered and preserved by the laboratory as soon as possible after receiving the samples. When filtered in the laboratory, the filtration and preservation are recorded in the Laboratory Sample Filtration and Preservation Logbook, including the preservative type and lot number. Filter acceptability is demonstrated by using filters of the same type to filter samples and batch QC samples when preparation batches include samples that were filtered in the laboratory. The results of the analysis of the batch QC samples are used to demonstrate that the filtration process neither adds nor subtracts target analytes from samples.

10.8.3 Mix the sample by shaking the container.

10.8.4 Measure and transfer 50 mL of the sample into a digestion tube. When using calibrated digestion tubes, pour the sample into the tube to the 50-mL mark. Record the lot number of the digestion tubes in TAL's

10.8.5 Measure two extra aliquots of the sample that is selected for the MS/MSD analysis. Spike each aliquot with 0.1 mL of each spiking solution (see Table 2). Record the standards and pipette identifications in TAL's

10.8.6 Measure and transfer 50 mL of reagent water into a digestion tube for the method blank. If a determination of dissolved metals is requested, use filtered reagent water for the method blank.

10.8.7 Measure and transfer 50 mL of reagent water into a digestion tube for the LCS and add 0.1 mL of spiking solution (see Table 2). Record the standards and pipette identifications in TAL's If determination of dissolved metals is requested (preparation method 3005A), and one or more samples were filtered in the laboratory, then filter the LCS using a filter of the same type that was used to filter the sample(s).

10.9 Proceed to the appropriate Section of this SOP for the desired preparation method as follows:

Preparation Method*	SOP Section	Analytical Method
3020A Total Metals	10.10	Method 6020
3005A Total Recoverable	10.11	Method 6020
3005A Dissolved Metals	10.11	Method 6020
200.8 Total Recoverable Metals	10.12	Method 200.8
200.8 Dissolved Metals	10.14	Method 200.8
3050B Special Sb prep	10.15	Method 3050B

10.10 Method 3020A - Preparation for Total Metals Analysis by ICP-MS Method 6020

10.10.1 To the sample in a digestion tube, add 1.5 mL of concentrated HNO_3.

10.10.2 Heat at 90 - 95 °C until the volume is reduced to approximately 5 mL. Record the start and stop times and the Hot Block temperature in TAL's

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared and reanalyzed.

10.10.3 Allow the digestion tube to cool in a fume hood.

10.10.4 Add 1.5 mL of concentrated HNO3. Replace the cover and reflux gently.

10.10.5 Continue heating, adding additional acid as necessary in 1-2 mL

increments to ensure a complete digestion. Record the start and stop times and the Hot Block temperature in TAL's.

NOTE: Digestion is complete when the digestate is light in color and does not change in appearance with continued refluxing.

10.10.6 Evaporate to low volume, approximately 3 - 5 mL.

10.10.7 Allow the digestion tube to cool, then add about 10 mL of reagent water.

10.10.8 Replace the cover and continue warming for 10 to 15 minutes to allow additional solubilization of any residue to occur. Record the start and stop times and the Hot Block temperature on the Supplemental Metals Prep Sheet.

10.10.9 Allow the sample to cool and rinse the watch glass into the digestion tube with reagent water.

10.10.10 Re-Volume to 50 mL with reagent water, cap and mix thoroughly.

10.10.11 The sample is now ready for ICP-MS analysis. Refer to SOP DV-MT-0018.

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to SOP DV-MT-0018 for additional details.

10.11 Method 3005A - Preparation for Total Recoverable and Dissolved Metals Analysis by ICP-MS Method 6020

10.11.1 To the sample in a digestion tube, add 2.0 mL of concentrated HNO3.

10.11.2 Heat the sample to 90 - 95 °C and cautiously evaporate to a low volume of 15 - 20 mL, while ensuring that no portion of the sample container is allowed to go dry. Record the start and stop times and the Hot Block temperature in TAL's.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

10.11.3 Allow the sample to cool in a fume hood.

10.11.4 Rinse the watch glass or cover into the digestion tube with reagent water.

10.11.5 Re-Volume to 50 mL with reagent water, cap and mix thoroughly.

10.11.6 The sample is now ready for ICP-MS analysis. Refer to SOP DV-MT-0018.

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to SOP DV-MT-0018 for additional details.

10.12 Method 200.8 - Preparation for Total Recoverable/Potentially Dissolved/Dissolved Metals Analysis by ICP-MS

10.12.1 To the sample, add 0.5 mL of concentrated HNO3 and 0.25 mL of concentrated HCl.

10.12.2 Adjust the digestion block to 85 °C so that the temperature of the solution in a covered container rises to approximately 90 - 95 °C. Record temperature on bench sheet.

10.12.3 Heat the sample until it evaporates to approximately 10 mL, while ensuring that no portion of the bottom of the digestion tube is allowed to go dry.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

10.12.4 Cover the sample and gently reflux for an additional 30 minutes. Avoid vigorous boiling to prevent the loss of the HCl-H2O azeotrope. Record the start and stop times and the Hot Block temperature in TAL's.

10.12.5 Allow the sample to cool in a fume hood.

10.12.6 Rinse the watch glass or cover into the container and re-volume to 25 mL with reagent water. Cap and mix thoroughly.

10.12.7 The sample is now ready for ICP-MS analysis. Refer to SOP DV-MT-0002.

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to SOP DV-MT-0002 for additional details.

10.13 **Method 3050B – Special Prep for Sb, Sn and Ag for Analysis by ICP-MS Method 6020**

10.13.1 To 25 mL of sample in a digestion tube, add 2.5 mL of HNO_3 and 2.5 mL of HCl.

10.13.2 Heat at 90-95 °C until the sample has reduced to a volume of 10-15 mL ensuring that no portion of the sample container is allowed to go dry.

10.13.2.1 Record the start and stop times and the Hot Block temperature in TAL's.

10.13.3 Remove the sample from the Hot Block and allow it to cool in a fume hood.

10.13.4 Add 1.0 mL of HCl to the digestion tube and cover with a ribbed watch glass.

10.13.5 Replace the watch glass and heat the sample for 15 minutes.

10.13.5.1 Record the start and stop times and the Hot Block temperature on the Supplemental Metals Prep Sheet.

10.13.6 Remove the sample from the Hot Block and allow it to cool in a fume hood.

10.13.7 Re-volume to 100 mL with reagent water, cap and mix thoroughly.

10.14 Calibration

The digestion block temperature must be maintained between 90 and 95 °C. The temperature must be monitored continuously while in use and must be recorded on the metals preparation bench sheet. The temperature must be monitored by measuring the temperature of reagent water contained in a digestion tube that is placed in each digestion block. The thermometer used and the start and end time temperatures are recorded in LIMS. The thermometer is calibrated in accordance with SOP DV-QA-0001, Thermometer Calibration.

11.0 Calculations / Data Reduction

Not applicable. See the determinative method SOP, DV-MT-0002, for data analysis and applicable calculations.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

- An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy DV-QA-005P. An MDL verification is performed once a year to satisfy NELAC 2003 requirement. For DoD, AFCEE, and DOE projects, an MDL verification is performed quarterly.
- The MDL studies and concentrations can be found at G:\QA\Read\MDL.

12.2 Demonstration of Capabilities

An initial demonstration of capability for each method must be performed prior to analyzing samples.

12.2.1 For the standard analyte list, the initial demonstration consists of the preparation and analysis of a QC check sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study.

12.2.2 Four aliquots of the QC check sample are analyzed with the same procedures used to analyze samples, including sample preparation.

12.2.3 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples.

12.2.4 For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is successful analysis of an extracted standard at the reporting limit and a single point calibration.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by and associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

13.1 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Safety Manual, and HS-001, "Waste Management Program."

14.2 The following waste streams are produce when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards: Contact Waste Coordinator

14.2.2 Acidic waste from sample digests: Waste Stream J.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.1.2 Method 3020A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy, Revision 1, July 1992.

15.1.3 Method 6020, Inductively Coupled Plasma - Mass Spectrometry, Revision 0, September 1994.

15.1.4 Method 3050B, Acid Digestion of Sediments, sludges and soils, Rev. 2, Dec. 1996.

15.2 Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983.

15.3 Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectroscopy, Revision 5.4, May 1994.

16.0 Miscellaneous Modifications:

16.1 Modifications and Interpretations Applicable to SW-846 Reference Methods

16.1.1 Chapter 1 of SW-846 states that the method blanks should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

16.1.2 The referenced methods, as well as Table 3-1 of SW-846, refer to the use of a 100-mL aliquot for digestion. This SOP requires the use of a 50-mL sample size to reduce waste generation. The use of reduced sample volumes is supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition", dated November 3, 1994. This document stated, "...flexibility to alter digestion volumes is addressed and 'allowed' by the table (3-1) and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..."

EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Oliver Fordham stated. " As a 'representative sample' can be assured, scaling causes no loss of precision and accuracy in the analysis."

16.2 Modifications Specific to Method 3005A

16.2.1 An additional 1.0 mL of HNO3 was included to replace the 5.0 mL of HCl. HCl was eliminated to reduce interferences from chloride.

16.3 Modifications and Interpretations Specific to Method 3020A

16.3.1 Section 10.10.6 of this SOP requires that the sample be reduced to a volume of 3 -5 mL. Section 7.2 of Method 3020A states that the volume should be reduced to 3 mL, but also states that no portion of the bottom of the digestion tube should go dry. The volume required by this SOP is a closer approximation of the volume required to provide an adequate covering of the bottom of the digestion tube so as to prevent the loss of critical analytes through volatilization.

16.3.2 The scope of 3020A has been expanded to include silver, based on comparison studies with 7760A. Method 3020A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water, and TCLP leachate) up to a concentration of 1 ppm silver.

16.4 Documentation and Records Management

The following documentation is contained within the LIMS system for each batch

- Batch number, Job and sample numbers, preparation date, and analyst name;
- Matrix, and prep type;
- Initial sample volume and final volume;
- Reagent manufacturer and lot number;
- Digestion tube lot information;
- Identification number for each standard used ;

  Calibrated measuring equipment used (thermometers, balances, pipettes, etc.).

17.0 Attachments

Figure 1.Method 3020A - Section 11.10
Figure 2.Method 3005A - Section 11.11
Figure 3.Method 200.8 - Section 11.12
Figure 4.Method 200.8 CLP Version - Section 11.13
Table 1.Approved Preparation Method Analytes
Table 2.ICP-MS Matrix Spike and Aqueous Laboratory Control Sample Levels
Table 3.TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels
Table 4.Summary of Quality Control Requirements
Appendix A.Contamination Control Guidelines
Appendix B. Work Instruction: Preparation of Water Samples for ICP-MS

18.0 Revision History

- Revision 3.4, dated 01 September 2010
  - Annual Technical Review
  - Removed all references to Supplemental Metals Prep Sheets
  - Removed all references to old LIMS prep codes
  - Updated Section 16.4 for new LIMS
  - Removed example prep sheets (Appendix A & B)
  - Updated Work Instruction Appendix D (now Appendix B)

- Revision 3.3, dated 24 August 2009
  - Updated temperature range in section 6.1.
  - Updated starting temperature in section 10.12.2
  - Added to section 10.12.4 to cover the samples

- Revision 3.2, dated 19 June 2009
  - Basic annual review
  - Updated some section references
  - Removed section 10.13.5 through 10.13.10
  - Updated Figure 4

- Revision 3.1, dated 13 June 2008
  - Added requirement to wait 24 hours after the addition of acid to samples.
- Revision 3, dated 03 March 2008
  - Integration for TestAmerica and STL operations.

18.1 Modifications from Previous Revision of the SOP

- Eliminated sample preparation methods for GFAA analysis from the SOP, because GFAA analysis is no longer performed at STL Denver. The title of the SOP and scope was changed accordingly.
- Added dissolved metals to Section 1.4.
- Added note to Section 1 to clarify that samples that consist of or contain oil or immiscible organic solvents are subcontracted to other laboratories.
- Deleted the GFAA sample prep methods from Section 2 and reformatted the section.
- In Section 6.5, clarified that filter media is checked by using the media to filter batch QC samples.
- Added Section 9.1 to summarize the laboratory QC program and reference QA-024 for QA requirements that apply to federal programs.
- Added note following Section 11.8.2 to explain filtration and preservation of samples for dissolved metals.
- In Sections 11.10, 11.11, 11.12, and 11.13, deleted final steps for filtering the digestate solution. This step, if needed, is performed by the ICP-MS analyst prior to analysis at the instrument.
- Revised Supplemental Metals Prep Sheet to change annotation for filtering samples for dissolved metals. The current version of the prep sheet is included as Appendix B.
- Revised Figures 1, 2, 3, and 4 to indicate that the pH of the sample is checked at the time it is received by the laboratory.

Figure 1:

Total Metals by 6020, Method 3020A- Section 10.10

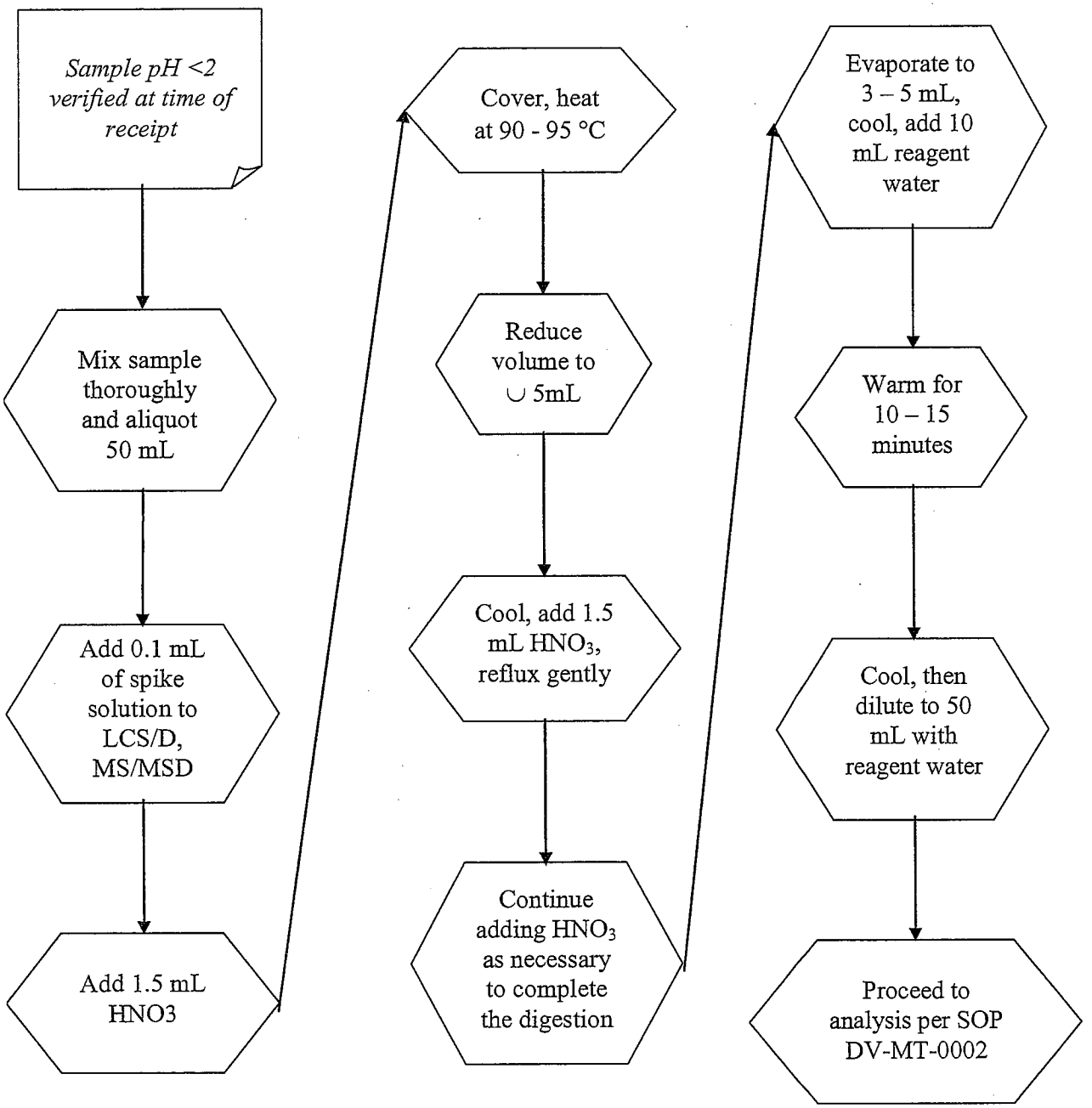

Figure 2.

Dissolved or Total Recoverable Metals by 6020, Method 3005A Section 10.11

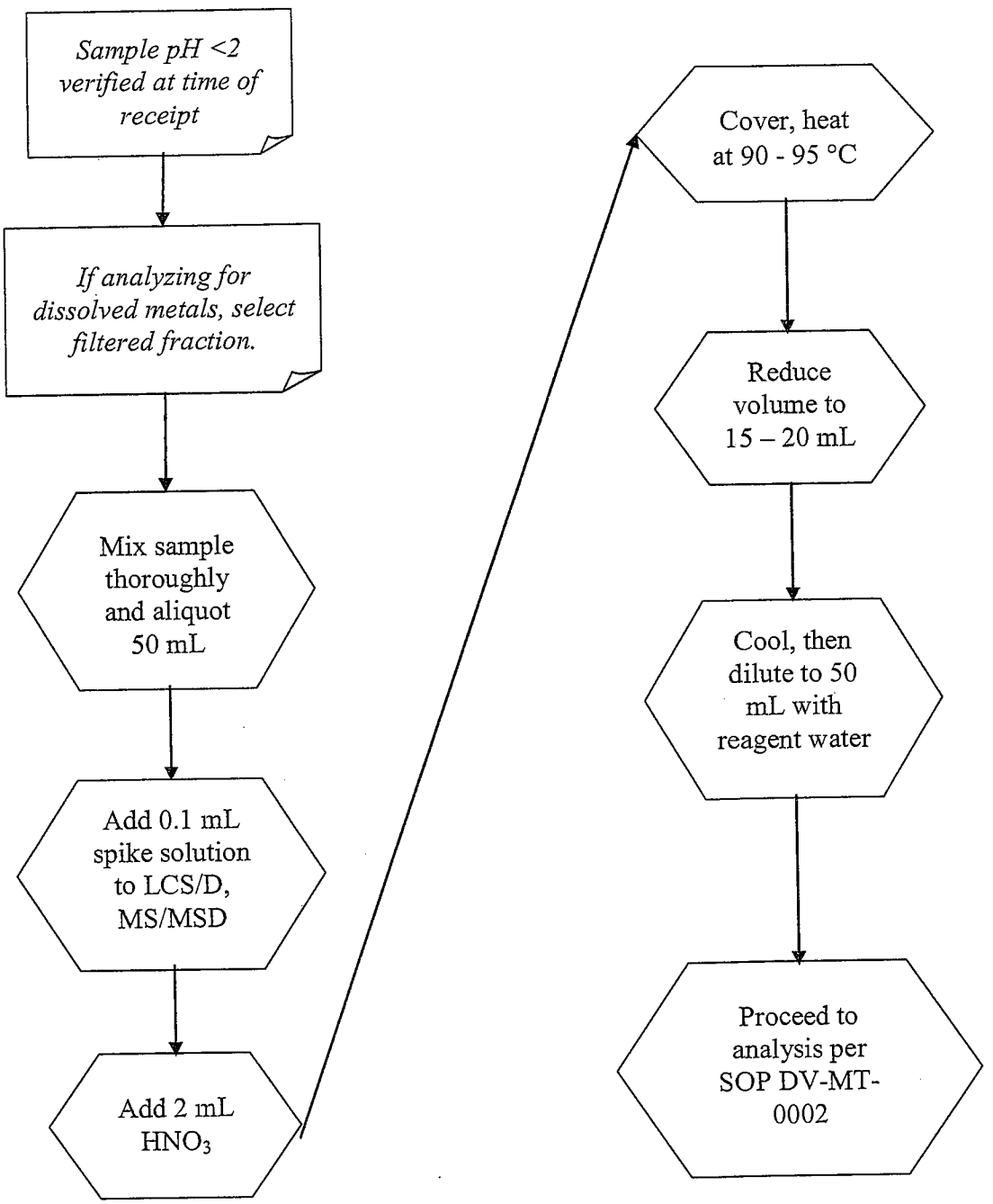

Figure 3.

Total Recoverable Metals, Method 200.8- Section 10.12

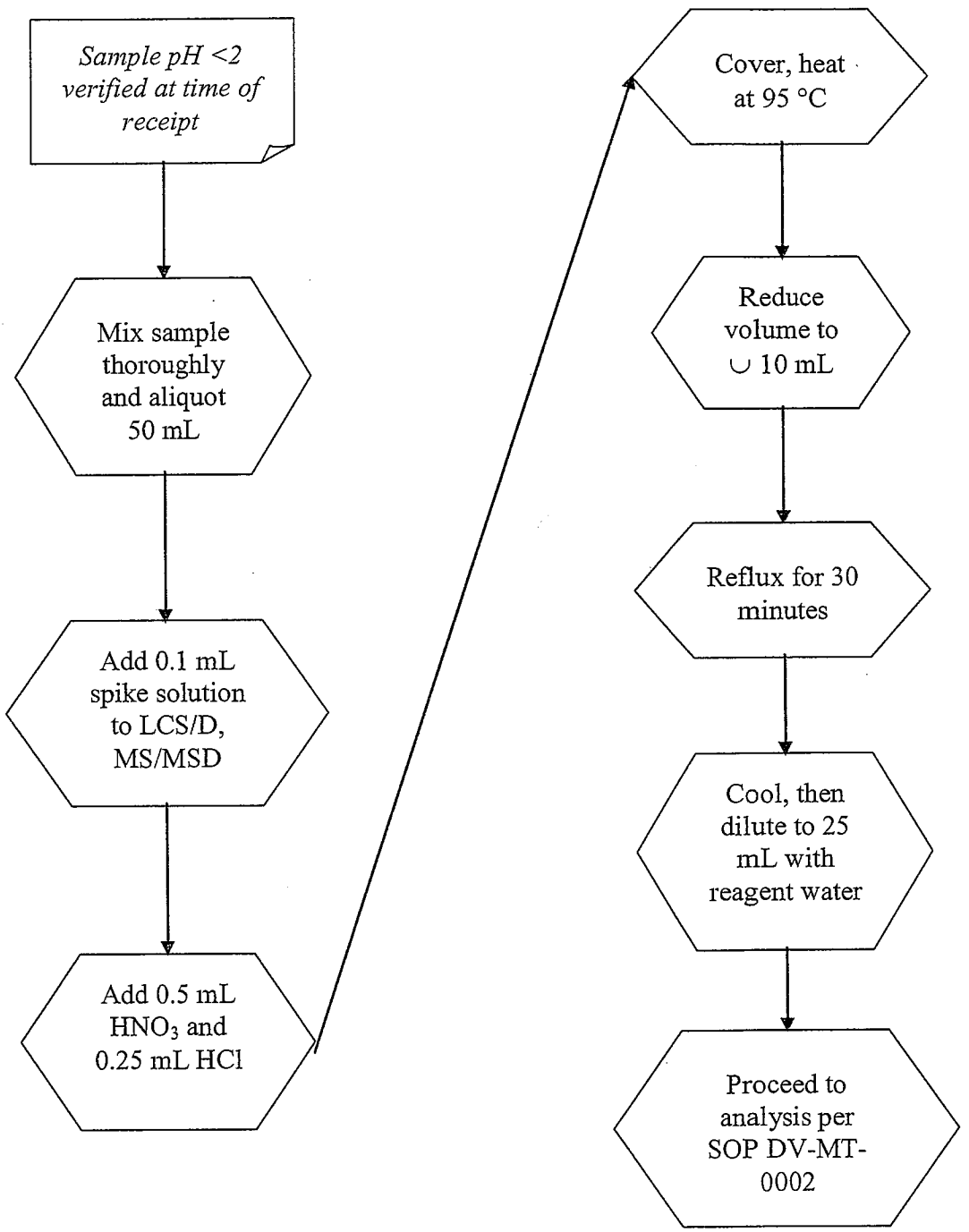

Figure 4

Prep for Sb, Sn and Ag, Method 6020- Section 10.13

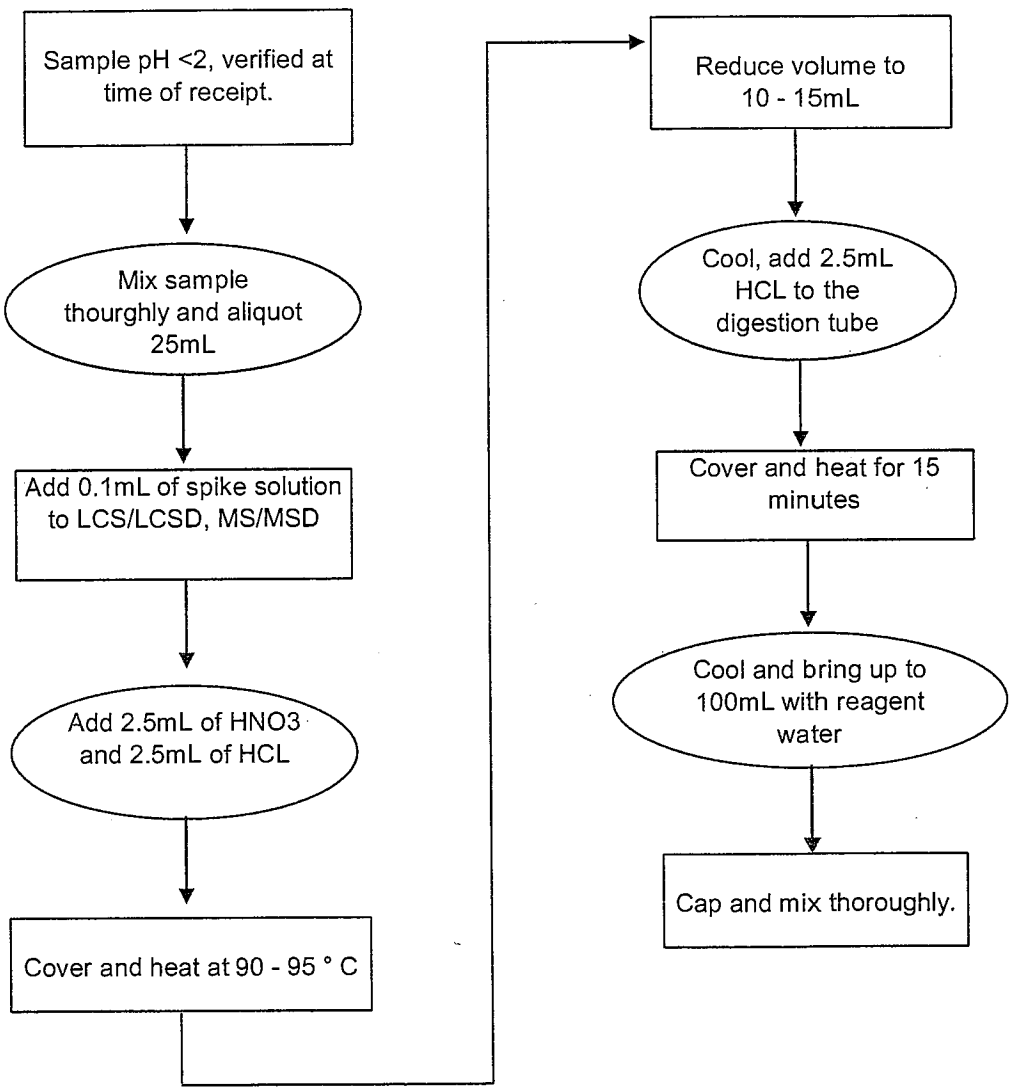

Table 1.

Approved Preparation Method Analytes

Element	Symbol	CAS Number	3005A	3020A	200.8
Aluminum	Al	7429-90-5	X		X
Antimony	Sb	7440-36-0	X		X
Arsenic	As	7440-38-2	X		X
Barium	Ba	7440-39-3	X		X
Beryllium	Be	7440-41-7	X	X	X
Cadmium	Cd	7440-43-9	X	X	X
Chromium	Cr	7440-47-3	X	X	X
Cobalt	Co	7440-48-4	X	X	X
Copper	Cu	7440-50-8	X		X
Lead	Pb	7439-92-1	X	X	X
Manganese	Mn	7439-96-5	X		X
Molybdenum	Mo	7439-98-7	X	X	X
Nickel	Ni	7440-02-0	X		X
Selenium	Se	7782-49-2	X		X
Silver	Ag	7440-22-4	X	X	X
Thallium	Tl	7440-28-0	X	X	X
Thorium	Th	7440-29-1			X
Uranium	U	7440-61-1			X
Vanadium	V	7440-62-2	X	X	X
Zinc	Zn	7440-66-6	X		X

X - Designates that the preparation method is approved for an element.

NOTE: Additional elements may be analyzed following digestion by these protocols, provided that the method performance criteria specified in Section 13.0 of this SOP are met.

Table 2.

ICP-MS Matrix Spike and Aqueous Laboratory Control Sample Levels

Element	Working LCS/MS Standard (mg/L)	Aqueous LCS/ MS Level* (µg/L)
Aluminum	20	40
Antimony	20	40
Arsenic	20	40
Barium	20	40
Beryllium	20	40
Cadmium	20	40
Chromium	20	40
Cobalt	20	40
Copper	20	40
Lead	20	40
Manganese	20	40
Molybdenum	20	40
Nickel	20	40
Selenium	20	40
Silver	20	40
Thallium	20	40
Vanadium	20	40
Zinc	20	40

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike, based on the addition of 0.1 mL of working spike standard to 50 mL of sample.

Table 3.

TABLE V. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

Table 4.

Summary of Quality Control Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOP: DV-MT-0002	Re-digest and reanalyze samples associated with the method blank.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOP: DV-MT-0002	Re-digest and reanalyze all samples associated with the LCS.
Matrix Spike (MS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOP: DV-MT-0002	Re-prep not required unless preparation error suspected.
Matrix Spike Duplicate (MSD)	See Matrix Spike	Refer to determinative SOP: DV-MT-0002	See Corrective Action for Matrix Spike.

Appendix A.

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Appendix B.

Work Instruction: Preparation of Water Samples for ICP-MS

TAL DENVER WORK INSTRUCTION	WI-DV-017	A	4/28/08	Page 28 of 28

TITLE:	**Preparation of Water Samples for ICP-MS**	**COPY#**
AUTHOR:	Richard Clinkscales	
QA REVIEW:		**DATE:**
SOP REFERENCE:	DV-IP-0014	*Acid Digestion of Aqueous Samples by SW-846 Methods 3005A, 3020A, 3050B and EPA Method 200.8 for ICP-MS*

Total Metals by ICP-MS 6020 (LIMS Prep Code MS/MH) – SW-846 Method 3020A

1. Shake sample to homogenize.
2. Pour 50 mL into sample vessel.
3. Spike LCS, LCSD, MS, and MSD with 0.1 mL 2008CAL-1 and 2008CAL-2 each.
4. Add 1.5 mL conc HNO_3 to each sample and QC sample.
5. Place on hot block set at 95 °C (covered container of water) until ~5 mL in volume.
6. Remove and let cool.
7. Add 1.5 mL conc HNO_3. Place back on the hot blocks for 30 minutes covered.
8. Remove and let cool.
9. Add ~10 mL reagent water and place back on the hot blocks for 10 minutes covered.
10. Remove and let cool completely.
11. Bring to final volume of 50 mL with reagent water. Cap and shake to mix.

Diss. / Tot. Rec. Metals by ICP-MS 6020 (LIMS Prep Codes 04/MH, MD/MH) – SW-846 Method 3005A

1. Select the appropriate sample container for either dissolved or total recoverable digestion.
2. Shake sample to homogenize.
3. Pour 50 mL into sample vessel.
4. Spike LCS, LCSD, MS, and MSD with 0.1 mL 2008CAL-1 and 2008CAL-2 each.
5. For lab-filtered samples, filter blank and LCS/LCSD using same type of filter that was used to filter the samples.
6. Add 2.0 mL conc HNO_3.
7. Place on hot block set at 95 °C (covered container of water) until ~5 mL in volume.
8. Remove and let cool completely.
9. Bring to final volume of 50 mL with reagent water. Cap and shake to mix.

Diss. / Total Rec. Metals by ICP-MS 200.8 (LIMS Prep Code 04/QV, 87/QV, PD/QV) – Method 200.8

1. Select the appropriate sample container for either dissolved, potentially dissolved or total recoverable digestion.
2. Shake sample to homogenize.
3. Pour 50 mL into sample vessel.
4. Spike LCS, LCSD, MS, and MSD with 0.1 mL 2008CAL-1 and 2008CAL-2 each.
5. Add 0.5 mL conc HNO_3 and 0.25 mL conc HCl to sample vessel.
6. Cover with watch glass and reflux on hot block set at 95 °C (covered container of water) until ~5 mL in volume.
7. Remove and let cool completely.
8. Bring to final volume of 25 mL with reagent water. Cap and shake to mix.

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Title: ACID DIGESTION OF SOLIDS [Method EPA 3050B]

Approvals (Signature/Date):

Doug Gomer 1/27/12
Doug Gomer Date
Metals Group Supervisor

Adam W Alban 03 Feb-12
Adam Alban Date
Health & Safety Manager / Coordinator

JPM 1/27/12
John P. Morris Date
Quality Assurance Manager

Robert C Hanisch 1/30/12
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This is a strong acid digestion procedure for the preparation of sediments, sludge, soils, and other types of solid materials by EPA Method 3050B for analysis by inductively coupled plasma atomic emission spectroscopy (ICP) or inductively coupled plasma-mass spectrometry (ICP/MS).

1.2 Method 3050B is designed to determine the concentration of "environmentally available" metals, and is not a true "total metals" digestion (see discussion below). The procedure is used primarily for hazardous waste characterization and other Resource Conservation and Recovery Act (RCRA) compliance testing.

1.3 The elements approved for Method 3050B are shown in Table I. The source method also mentions that other elements may be prepared by the method if the quality control requirements are met. The complete list of elements routinely included in this procedure by TestAmerica Denver is shown in Table II.

1.4 If sample preparation utilizing the Incremental Sampling Method is required, see SOP DV-OP-0013 for the procedure required prior to acid digestion for metals incorporating this procedure.

2.0 Summary of Method

A representative 1 to 2 gram portion of sample is digested with two cycles of nitric acid additions, followed by hydrogen peroxide digestion. For ICP analysis, the sample is also refluxed with hydrochloric acid. The resulting solution is filtered and diluted to 100 mL with reagent water. For the Incremental Sampling Method, 10g of sample is used and brought to a final volume of 500ml.

3.0 Definitions

3.1 Total Metals: Although Method 3050B is often referred to as a "total metals" digestion, it is important to understand that there are many compounds formed from these elements that are not efficiently dissolved using this digestion procedure. It is more accurately termed a strong acid digestion procedure. The limitations are discussed further in Section 4 (Interferences) below. The method itself states, "This method is not a total digestion technique for most samples." There are a variety of total digestion procedures used for metal assay, geochemical analysis, etc., that involve more vigorous digestions than 3050B.

3.2 Preparation Batch: A group of up to 20 samples that are of the same matrix and are processed together using the same lots of reagents and standards. The minimum QC elements in a batch are outlined in Section 9.

3.3 Other quality control terminology used in this procedure is based on SW-846, and is defined in the glossary section of the TestAmerica Denver Quality Assurance Manual (QAM).

4.0 Interferences

4.1 There are common compounds formed by the elements of interest (e.g., barium sulfate, beryllium oxide, silicon dioxide, crystalline silicates, titanium dioxide, etc.) that are not efficiently dissolved using this EPA approved procedure.

4.2 Silicon or silica are occasionally requested as part of the Method 3050B digestion. However, this digestion will include only acid-soluble silicon, and will not dissolve crystalline silica. The analysis is for silicon, but the final result is sometimes expressed as silica rather than silicon.

4.3 Antimony and silver have poor solubility in dilute nitric acid solution. Therefore it is strongly recommended that these elements are determined by the ICP procedure that includes HCl as the final digestion acid. See Section 11.7.8 of this SOP.

4.4 Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.5 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Attachment 1 for additional contamination control guidelines.

4.6 Boron and silica from the glassware will dissolve into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.7 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrix materials may not be digested using these methods if they are not soluble in acids. If physical interferences are present, they should be documented.

4.8 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals or conversion of metals to insoluble forms. For example, antimony is easily lost by volatilization from hydrochloric media. If this occurs the sample must be re-prepared.

4.9 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.10 Samples Requiring Additional Digestion Reagents

A few examples of types of samples that might require additional digestion reagents follow. It is very important to note situations where samples are not behaving normally. However, do not assume that adding additional reagents will be acceptable for the project, even if it is obvious that the digestion will be incomplete without it. The situation must be discussed with the project manager and documented in a Nonconformance Memo (NCM), whether or not the variations suggested in the following examples are approved.

4.10.1 Samples with high organic content may require additional nitric acid and/or hydrogen peroxide for a thorough digestion, but these oxidizing reagents should be added very carefully to avoid violent reactions.

4.10.2 Samples with high concentrations of metal in the elemental form or refractory oxides may require additional hydrochloric acid for a thorough digestion. As an example, blasting sand used to remove paint from the hull of ships typically consists of 30% cupric oxide. Following 3050B exactly will produce results as low as 0.1% without additional hydrochloric acid, and increasing the volume of hydrochloric acid can produce results approaching the true copper concentration.

4.10.3 Highly alkaline materials may require larger volumes of acid than specified in this procedure.

4.10.4 If the use of extra digestion reagents is approved, the same volume of reagents must be added to all field samples and QC samples in the batch. Usually the method blank results will not be elevated. To ensure that the QC sample results accurately reflect sample results, the QC samples must be treated exactly like the samples.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Hydrogen Peroxide, H_2O_2	Oxidizer Corrosive Poison	1 ppm TWA 1.4 mg/m^3 TWA 75 ppm IDLH	Contact with other materials may cause fire. Eye contact may result in permanent eye damage. Causes eye and skin burns. Corrosive: May cause severe respiratory tract irritation. Harmful if swallowed, may cause digestive tract irritation or burns.
Nitric Acid, HNO_3	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid, HCl	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Top-loading balance capable of accurately weighing to the nearest 0.01 grams.

NOTE: Balances are serviced annually and the accuracy checked daily using 3 standard masses. See SOP DV-QA-0014 for details.

6.2 Digestion "Hot Block" or equivalent heating device capable of maintaining a temperature of 90-95°C. The Hot Block temperature must be monitored separately for each unit. The temperature of each Hot Block is checked by placing a calibrated thermometer through a cap on a digestion tube that is filled approximately to the middle of the tube with water. The temperature is recorded on the preparation benchsheet.

6.3 Thermometers (non-mercury liquid filled or digital) that cover a temperature range including 80-110°C with 1°C increments clearly visible.

NOTE: Thermometers are calibrated before use and periodically as described in SOP DV-QA-0001.

6.4 Hot Block plastic digestion tubes, 50ml and 125 mL, disposable. The volumetric markings on the tubes are checked for each lot received to ensure accuracy of at least ± 3%. The documentation is kept on file in the Metals area.

6.5 Ribbed plastic cover, similar to a watch glass, for the digestion tubes; disposable.

6.6 Ahlstrom grade 55 filter paper, Fisher Q8 filter paper (acid washed), or equivalent.

6.7 Disposable plastic funnels.

6.8 Disposable wooden spatula for subsampling.

6.9 Centrifuge, capable of at least 2,000 rpm.

6.10 Graduated cylinder, 100 mL and 500 mL, capable of ± 3% accuracy.

6.11 Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

NOTE: Mechanical pipettes are calibrated before use and monthly as described in SOP DV-QA-0008.

6.12 Class A volumetric flasks.

6.13 pH indicator strips (pH range 0 – 6).

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of

sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagent water – Millipore DI system or equivalent, 10-18.2 megohm-cm. See SOP DV-QA-0026 for daily water monitoring procedure.

7.2 Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.3 Nitric acid (HNO_3), 5%

Add 5 mL of concentrated HNO_3 to approximately 900 mL of reagent water and dilute to 1 liter.

7.4 Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.5 30% Hydrogen peroxide (H_2O_2), reagent grade.

7.6 Glass beads, ≤ 1 mm diameter, washed with aqua regia (for AFCEE and DoD projects)

7.7 Standards

7.7.1 All standards must be NIST traceable. Unless purchased directly from NIST, the accuracy of each standard is verified before use, as described in SOP DV-QA-0015.

7.7.2 Storage and Shelf Life of Metal Standards

7.7.2.1 Standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. They are stored at room temperature.

7.7.2.2 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.7.3 LCS and MS Spike Solution for ICP

7.7.3.1 ICP spike solutions are purchased as custom-made solutions from a commercial vendor at ready-to-use concentrations. No further dilutions are needed. Spikes are prepared as follows:

- Routine ICP: Add 1.0 mL of spike
- AFCEE ICP: Add 1.0 mL of spike to 1.0 g of glass beads

The resulting spike concentrations for each element are given in Table II.

7.7.3.2 ICP/MS spike solutions are also purchased as custom-made solutions from a commercial vendor at ready-to-use concentrations. No further dilutions are needed. 1.0 mL of spike solution is added to samples. The concentrations of the elements in the stock standard and the resulting concentrations in samples are shown in Table III.

7.7.4 If a non-routine element is required that is not contained in the custom-made solution, single-element solutions from a commercial vendor may also be used.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2 Soil samples do not require chemical preservation, but are stored at 4 ± 2 °C until the time of analysis.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Glass	3 grams	Cool 4 ± 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. The process of establishing control limits, and the use of control charts are described more completely in DV-QA-003P, *QualityAssurance Program*. Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents.

9.3 Initial Demonstration of Capability

An initial demonstration of capability must be performed by analysts before digesting samples using this procedure. See Section 13 of this SOP for further details.

9.4 Minimum QC Elements in a Preparation Batch

Each preparation batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD) pair. Note that some programs require an unspiked duplicate sample in place of or in addition to the duplicate matrix spike. Be sure to check special instructions in the laboratory LIMS. If clients specify specific samples for MS and MSD, the batch may contain multiple MS/MSD pairs.

9.5 Sample Count

Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.6 Method Blank (MB) One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. Soil method blanks are prepared by taking 5 mL or 5 g of reagent water through the procedure described in Section 11.

The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

Acceptance Criteria: Criteria for the acceptance of blanks are contained within the individual analytical method SOPs.

Corrective Action: If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be re-digested and reanalyzed.

9.7 Laboratory Control Sample (LCS)

9.7.1 One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure.

9.7.2 The spike solution described in Section 7.7.3 is used to prepare LCSs as follows:

- Routine ICP: Add 1.0 mL of spike
- AFCEE/DoD ICP: Add 1.0 mL of spike to 1.0 g of glass beads
- ICP/MS: Add 1.0 mL of spike

The resulting spike concentrations for each element are given in Table 2 and Table 3.
Incremental Sampling Method LCS's are spiked with 5ml of spike.

9.7.3 The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

Acceptance Criteria: Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs.

Corrective Action: When LCS results fail to meet control limits, the LCS and all associated samples in the batch must be re-prepared and reanalyzed.

9.8 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.8.1 One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a second aliquot of a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a third aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.8.2 The spike solution described in Section 7.7.3 is also used to prepare matrix spikes, as follows:

- ICP: Add 1.0 mL of spike
- ICP/MS: Add 1.0 mL of spike

The resulting spike concentrations for each element are given in Tables II through IV.

Incremental Sampling Method MS/MSD's are spiked with 5ml of spike.

9.8.3 The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process.

Note: The spike must be added after the sample aliquot but before any reagents.

Acceptance Criteria:	Criteria for the acceptance of MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results are contained within the individual analytical method SOPs.
Corrective Action:	If any analyte recovery or RPD falls outside the established acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery for the LCS is also outside of established limits, corrective action must be taken. Corrective action will include re-preparation and reanalysis of the batch. Corrective action when MS results alone fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred.

10.0 Calibration

Not applicable. This SOP addresses sample preparation only for subsequent ICP or ICP/MS analysis. Calibration of the measurement system is covered in the SOPs for the determinative methods.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Sample Custody

11.2.1 Custody of samples is transferred from the Sample Control group to the Metals group, which is documented using the internal program, Sample Transfer Utility (see SOP DV-QA-0003 for details).

11.2.2 Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be done in a manner to ensure connection with the proper sample.

11.3 Subsampling

11.3.1 It is not acceptable to simply collect 1.0 g off of the top of the sample. Samples

must be mixed and incrementally subsampled to obtain a representative subsample. At a minimum, mix by stirring with a disposable wooden spatula. If there is insufficient room in the sample container to allow for proper mixing, refer to SOP DV-QA-0023, "Subsampling" for directions.

11.3.2 Select at least three incremental subsamples from different locations in the original sample to obtain a final subsample weight of 1.0 to 1.2 g in a digestion tube, and record the exact weight to the nearest 0.01 g. A 2.0-g sample size may also be used if needed to meet the reporting limits.

11.3.3 Measure additional aliquots for QC samples required in the batch and spike as required (see Section 9 for details).

11.4 Digestion of 10g sample aliquot obtained utilizing previously prepared Incremental Sampling Method soil aliquot

11.4.1 The Method 3050B digestion reagents are increased 5x to maintain the same chemistry as is used for a 1-2 gram subsample. 10g of sample is digested in 125ml tubes.

11.5 Initial Digestion Cycle with 1:1 Nitric Acid

11.5.1 Add approximately 5mL of reagent water to each digestion tube.

11.5.2 Add 5 mL of concentrated HNO_3, and mix the sample.

11.5.3 Place a ribbed cover on each tube.

11.5.4 Heat samples to 95°C, and reflux for 15 minutes without boiling.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.5.5 Allow sample to cool before proceeding with the next step.

11.5.6 Record the start time, starting temperature, end time, and ending temperature in LIMS.

11.6 Second Digestion Cycle Using Concentrated Nitric Acid

11.6.1 Add 5 mL of concentrated HNO_3, and replace the ribbed cover.

11.6.2 Reflux at 95°C for 30 minutes. Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.

11.6.3 If brown fumes are observed, this means that material in the sample is actively being oxidized. There may not be enough HNO_3 acid to complete the oxidation, and there could be violent reaction of the sample with peroxide in the third digestion step. For that reason, it is necessary to repeat the previous two steps until no more fumes are evolved.

11.6.4 Allow the sample to evaporate to 5 mL, while ensuring that no portion of the bottom of the beaker is allowed to go dry. Alternatively, heat at 95^{o}C for 2 hours.

11.6.5 Allow the samples to thoroughly cool before proceeding.

11.7 Third Digestion Cycle Using Hydrogen Peroxide

11.7.1 Add 2 mL of reagent water to each tube.

11.7.2 Add 3 mL of 30 % H_2O_2 a few drops at a time. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.7.3 Replace the ribbed cover and heat sample until effervescence subsides.

11.7.4 Allow the sample to cool.

11.7.5 Continue adding 30% H_2O_2 in 1-mL increments with warming until effervescence is minimal or sample appearance is unchanged.

NOTE: Do not add more than a total of 10 mL of 30 % H_2O_2.

11.7.6 Continue heating at 95^{o}C until the volume is reduced to approximately 5 mL. Alternatively the sample may be heated for 2 hours.

11.7.7 Allow the sample to cool

11.7.8 If samples will be analyzed by ICP, continue on with the fourth digestion step using HCl in step 11.7.8. If the samples will be analyzed by ICP/MS, skip the HCl digestion step and go to step 11.9.

11.8 Fourth Digestion Cycle for ICP Using Concentrated Hydrochloric Acid

11.8.1 If the sample is being prepared for ICP analysis, add 10 mL of concentrated HCl to the sample in the digestion tube, and cover with ribbed cover.

11.8.2 Reflux for an additional 15 minutes without boiling.

11.8.3 Allow the sample to cool.

11.9 Separating Undigested Solids from the Digestion Solution

11.9.1 Filter sample through filter paper into a measured 125ml bottle whose accuracy is documented to be better than $\pm$ 3%.

NOTE: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.9.2 For samples digested by the incremental Sampling Method use a 500mL poly bottle that has been marked by pouring out 500mL of DI water from a graduated cylinder.

11.9.3 Wash the digestion tube and ribbed cover with reagent water to ensure quantitative transfer of all of the digestion solution.

11.9.4 Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.9.5 Re-volume sample to 100 mL with reagent water. This must be done volumetrically, rather than by weight. Record the final volume in TAL's. For Multi-Incremental samples the final volume is 500ml.

11.10 Documentation and Record Management

11.10.1 The following information must be recorded for each preparation batch. This information is entered into the LIMS system

- Batch number,
- List of samples,
- Initial sample weight and final digestion volume,
    - Preparation analyst and date,
    - Matrix,
    - Preparation type,
    - Identification of reagents and standards used, and
    - Identification of all measuring and test equipment used (e.g., balances, thermometers, pipettes).

11.11 Antimony for Analysis by ICP-MS

11.11.1 Weigh 1.0 to 1.2g soil sample using sub sampling in digestion vessel.

11.11.2 Add 5-mL of reagent water to the blank and LCS/LCSD.

11.11.3 Spike LCS/LCSD, MS, MSD with 1.0 mL 200.8 CAL-1. (Only CAL-1 needed)

11.11.4 Add 2.5-mL conc. HNO_3 and 2.5 mLs conc. HCI to each sample and QC.

11.11.5 Cover with a watch glass and reflux on hot block set at 95° C (covered container of water) for 15 minutes.

11.11.6 Filter through Ahlstrom 55 into 100-mL vessel while still hot.

11.11.7 Rinse with hot 1.25mls (~95° C) conc. HCI.

11.11.8 Rinse 3X with hot (95° C) reagent water (5mL rinses.)

11.11.9 Place the filter paper and soil residue back into the original sample digestion vessel. Add 2.5 mLs conc. HCI, cover and reflux on hot block for 20 minutes or until paper dissolves.

11.11.10 Filter through Ahlstrom 55 adding to the original filtrate. Rinse 3X with reagent water. (5mL rinses.)

11.11.11 Bring to final volume of 100 mL with reagent water.

12.0 Calculations / Data Reduction

Not applicable. Calculations of final results are described in the determinative analytical SOPs.

13.0 Method Performance

13.1 Method Detection Limit (MDL)

An MDL must be determined for each analyte/matrix prior to the analysis of any samples. See the SOPs for the determinative analysis methods for details.

13.2 Initial Demonstration Study

This requires the analysis of four QC check samples. The QC check sample is a well-characterized, laboratory-generated sample used to monitor method performance, which should contain all the analytes of interest. Typically this is the LCS. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

13.2.1 Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

13.2.2 Calculations and acceptance criteria for QC check samples are given in the determinative SOPs for ICP/MS and ICP (DV-MT-0002 and DV-MT-0012, respectively).

13.3 Training Qualification:

13.3.1 The group leader or supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, “Waste Management and Pollution Prevention”, of the Environmental Health and Safety Manual, and DV-HS-001P, “Waste Management Program.”

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Aqueous Acidic (Metals) - Corrosive – Waste Stream J

15.2.2 Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996; Method 3050B.

17.0 Method Modifications:

Item	Method	Modification

1	3050B	Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.
2	3050B	Section 7.5 digestion for Sb was modified to add less HCL to limit the interference on the ICPMS instrument.

18.0 Figures, Tables, and Attachments

Figure 1: Soil Sample Preparation Flowchart
Figure 2: Soil Sample Preparation Flowchart for Antimony by ICP-MS
Table 1: Method 3050B Approved Analyte List for ICP/ICP-MS
Table 2: Soil LCS and MS/MSD Spikes for ICP
Table 3: Soil LCS and MS/MSD Spikes for ICP-MS

Attachment 1: Contamination Control Guidelines
Attachment 2: Work Instruction: Preparation of Soil Samples for ICP and ICP-MS

19.0 Revision History

- Revision 4 dated 3 February 2012
  - Changed references of Multi-Incremental Sampling to Incremental Sampling Method throughout document
  - Section 2.0 Added reference to Incremental Sampling Method
  - Section 6.4 Added 50ml digestion tubes
  - Added introductory statement to section 7.0 regarding reagent purity
  - Section 7.1 Updated acceptable criteria for the reagent water
  - Section 9.7.2 Added LCS Incremental Sampling Method spike amounts
  - Section 9.8.2 Added MS/MSD Incremental Sampling Method spike amounts
  - Section 11.4 Updated sample amount for Incremental Sampling Method to 1 10g aliquot
  - Section 11.9 Added Incremental Sampling Method final volume

- Revision 3.5, dated 24 August 2011
  - A note has been added to section 9.8.3 for the addition of the LCS/MS spike before reagents.

- Revision 3.4, dated 01 September 2010
  - Annual Technical Review
  - Updated documentation in section 11.10 to reference new LIMS.
  - Updated Section 11.11 and Figure 2 to reference current HCL amount used. Added method modification to digestion in Section 17.
  - Added Bismuth to Table 2
  - Updated Figure 2
  - Removed Example prep sheets (Attachments 2 – 4)
  - Updated work instruction WI-DV-015 (Attachment 5 now Attachment 2)

- Revision 3.3, dated 17 August 2009

- Sections 11.3.2 and 11.11.1: Updated amount of sample used to range from 1.0 to 1.2g.

- Revision 3.2, dated 28 April 2009
    - Sec 6.6: Updated filter paper from Whatman No. 541 to Ahlstrom grade 55.
    - Sec 7.6: Added glass beads to DoD projects.
    - Sec 7.7.3.1: Removed ICPMS spike from this section.
    - Sec 7.7.3.2: Updated spike volume to 1.0ml for ICPMS.
    - Table 3: Updated ICPMS spike level to 1.0ml.

- Revision 3.1, dated 28 May 2008
    - The spiking amount for LCS, MS and MSD was changed from 0.2 mL to 1.0 mL for ICP-MS.

- Revision 3, dated 12 October 2007
    - Integration for TestAmerica and STL operations.
    - Added reference to SOP DV-OP-0015 for Multi-Incremental Subsampling Preparation of Soil Aliquots
    - Removed references to Graphite Furnace Atomic Absorption Analysis

- Revision 2.3, dated 11 November 2005
    - Section 6.3: Replaced the specification to use a mercury thermometer with a non-mercury thermometer.
    - Section 6.4: The Hot Block digestion tube volume was changed from 60 mL to 125 mL.
    - Section 6.6: Whatman No. 41 filters were changed to Whatman 541, or Fisher Q8 (acid washed) filters.
    - Section 6.14: This section, which specified HDPE plastic bottles for storing digested samples, was removed. The digests are stored in the Hot Block tubes.

    - Section 7.7.3.2: The volume of spike solution used for ICP/MS samples was changed from 1.0 mL to 0.2 mL.
    - Section 9.7.2: Added the volume of spike used for ICP-MS, i.e., 0.2 mL.
    - Item 9.8.2: Added the volume of spike used for ICP-MS, i.e., 0.2 mL.
    - Section 11.7.8: Added information concerning the use of HCL for Sb analysis.
    - The Safety Section 5 and the Waste Management section 15 were updated to current STL Corporate requirements.
    - Added examples of preparation bench sheets as Attachments 2, 3, 4, and 5.

Figure 1.

Soil Sample Preparation Flowchart
(Page 1)

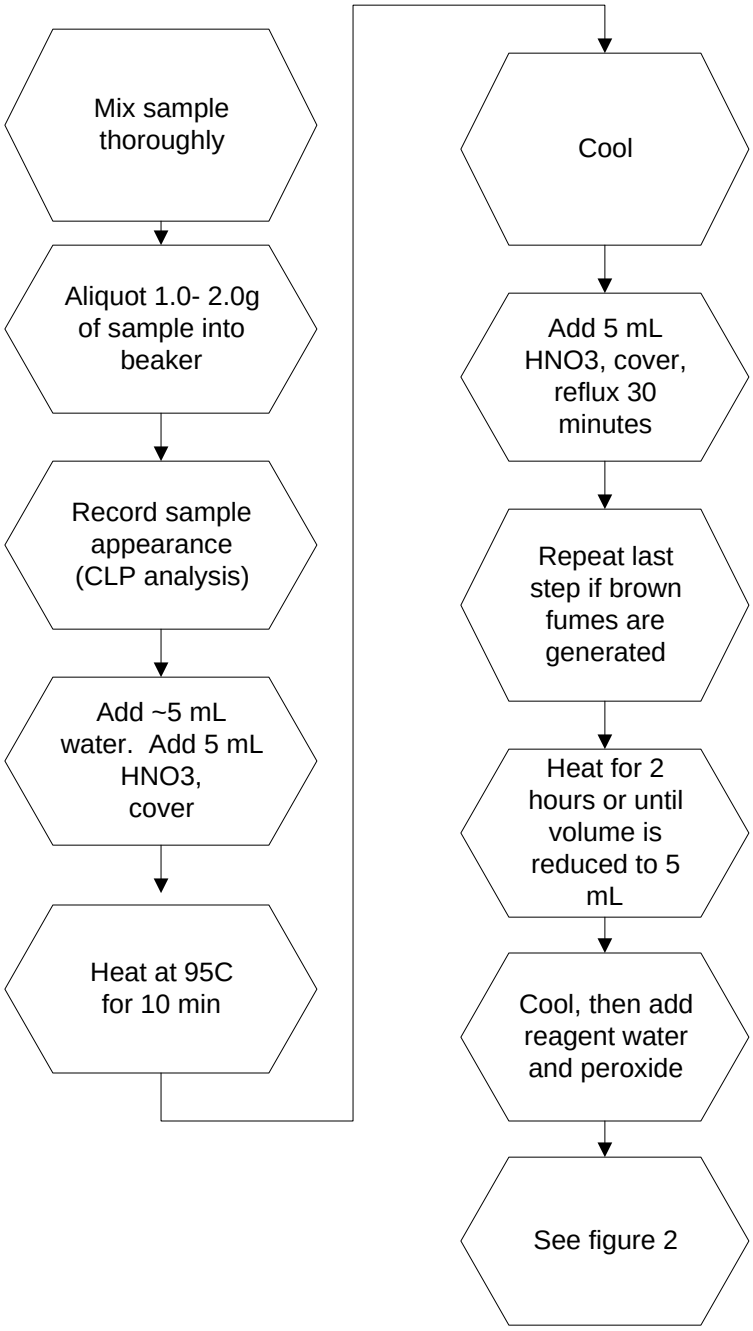

Figure 1. (continued)

Soil Sample Preparation Flowchart
(Page 2)

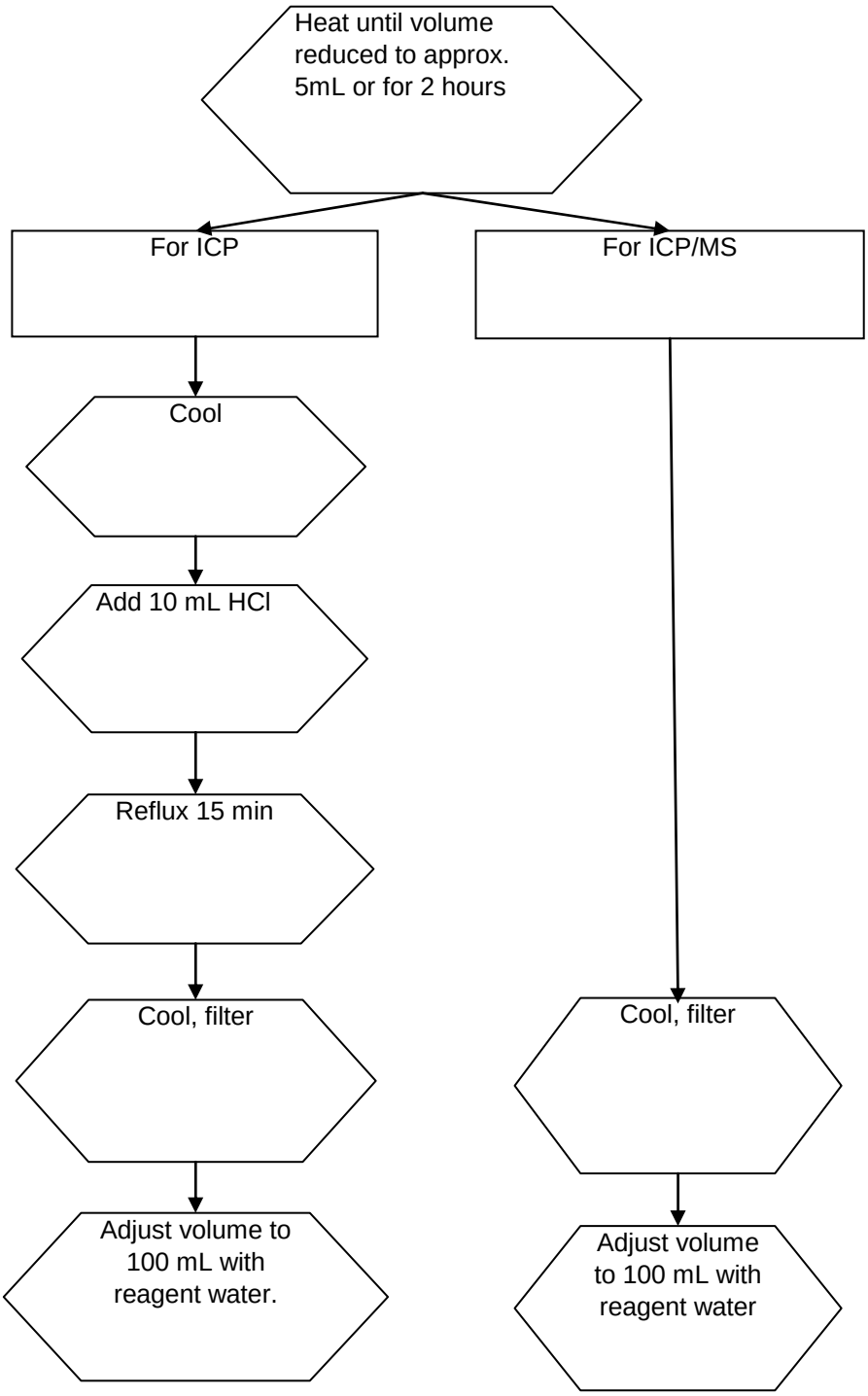

Figure 2

Soil Sample Preparation Flowchart for "Hot" Antimony by ICP-MS

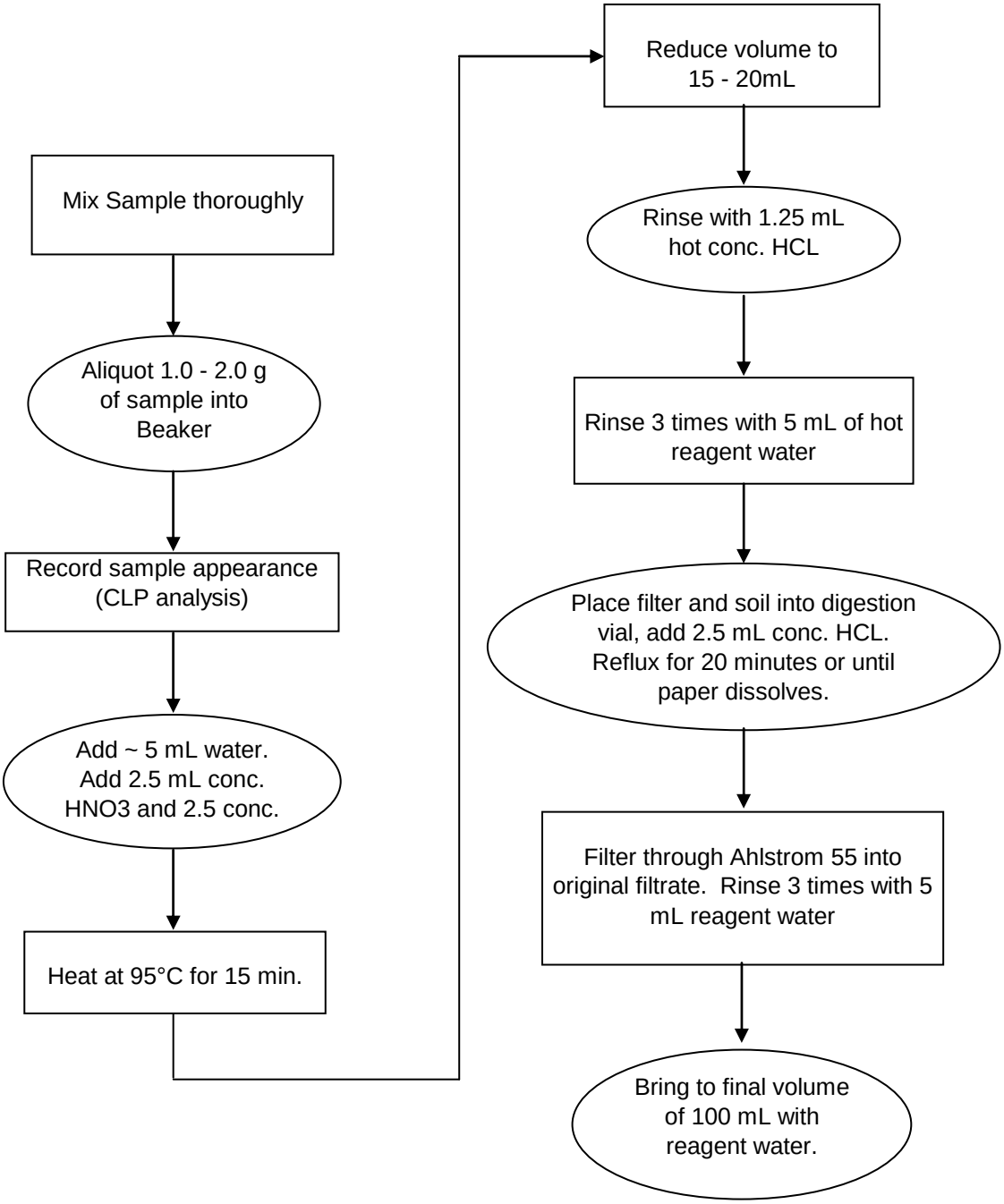

Table 1.

Method 3050B Approved Analyte List for ICP/ICP-MS

ELEMENT	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

Table 2.

Soil LCS and MS/MSD Spikes for ICP

ELEMENT	Stock Standard (mg/L)	Sample Spike (mg/kg)	Final Digested Solution (mg/L)
Aluminum	200	200	2.0
Antimony	50	50	0.5
Arsenic	100	100	1.0
Barium	200	200	2.0
Beryllium	5	5	0.050
Bismuth	200	200	2
Boron	100	100	1.0
Cadmium	10	10	0.1
Calcium	5000	5000	50.
Chromium	20	20	0.20
Cobalt	50	50	0.50
Copper	25	25	0.25
Iron	100	100	1.0
Lead	50	50	0.50
Lithium	100	100	1.0
Magnesium	5000	5000	50.
Manganese	50	50	0.50
Molybdenum	100	100	1.0
Nickel	50	50	0.50
Phosphorous	1000	1000	10.
Potassium	5000	5000	50.
Selenium	200	200	2.0
Silica	1000	1000	10.
Silver	5	5	0.050
Sodium	5000	5000	50.
Strontium	100	100	1.0
Thallium	200	200	2.0
Thorium	100	100	1.0
Tin	200	200	2.0
Titanium	100	100	1.0
Uranium	200	200	2.0
Vanadium	50	50	0.50
Zinc	50	50	0.50
Zirconium	50	50	0.5

NOTE: Final soil spike concentration based on the addition of 1.0 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

Table 3.

Soil LCS and MS/MSD Spikes for ICP-MS

ELEMENT	Stock Standard (mg/L)	Sample Spike (mg/kg)	Final Digested Solution (μg/L)
Aluminum	20	20	200
Antimony	20	20	200
Arsenic	20	20	200
Barium	20	20	200
Beryllium	20	20	200
Cadmium	20	20	200
Chromium	20	20	200
Cobalt	20	20	200
Copper	20	20	200
Lead	20	20	200
Manganese	20	20	200
Molybdenum	20	20	200
Nickel	20	20	200
Selenium	20	20	200
Silver	20	20	200
Thallium	20	20	200
Tin	20	20	200
Titanium	20	20	200
Uranium	20	20	200
Vanadium	20	20	200
Zinc	20	20	200

NOTE: Final soil spike concentration based on the addition of 0.2 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

Attachment 1

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered or Latex Gloves should not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Attachment 2

Work Instruction: Preparation of Soil Samples for ICP and ICP-MS

TAL DENVER WORK INSTRUCTION		WI-DV-015	A	5/28/08	Page 24 of 24
TITLE:	**Preparation of Soil Samples for ICP and ICP-MS**			**COPY#**	
AUTHOR:	Richard Clinkscales				
QA REVIEW:				**DATE:**	
SOP REFERENC	DV-IP-0015 *Acid Digestion of Soil Samples by SW-846 Method 3050B for ICP and ICP-MS*				

Metals in Soils by ICP-MS 6020 and ICP 6010 (LIMS Prep Codes 46/MH, 46/QO)

1. Open container and mix with tongue depressor to homogenize samples.
2. Weigh a representative 1.0 ± 0.3g of the sample into the digestion tube. Record the weight.
3. Weigh additional aliquots for QC samples into their respective digestion tubes.
4. Add 5 mL of reagent water into each digestion tube and swirl to mix

For ICP-MS

1Spike LCS, LCSD, MS, and MSD with 1.0 mL of the ICP-MS spike solutions for each QC sample.

For ICP

5 Spike LCS, LCSD, MS, and MSD with 1.0 mL of the ICP spike solutions for each QC sample.
5. Add 5 mL of concentrated HNO_3 into each digestion tube, swirl to mix and cover with a watch glass.
6. Heat at 95 ºC for 15 minutes. **DO NOT ALLOW SAMPLE TO GO DRY**.
7. Remove from the HotBlock and let cool.
8. Add 5 mL of concentrated HNO_3 into each digestion tube, swirl to mix and cover with a watch glass.
9. Heat at 95 ºC for 2 hours minutes. Add reagent water as needed to keep the volume to at least 5 mL.
10. Remove from the HotBlock and let cool completely.
11. Add 2 mL of reagent water to each tube.
12. Slowly add 3 mL of H_2O_2 to each tube and cover with a watch glass.
13. Heat sample at 95° C for 2 hours. Remove from the HotBlock and cool.

For ICP-MS

14. Skip to Step 18.

For ICP

15 Add 10 mL of concentrated HCl to each digestion tube and cover with a watch glass.
15. Heat sample for 15 minutes.
16. Remove from the HotBlock and allow to cool.
18. Filter the sample and re-volume to 100 mL with reagent water.

Antimony in Soils by ICP-MS 6020 (LIMS Prep Code U1/MH)

1. Open container and mix with tongue depressor to homogenize samples.
2. Weigh a representative 1.0 ± 0.3g of the sample into the digestion tube. Record the weight.
3. Weigh additional aliquots for QC samples into their respective digestion tubes.
4. Add 5 mL of reagent water into each digestion tube and swirl to mix.
5. Add 2.5 mL of conc. HNO_3 and 1.5 mL conc, HCl into each digestion tube and cover with a watch glass.
6. Heat at 95 ºC for 15 minutes. **DO NOT ALLOW SAMPLE TO GO DRY**.
7. Filter while the digestion tube is still hot. Rinse with 5 mL of hot HCl.
8. Rinse 3X with 5 mL of hot reagent water.
9. Place the filter paper and residue back into the original digestion tube and add 5 mL of reagent water and 1.5 mL of conc. HCl. Heat for 20 minutes or until the filter paper dissolves.
10. Remove from the HotBlock and let cool completely.
11. Filter the sample and re-volume to 100 mL with reagent water.

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Title: Screening for Volatile Organics by Headspace GC/FID

Approvals (Signature/Date):

William Rhoades 5/29/12
William Rhoades Date
Technical Manager

Adam W Alban 30 May 12
Adam Alban Date
Health & Safety Manager / Coordinator

5/30/12
John Morris Date
Quality Assurance Manager

Robert C Hanisch 5/30/12
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 Volatiles screening analysis by Headspace GC/FID is done to establish the approximate concentration of the following analytes to determine a workable dilution for final analysis by GC/MS. This is a semi-quantitative technique.

1.2 Analytes - The analytes that are evaluated by this method are listed in Table 1. Other compounds can be detected, and if present, would enter into the screening outcome.

1.3 Detection Limits - Since this procedure is a screen to determine approximate concentrations, detection limits do not apply.

1.4 Approximate analytical time - 20 minutes per sample.

2.0 Summary of Method

An aliquot of sample is analyzed using a special headspace vial, or by mini-extraction using methanol as an extraction solvent. The extracts are then analyzed by the headspace-GC/FID. The data are then evaluated to determine an applicable dilution for GC/MS detection limits.

3.0 Definitions

Volatile Organics: Any purgeable organic compounds that chromatograph when the column is operated in the 25-260 degree C range.

4.0 Interferences

Carbon tetrachloride co-elutes with the fluorobenzene internal standard. When a sample is found to contain carbon tetrachloride, it must be screened on a GC/MS instrument.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must

review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Hewlett Packard 5890A Gas Chromatograph or equivalent

6.1.2 Supelco 20 mm LB2 Septa or equivalent

6.1.3 Hewlett Packard frosted quartz 2 mm ID injection port liner or equivalent

6.1.4 J & W Scientific fused silica megabore 30 m x 0.54 mm open tubular column with DB-624 liquid phase or equivalent

6.1.5 Hewlett Packard Flame Ionization Detector or equivalent

6.1.6 Instrument maintenance is described in Attachment A.

6.1.7 Tekmar 7000 NT

6.1.8 HP Chemstation Chromatographic Data System or equivalent

6.1.9 Compaq Personal Computer or equivalent

6.1.10 Centrifuge

6.2 Supplies

6.2.1 Sun 20 mL VOA vials with TFE-lined 24-40 solid caps or equivalent

6.2.2 Hewlett Packard 10 mL headspace vials part # 9301-0717 or equivalent

6.2.3 Hewlett Packard aluminum crimp caps 20 mm par t# 9301-0718 or equivalent

6.2.4 Hewlett Packard septa - 20 mm TFE-faced silicone part # 9301-0976 or equivalent

6.2.5 Hewlett Packard crimper for 20 mm caps part # 9301-0720 or equivalent

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent water – Analyte Free (D.I. water that has been boiled and purged with nitrogen.)

7.2 Reagent grade Sodium Chloride

7.3 High purity Methanol (purge and trap grade)

7.4 8240B primary VOC calibration standard. Refer to the MS VOA standards prep database for contents and concentrations.

7.5 Screening Internal Standard (IS): BFB and fluorobenzene in methanol at 200 ppm, prepared at TestAmerica Denver.

7.6 MSVOA-SCS prepared at TestAmerica Denver (Table 2).

7.7 MSVOA-LCS prepared at TestAmerica Denver (Table 3).

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 The GC/MS volatile organic analysis SOP DV-MS-0010 has complete details. In brief, water samples for volatile organic analysis come in 40 mL septum-cap vials with no headspace. Soil samples normally include at least one portion of sample in 4 oz wide-mouth jars, which should be used rather than the portion in EnCores or sealed vials.

8.2 All samples to be screened by Headspace-GC/FID for final analysis by MS must be extracted and analyzed within 48 hours of sample receipt.

9.0 Quality Control

9.1 Quality control batches consist of 20 samples. For every 20 samples, one LCS and one Method Blank are prepared. Once a batch has 20 samples a new batch will need to be started.

9.2 Low Level Extraction Method Blank:

9.2.1 Add 0.5 to 1.0 g of NaCl to a 10 mL headspace vial.

9.2.2 Add 5 mL of DI water to a salted headspace vial.

9.2.3 Using a 25 uL Hamilton syringe, add 5 uL of each of the screening IS standard.

9.2.4 Cap tightly.

9.3 Medium Level Extraction Method Blank:

9.3.1 Whenever matrices are extracted with methanol, whether they are medium level soils or wastes, a methanol method blank must be prepared.

9.3.2 Add 5.0 mL purge and trap grade methanol and 5 g of Ottawa sand to a 20 mL screw cap vial.

9.3.3 Add 4.0 uL MSVOA-SCS at 2,500 ug/mL.

9.3.4 Shake briefly.

9.3.5 Save the method blank extract in two 2.0 mL vials that are labeled “BLANK”, with the lot number and the date prepped.

9.3.6 The number of method blanks can be minimized by preparing all methanol extracts at the same time on a given day.

9.4 Low Level Extraction LCS:

9.4.1 Add 0.5 to 1.0 g of NaCl to a 10 mL headspace vial.

9.4.2 Add 5 mL of DI water to a salted headspace vial.

9.4.3 Using a 25 uL Hamilton syringe, add 5 uL of the screening IS standard.

9.4.4 Add 5 uL of the MSVOA-LCS standard.

9.4.5 Cap tightly.

9.5 Medium Level LCS:

9.5.1 Weigh 5.0 g pre-baked Ottawa Sand in to a 20 mL screw cap vial.

9.5.2 Add 5.0 mL purge and trap methanol, 4.0 µL MSVOA-LCS at 2,500 ug/mL, and 40. µL MSVOA-SCS at 250 ug/mL. Note that different QC codes may have different spiking requirements. Be sure to check the instructions for each lot.

9.5.3 Shake for two minutes and centrifuge as necessary.

9.5.4 Save the LCS extract in two 2.0 mL screw top vials that are labeled to indicate the lot number and that they are LCS samples.

9.5.5 Some clients may require Duplicate LCS. In those circumstances, prepare a LCS duplicate with the LCS.

9.6 The results of the method blank and LCS are strictly for internal reference to ensure that the screening results are reliable enough for the lab’s own screening purposes. Strict SW-846 type quality control acceptance criteria do not apply.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Low Level Extraction Samples:

10.1.1.1 Add 0.5 to 1.0 g of NaCl to a 10 mL headspace vial.

10.1.1.2 Add 5 mL of sample to a salted headspace vial.

10.1.1.3 Using a 25 uL Hamilton syringe, add 5 uL of the screening IS standard.

10.1.1.4 Cap tightly.

10.1.2 Low-level Extraction of Industrial Soils:

Note: If sample is not homogenous, mix with spatula to ensure homogeneity before use.

10.1.2.1 Weigh 2.0 to 2.5 g soil sample designated for screen analysis into a prebaked salted headspace vial.

10.1.2.2 Add 5 mL reagent water.

10.1.2.3 Add 5 uL screening IS (at 200 ug/mL). Cap tightly.

10.1.3 Medium-level Extraction of Solids and Wastes:

Note: If sample is not homogenous, mix with spatula to insure homogeneity before use.

10.1.3.1 Weigh 4.95 - 5.05 g soil designated for screen analysis into a 20 mL vial. Record in log book.

10.1.3.2 Add 4.95 mL methanol (add 4.90 mL methanol depending on method code). Recap tightly.

10.1.3.3 Add 4.0 uL MSVOA-SCS at 2,500 ug/mL.

10.1.3.4 Shake vigorously by hand for ~2 minutes.

10.1.3.5 Centrifuge at setting #4 for 3 to 5 minutes to separate solid matter.

10.1.3.6 If the sample absorbs a significant amount of methanol and there is less than 3 mL of free methanol:

- repeat the extraction with increasing amounts (5 mL increments) of methanol until there is enough methanol to fill at least one vial with no headspace plus a second vial one-half full
- add additional MSVOA-SCS standard in the proportion of 4 µL per 5 mL of methanol
- record the amount of methanol and standard in the logbook

10.1.3.7 Carefully draw the extract off the top, trying not to disturb the lower solid layer. Transfer the extract to two 2 mL screw top vials that have been labeled and taped (for GC/MS analysis). Fill vial A to the top, leaving as little headspace as possible. Fill vial B at least ½ full.

10.1.3.8 One vial is used for the screening analysis. The second is used for the determinative analysis by GC/MS.

10.1.4 Extraction of oils:

10.1.4.1 If miscible: Use 1 mL volumetrically, record the exact weight, and then add 4 mL of methanol.

10.1.4.2 If not miscible: Use approximately 1 g of sample and add 5 mL of methanol

10.2 Calibration

None

10.3 Sample Analysis

10.3.1 GC conditions temperatures and temperature programs are listed in Table 4.

10.3.2 19395A Headspace Sampler, Tekmar 700HT conditions are listed in Table 5.

10.3.3 Multichrom analysis

10.3.3.1 Determine sample order for each column and record it on the instrument log.

10.3.3.2 It is important that the analyst refer to the Multichrom Reference Manual Section 5, “Run Sequence File," pp. 77-113.

10.3.3.3 All runs will begin with a blank followed by the primary standard.

10.4 Start the run

10.4.1 Make sure the autosampler is in “Auto” mode. Set the autosampler to run the desired vial range by hitting the “A/S” button and changing the start and stop values as needed.

10.4.2 Make sure the instrument purges for A & B are off.

10.4.3 Make sure remote switches on Headspace Sampler on.

10.4.4 In the HP ChemStation software, from the “Sequence” pull-down menu, load the sequence file for the correct day and change the filename for the data file to the current day.

10.4.5 Under the sequence table option, enter the sample IDs in the sequence. For instrument I, choose the “8260SCRE” method. Choose “TEKMAR2” for instrument T.

10.4.6 After the sample IDs have been entered into the sequence and the vials are loaded on the autosampler, hit the "Start" button and then press "OK" to start the run.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

11.1.1 All identification is based on retention time. The internal standards (ISs) will reflect any fluctuations in retention time between runs.

11.1.2 Retention time windows for the first eight compounds (1,1-dichloroethene through carbon tetrachloride) are 3%. Windows for remaining compounds are 2.5%. Retention times must be reviewed daily with each daily calibration, updating the method if necessary. If changes are made, be sure to process samples with the updated method.

11.1.3 Review the daily calibration standard to insure that all peaks have been identified correctly. Except after column change or maintenance, retention times and areas should be similar to the previous daily calibration.

11.2 Estimating Dilution Levels Based on Screening Results

VOA screening analysis establishes the approximate concentration of target analytes to determine a dilution, which will place the highest concentration target compound at a level of 30 µg/mL or non-target compounds at a level of 60 µg/mL.

The objective of this process is to use a dilution factor that will put the GC/MS analytical results in the middle of the upper range of the calibration. Since the FID response is variable for the target compounds, the screening system is more effective for some classes of compounds than others.

The GC/MS analyst should evaluate one sample in a project at the recommended dilution determined from the following steps. This can be useful to evaluate screening information for other samples in the project.

11.2.1 Estimating Dilution Levels for Different Classes of Compounds

11.2.1.1 Since the FID response is variable for the target compounds, the screening system is more effective for some classes of compounds than others.

11.2.1.2 System is effective for Benzene, Toluene, Xylenes, and most hydrocarbons (good responders).

11.2.1.3 System is moderately effective for chlorinated compounds (moderate responders).

11.2.1.4 System is poor for ketones and trihalomethanes (poor responders).

11.2.1.5 System is not effective for acetone (does not extract well).

11.2.1.6 Dilutions based on moderate and poor responders can lead to over-dilution if the peak identifications are not correct. Because the identifications for bromoform, dibromochloromethane, and bromodichloromethane are not reliable with this screening method, dilutions should not be based on these compounds. See Table 6 for reduction of data.

11.2.2 Estimating Dilution Level Needed for Low-Level Waters

11.2.2.1 Determine the dilution factor by dividing the concentration of the highest analyte until it is approximately 30-45 ug/mL. For example, if a compound is present at 300 ug/mL, a 10x dilution is needed, and this will be obtained by diluting 2.0 mL of sample to 20 mL for the final analysis.

11.2.2.2 Unknowns are calculated using the response factor of 1,1-DCE for unknowns that elute before benzene. Meta- and para-xylenes are used for everything else. If any unknown is greater than the highest concentration target, the dilution recommendation is based on the unknown using the same calculation as above. If the identification of chlorinated compounds like chloroform or 2-chloroethyl vinyl ether is suspect, recalculate the compound as an unknown and present both results.

11.2.2.3 If no targets or unknowns are >20 ug/L in the sample, the recommended volume is 20 mL or 100%.

11.2.2.4 If the screen recommendation is <0.1 mL, serial dilute the sample and rescreen. See Table 6.

11.2.3 Estimating Dilution Levels for Low-level Soils

11.2.3.1 Use the same techniques and calculations as water samples in the previous section, substituting g for mL.

11.2.3.2 If screen indicates less than 0.5 g needed for industrial, or <1.0 g for AFCEE, prepare a medium level extract. See Table 6.

11.2.4 Estimating Dilution Levels for Medium-level Solids and Wastes

11.2.4.1 These extractions use 5 grams of soil in 5 mL of methanol. The optimum sample size for GC/MS = 0.100 mL methanol extract in 5 mL water (0.100 mL 5000 ug/L = 5.0 mL 100 ug/L)

11.2.4.2 Techniques and calculations are similar to water samples, except divide the highest concentration of a target compound by 2. The result is equal to the recommended volume of extract to add to 5 mL of water for GC/MS analysis.

11.2.4.3 Unknowns are calculated using the response factor of 1,1-DCE for unknowns that elute before benzene. Meta- and para-xylenes are used for everything else. If any unknown is greater than the highest concentration target, the dilution recommendation is based on the unknown using the same calculation as above. If

the identification of chlorinated compounds like chloroform or 2-chloroethyl vinyl ether is suspect, recalculate the compound as an unknown and present both results.

11.2.4.4 If the screen recommendation is <5 uL, serial dilute the methanol extract and rescreen. See Table 6.

11.3 Reporting Requirements

11.3.1 Screening results are not entered into the LIMS system.

11.3.2 The record in LIMS must be completed and released on the same day that samples are extracted. This is important for medium level methanol extractions because the test completion date is equal to the preparation test for GC/MS VOA tests. If a sample is re-extracted at a later date, the screen test completed test must be changed to reflect the latest extraction date.

11.3.3 All data should be analyzed, reduced, and reviewed by the day after extraction. Data from each project analyzed is grouped into separate folders.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

There is no MDL study for a screening process.

12.2 Demonstration of Capabilities

Manual Initial Demonstration of Proficiency (IDOC) forms are used to document analyst proficiency initially and on-going on an annual basis.

12.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.0 Pollution Control

The volumes of methanol used for the medium-level extraction are kept as small as practical to minimize the generation of flammable and poisonous waste.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Environmental Health and Safety Manual for “Waste Management and Pollution Prevention.”

14.1 Waste Streams Produced By This Procedure

The following waste streams are produced when this method is carried out.

- Methanol extract vial waste – Expired Extract Vials (A)

Note: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, and method revisions through Final Update IV, 2007. Determinative Chromatographic Separations, Methods 8000B, 8000C, and Nonhalogenated Organics by Gas Chromatography, Methods 8015B, 8015C and 8015D.

15.2 Manuals for Hewlett Packard Headspace Autosampler 19395A and Tekmar Dohrmann Headspace Autosampler 7000HT.

16.0 Method Modifications:

There are none for this procedure.

17.0 Attachments

Table 1: Analytes Determined by This Method
Table 2: MSVOA-SCS
Table 3: MSVOA-LCS
Table 4.1: GC Operating Conditions for Method 8260 SCR.M
Table 4.2: GC Operating Conditions for Method TEKMAR2.M
Table 5: GC and Sampler Conditions
Table 6: Dilution Determination from Screen Results
Attachment 1: Instrument Maintenance

18.0 Revision History

- Revision 3.4, dated May 31, 2012
  - Annual Technical Review
  - Grammatical, spelling and formatting changes throughout
- Revision 3.3, dated May 31, 2011
  - Revised the target weight for low-level extraction of industrial soils
  - Corrected table references
  - Expanded references to include method updates to SW-846
  - Grammatical, spelling and formatting changes throughout
- Revision 3.2, dated May 31, 2010
  - Annual review, only clerical changes made.
- Revision 3, dated 29 February 2008

- Integration for TestAmerica and STL operations.
- Method was updated to match current practices (throughout the SOP).
- Tables and attachments updated to current practice.

- Revision 2, dated 06 October 2004
  - STL Corporate Safety and Waste Management requirements are added to Sections 5 and 15, respectively.
  - Instrument maintenance is described in Attachment A.
  - The volumes and concentrations of spike mixes are changed.
  - QC sample instructions are moved to the QC section.
  - The operating conditions and instructions are given for current instrumentation, which are different than in the previous revision.
  - The target concentration for dilutions is changed to a lower concentration to match the current working range for our 8260B procedures.
  - Instructions about IDOCs documentation for screening analysts are added to Section 13.
- Revision 1, dated 11 February 2002
  - Changes from previous revision include reformatting for STL requirements, and inclusion of instrument conditions for a second screening instrument.

Table 1.

Analytes Determined by This Method

1,1,1-Trichloroethane	Chlorodibromomethane
1,1,2,2-Tetrachloroethane	Chloroform
1,1,2-Trichloro-1,1,2-trifluoroethane	Cis-1,2-dichloroethene
1,1,2-Trichloroethane	Cis-1,3-dichloropropene
1,1-Dichloroethane	Dibromoethane
1,1-Dichloroethene	Ethanol
1,2,3-Trichloropropane	Ethyl methacrylate
1,2-Dibromoethane	Ethylbenzene
1,2-Dichlorobenzene	Hexane
1,2-Dichloroethane	Iodomethane
1,2-Dichloropropane	m-Xylene
1,3-Dichlorobenzene	Methyl tert-butyl ether
1,4-Dichlorobenzene	Methylene chloride
1,4-Dioxane	Naphthalene
2-CLEVE	o-Xylene
2-Butanone	p-Xylene
2-Hexanone	Styrene
4-Methyl-2-pentanone	t-Butanol
Acetone	Tetrachloroethene
Benzene	Tetrahydrofuran
Bromodichloromethane	Toluene
Bromoform	Trans-1,2-dichloroethene
Carbon Disulfide	Trans-1,3-dichloropropene
Carbon tetrachloride	Trans-1,4-dichloro-2-butene
Chlorobenzene	Trichloroethene

Table 2.

MSVOA-SCS

250 ug/mL of following components in methanol

1,2-Dichloroethane-d4
4-Bromofluorobenzene (BFB)
Toluene-D8
Dibromofluoromethane

Table 3.

MSVOA-LCS

200 ug/mL of following components in methanol

Toluene
Trans-1,2-Dichloroethene
Trans-1,3-Dichloropropene
Trichloroethene
1,1-Dichloroethane
1,1-Dichloroethene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
1,1,2,2-Tetrachloroethane
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloropropane
1,3-Dichlorobenzene
1,4-Dichlorobenzene
2-Chloroethyl Vinyl Ether
Benzene
Bromodichloromethane
Bromoform
Carbon Tetrachloride
Chlorobenzene
Chloroform
Cis-1,3-Dichloropropene
Dibromochloromethane
Ethylbenzene
Methylene Chloride
Tetrachloroethane

Table 4.1.

GC Operating Conditions for Method 8260 SCR.M

TABLE 4.1
Gas Chromatograph Operating Conditions
8260SCRE.M

Run Time Checklist			
Pre-Run Cmd/Macro	Off		
Data Acquisition	On		
Standard Data Analysis	Off		
Customized Data Analysis	Off		
Save GLP Data	Off		
Post Run Cmd/Macro	On		
Name	macro c:\gc_Itran.mac, GO		
Save Method with Data	Off		
Injection Source and Location			
Injection Source	Manual		
Injection Location	Front		
Oven/Det			
Runtime (min):	15.9		
Zone Temperatures			
	State	Setpoint (°C)	
Inlet A	Off	220	
Inlet B	Off	220	
Detector A	On	300	
Detector B	On	300	
Aux	Off	50	
Oven Zone			
Oven max	400 °C		
Equib Time	0.1 min		
Oven State	On		
Cryo State	Off		
Ambient	25 °C		
Cryo Blast	Off		
Oven Program			
	Setpoint		
Initial Temp	50 °C		
Initial Time	0.00 min		
Level	Rate (°C/min)	Final Temp (°C)	Final Time (min)
1	8.00	120	0.00
2 (A)	20.0	260	0.20
Purge Valve Settings			
Purge A/B			
	Init Value	On Time (min)	Off Time (min)
A (Valve 3)	Off	0.00	0.00
B (Valve 4)	Off	0.00	0.00
A - Splitless Injection	Yes		
B - Splitless Injection	Yes		
Valves/Relays Information			
Initial Setpoints			
5890 Valves			
	Valve 1	Off	
	Valve 2	Off	
	Valve 3 (Purge A)	Off	
	Valve 4 (Purge B)	Off	
Detector Information			
Detector A:			
Type	FID		
State	On		
Detector B:			
Type	FID		
State	On		
Signal Information			
Save Data	Signal 1		
Signal 1			
Signal	Det. A		
Data Rate	20.000 Hz		
Peakwidth	0.013 min		
Start Time	1.00 min		
Stop Time	35.00 min		
Signal 2			
Signal	Det B		
Data Rate	20.000 Hz		
Peakwidth	0.013 min		
Start Time	1.00 min		
Stop Time	35.00 min		

Table 4.2.

GC Operating Conditions for Method TEKMAR2.M

TABLE 4.2
Gas Chromatograph Operating Conditions
TEKMAR2.M

Run Time Checklist			
Pre-Run Cmd/Macro	Off		
Data Acquisition	On		
Standard Data Analysis	Off		
Customized Data Analysis	Off		
Save GLP Data	Off		
Post Run Cmd/Macro	On		
Name	macro c:\gc_Ttran.mac, GO		
Save Method with Data	Off		
Injection Source and Location			
Injection Source	Manual		
Injection Location	Front		
Oven/Det			
Runtime (min):	15.9		
Zone Temperatures			
	State	Setpoint (°C)	
Inlet A	On	220	
Inlet B	Off	50	
Detector A	On	330	
Detector B	Off	50	
Aux	Off	50	
Oven Zone			
Oven max	400 °C		
Equib Time	0.1 min		
Oven State	On		
Cryo State	Off		
Ambient	25 °C		
Cryo Blast	Off		
Oven Program			
	Setpoint		
Initial Temp	50 °C		
Initial Time	0.00 min		
Level	Rate (°C/min)	Final Temp (°C)	Final Time (min)
1	8.00	120	0.00
2 (A)	20.0	260	0.20
Purge Valve Settings			
Purge A/B			
	Init Value	On Time (min)	Off Time (min)
A (Valve 3)	Off	0.00	0.01
B (Valve 4)	Off	0.00	0.01
A - Splitless Injection	No		
B - Splitless Injection	No		
Valves/Relays Information			
Initial Setpoints			
5890 Valves			
	Valve 1	Off	
	Valve 2	Off	
	Valve 3 (Purge A)	On	
	Valve 4 (Purge B)	On	
Detector Information			
Detector A:			
Type	FID		
State	On		
Signal Information			
Save Data	Signal 1		
Signal 1			
Signal	Det. A		
Data Rate	20.000 Hz		
Peakwidth	0.013 min		
Start Time	1.00 min		
Stop Time	650.00 min		
Signal 2			
Signal	Testplot		
Data Rate	5.000 Hz		
Peakwidth	0.053min		
Start Time	0.00 min		
Stop Time	650.00 min		

Table 5.
GC and Sampler Conditions

GC	Conditions
Sample Loop	1 mL Loop
Platen Temperature	85°C
Platen Equilibration Time	0 minute
Sample Equilibration Time	30 minutes
Vial size	22 mL
Mixer	On
Mixing Time	2 minutes
Mix Power	5
Stabilize Time	2 minutes
Cryo Cool down / Minutes at	NI
Pressurize Setting	6 psi at 40 mL/min
Pressurize Time	0.3 minute
Pressurize Equilibration Time	0.05 minute
Loop Fill Time	0.3 minute
Loop Equilibrium Time	0.05 minute
Inject	1 minute
Cryo Inject / Minutes at	NI
Valve Temperature	85°C
Line Temperature	85°C
Cryo Union Heater	NI
Injections Per Vial	1
GC Cycle Time	42 minutes
Parameter Optimization	Off
Detector	FID
Column	DB-624, 60M, 0.53 um
Carrier Flow	6 cc/min
Initial Temperature	35°C for 5 minutes
Rate	3°C/min to 100°C
Hold Time	1 minute

Tekmar 7 - HT	Sampler Conditions
Platen plate	85°C
Sample Loop	200°C
Sample Line	200°C
Sample Equilibration	7 minutes
Pressurize	0.5 minute
Pressurize Equilibration	0.1 minute
Loopfill time	0.5 minute
Loop Equilibration	0.2 minute
Inject	0.5 minute

Table 6.

Dilution Determination from Screening Results

The following table lists minimum and maximum sample size for various VOA tests based on results of the screening analysis.

TYPE	MATRIX	PREP	GC/MS MIN RUN	GC/MS MAX RUN	COMMENTS
Industrial	Water	LL-1.0 mL/5.0mL	DILN	20 mL	If <0.1 mL, serial dilute
Industrial	LL Soil	LL-1.0 g/5.0 mL	1.0 g	5.0 g	If <1 g, go to ML prep
Industrial	ML soil	ML-5.0 g/5.0 mL	DILN	100 uL	Standard Prod. If <5uL, serial dilute
Industrial	Waste	ML-5.0 g/5.0 mL	DILN	100 uL	If <5 uL, serial dilute
Industrial	TCLP	LL-100 uL/5.0 mL	DILN	1.0 mL	May do oil and water phase
AFCEE	Soil	ML-5.0 g/5.0 mL	1.0 g	5.0 g	If <1.0 g, go to ML prep
AFCEE	Soil	ML-5.0 g/5.0 mL	DILN	500 uL	If <5 uL, serial dilute

LL = Low level
ML = Medium level

Attachment 1.

Instrument Maintenance (Part 1)

1.0 Semi-annual Instrument Maintenance

1.1 Change septa. See HP instrument manual for instructions

1.2 Check flows. Optimum flows are:

Helium 12 mL/min
Nitrogen 10 mL/min
Air 370 mL/min
Hydrogen 30 mL/min
Adjust if necessary.

1.3 Cut the column

1.3.1 Remove fitting on injection port end of column.

1.3.2 Unwrap column one revolution and using a glass scorer, cut ~2 inches of column from injection port end. Inspect the cut end to be sure the cut was clean and square, leaving no jagged ends.

1.3.3 Replace the connecting nut and place a new 0.8 mm graphite/vespel ferrule on the end flush with the nut.

1.3.4 Using a ruler, measure 1 cm from the cut end of the column to the top of the nut thread. Mark the column under the nut so that when it is put in place in the injection port liner, the column amount measured extends up into the injection port liner. Turn the connecting fitting nut finger tight, then turn 3/4 to a full turn with a 3/16" wrench.

1.3.5 When cutting the detector end of the column, follow steps 1.3.1 through 1.3.3, applying to detector end.

1.3.6 When reconnecting detector end, feed column into detector as far as it will go, connect with nut, and withdraw 0.5 cm column out. Finish connection as in 8.2.4.4.

Attachment 1.

Instrument Maintenance (Part 2)

1.4 Annual Instrument Maintenance

1.4.1 Lower injection port temperatures and detector temperatures to ambient

1.4.2 Change injection port liners

1.4.2.1 Remove H.S. transfer lines. Be careful not to bend needle end. **CAUTION** - transfer line is hot.

1.4.2.2 Remove lower injection port nut

1.4.2.3 Carefully remove injection port liner and replace with freshly silanized liner.

1.4.2.4 Reassemble

1.4.3 Silanizing injection port liners

1.4.3.1 Place dirty injection port liners in acid soak for 1 hour minimum. Remove.

1.4.3.2 Rinse first with cold water, then hot water, then liberally with acetone, hexane, methylene chloride, and methanol. Let dry thoroughly.

1.4.3.3 Soak injection port liners in a 10% solution of Hexamethyldisilazane in Hexane for a minimum of 8 hours. Remove, rinse with 200-300 mL Hexane, and let dry.

1.4.4 Visually inspect FIDs for contamination by removing housing. If dirty, remove parts and sonicate in applicable solvent. Reassemble.

1.4.5 Restore operating temperatures.

Helpful hints: If normal signals of 2 to 5 cannot be achieved after maintenance, bake oven at 260^{O}C for 1-2 hours, then run 2 to 3 solvent blanks. This usually restores the signal.

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Title: Determination of Volatile Organics by GC/MS [8260B and 624]

Approvals (Signature/Date):

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1.0 Scope and Application

1.1 This method is applicable to the determination of volatile organic compounds (VOCs) in water, wastewater, soils, sludges, and other solid matrices. Standard analytes are listed in Table 1. Additional analytes that can be determined by this SOP are listed in Tables 2, 3 and 4.

1.2 This SOP is applicable to Method 8260B, which is appropriate for compliance testing under RCRA regulations and Method 624 (CWA compliance testing). It is important that the procedural differences described in this document for these methods are carefully observed.

1.3 Appendix A of this SOP contains the modifications needed to run the instrument in the selected ion monitoring mode.

1.4 This method can be used to quantify most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.5 The method is based upon a purge-and-trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 0.5 to 60 μg/L for 8260B waters, 2.5 to 200 μg/kg for low-level soils, and 200 to 30,000 μg/kg for medium-level soils. The working range for Method 624 (5 mL purge) is 5-200 μg/L.

1.6 Reporting limits for Method 8260B are listed in Tables 1, 2, and 3. Reporting limits for Method 624 and 8260B SIM are given in Table A1 and Table Ap-1, respectively. Reporting limits for soil samples prepared by the AK methanol technique are listed in Table Bp-1.

1.7 Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates (MS/MSD), and laboratory control spike samples (LCS).

2.0 Summary of Method

2.1 Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the gas chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2 Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be preserved in water (with or without sodium bisulfate) and purged directly.

2.3 In the purge-and-trap process, an inert gas is bubbled through the solution at ambient temperature or at 40 °C (40 °C is required for low-level soils), and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column (trap) is heated and backflushed with inert gas to desorb

the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

2.4 Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3.0 Definitions

3.1 Terms

The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and in SOP DV-QA-003P, Quality Assurance Program.

3.2 Calibration Check Compound (CCC)

CCCs are a representative group of compounds that are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and percent drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.3 System Performance Check Compounds (SPCC)

SPCCs are compounds that are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

3.4 Initial Calibration Verification (ICV)

The ICV is a second-source calibration verification standard. In this SOP, the LCS and the MS/MSD spikes are second-source standards.

3.5 Continuing Calibration Verification (CCV)

A solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

3.6 Selected Ion Monitoring (SIM)

Operation of the mass spectrometer in the selected ion monitoring mode to optimize the quantitative information at the expense of qualitative information gained from other methods of analysis.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must

be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge-and-trap-grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3 Matrix interferences may be caused by non-target contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5 Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample. (See Section 10.7.4.12.)

4.6 Interferences are observed with the surrogate Toluene-d_8 when the samples appear to be treated with potassium permanganate.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.3.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.3.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.1.2 Sample Purger: The recommended purging chamber is designed to accept between 5 mL and 25 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.1.3 Trap: A variety of traps may be used, depending on the target analytes required. The O.I. #10 (Tenax / Silica gel / Carbon Molecular Sieve) is recommended. Other traps such as the Vocarb 3000 or Vocarb 4000 may be used if the Quality Control criteria are met.

6.1.4 Desorber: The desorber should be capable of rapidly heating the trap up to 270 °C depending on the trap packing material. Many such devices are commercially available.

6.1.5 Sample Heater: A heater capable of maintaining the purge device at 40 °C is necessary for low level soil analysis.

6.1.6 Purge-and-trap Autosampler: An autosampler capable of sampling from a sealed vial, Varian Archon, or equivalent.

6.1.7 Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.1.8 Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.1.8.1 Column 1: 60 m X 0.25 ID DB-624 with 1.4 µm film thickness.

6.1.8.2 Column 2: 75 m X 0.53 ID DB-624 wide bore with 3 µm film thickness.

6.1.9 Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 amu every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.1.10 GC/MS interface: In general, glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.

6.1.11 Data System: A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. In addition, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.2 Computer Software and Hardware

6.2.1 Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

6.3 Supplies

6.3.1 Microsyringes: 10 µL and larger, 0.006-inch ID needle.

6.3.2 Syringe: 5 or 25 mL glass with Luerlok tip, if applicable to the purging device.

6.3.3 Balance: Analytical balance capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.3.4 Vials: 2 mL, 20 mL, and 40 mL with screw caps and Teflon liners

6.3.5 Disposable magnetic stirrers for low-level soil analyses

6.3.6 Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.3.7 Spatula: Stainless steel.

6.3.8 Disposable pipettes: Pasteur.

6.3.9 pH paper: Wide range.

6.3.10 Gases:

6.3.10.1 Helium: Ultra high purity, grade 5, 99.999%.

6.3.10.2 Compressed nitrogen: Used for instrument pneumatics.

7.0 Reagents and Standards

7.1 Methanol: Purge and Trap Grade, High Purity

7.2 Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See Section 9.3.) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.3 Sand: Reagent grade Ottawa sand or equivalent.

7.4 Antifoam B, Silicon Emulsion, J. T. Baker, 100% purity.

7.5 Sodium bisulfate ($NaHSO_4$), reagent grade

7.6 If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers' expiration date.

7.7 Calibration Stock Standard Solutions

Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10 to -20 °C. Stock standards and aliquots for gases must be replaced at least every week. The Gas Standards Tracking Log is used to verify track open dates to assist in weekly replacement of the gas standards. See Attachment 1. Other stock standards must be replaced at least every 6 months.

7.8 Calibration Working standards

A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20%, then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

7.9 Aqueous calibration standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.10 Internal standards (IS) are added to all samples, standards, and blank analyses. Refer to Tables 7 and 7A for internal standard components.

7.11 Surrogate Standards: Refer to Tables 8 and 8A for surrogate standard components and spiking levels.

7.12 Laboratory Control Sample Spiking Solutions: Refer to Table 10 for LCS components and spiking levels.

7.13 Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 10.

7.14 Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 50 ng/µL of BFB in methanol is prepared from stock standards as described in Sections 7.7 and 7.8.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples

8.1.1 Water samples are collected in triplicate in 40 mL glass VOA vials with PTFE-lined septum caps with minimal headspace. There should be no bubbles present in the container larger than ~6 mm.

8.1.2 Preservation depends upon the target analytes and the sampling location. At a minimum, aqueous samples are stored refrigerated at ≤ 6 ^{o}C and not frozen. Specific preservation requirements are given in the following table. If multiple analytes are requested, it may be necessary to provide aliquots with different preservations. For each preservation technique, the samples should be collected in triplicate.

8.1.3 The State of Colorado Attorney General's office issued a letter on July 1, 1998 requiring that all samples collected for analysis of volatile organic compounds in groundwater must be collected without acid preservation. The letter explains that this is done to avoid effervescence with alkaline samples and loss of volatiles. The letter also explains that the holding time for unpreserved ground waters is 14 days.

8.1.4 SW-846 states that if carbonaceous materials are present, or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. The holding time for these unpreserved samples is 7 days. SW-846 does not otherwise provide guidance for processing unpreserved samples. EPA MICE has interpreted the holding time on an unpreserved sample as 7 days.

Preservation and Holding Time for Volatiles in Water

Analyte(s)	Reference	Preservation[1]	Holding time	Dechlorination Required[2]
Routine target analytes[3]	SW-846, Ch. 4	Cool, ≤6ºC, pH < 2 with 1:1 HCl	14 days	Y
	SW-846, Ch. 4	Cool, ≤6ºC	7 days	Y
	624	Cool, ≤6ºC, pH < 2 with 1:1 HCl	14 days	Y
	624	Cool, ≤6ºC	7 days	Y
Acrolein[4]	SW-846, Ch. 4	Cool, ≤6ºC, pH 4-5	7 days	N
	603	Cool, ≤6ºC (no HCl)	3 days	Y
	603	Cool, ≤6ºC, pH 4-5	14 days	Y
Acrylonitrile[4]	SW-846, Ch. 4	Cool, ≤6ºC, pH 4-5	7 days	N
	603	Cool, ≤6ºC (no HCl)	14 days	Y
	603	Cool, ≤6ºC, pH 4-5	14 days	Y
2-Chloroethylvinyl ether (2-CLEVE)[5]	SW-846, Ch. 4	Cool, ≤6ºC (no HCl)	7 days	Y
	624	Cool, ≤6ºC (no HCl)	14 days	Y

[1] See Section 8.1.3 for samples collected in Colorado and Section 8.1.4 for samples to be analyzed by Method 8260B that are unpreserved.
[2] If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added
[3] Separate aliquots must be collected and preserved as indicated if acrolein, acrylonitrile, 2-CLEVE (by Methods 8260B or 624), vinyl chloride (by Method 8260B) or styrene (by Method 8260B) are also to be analyzed. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary.
[4] According to the source methods, the preferred method for acrolein and acrylonitrile is Method 603. In the Method Update Rule published in the Federal Register on May 18, 2012 (40 CFR Parts 136, 260, et. al.) EPA approved Method 624 for the determination of acrolein and acrylonitrile in wastewater. The current sample preservation and holding time requirements for acrolein and acrylonitrile apply to these compounds when analyzed by Method 624. Implementation of this rule is subject to individual state program decisions and timetables.
[5] SW-846 includes vinyl chloride and styrene in the list of compounds which require unpreserved sample for analysis. Method 624 does not include these two analytes on the standard analyte list.

8.2 Soil Samples

8.2.1 Soil samples can be taken using the EnCore™ sampler. Typically three Encores are collected per sampling location. At specific client request, unpreserved soil samples may be accepted for preservation at the lab.

8.2.1.1 Samples sent in the EnCore™ sampler to the lab for preservation must be preserved within 48 hours of sampling. They are preserved by extruding one sample into a clean VOA vial containing methanol for medium level analysis. The remaining two samples are extruded into vials containing water or sodium bisulfate ($NaHSO_4$) and water for low level analysis.

8.2.1.2 Samples are stored frozen after transfer from the EnCore™ sampler.

8.2.2 The more common way to collect soils is with Terra Core kits. Typically three aliquots are collected. Terra Core kits consist of the Terra Core sampling device and three 40 mL tared VOA vials. There are several ways to preserve the samples once sampled.

8.2.2.1 The samples collected with the Terra Core sampling device are extruded into empty vials and frozen in the field. The lab freezes the samples on receipt. These samples have a 14 day holding time from sampling.

8.2.2.2 The samples can be extruded into empty vials and shipped to the lab refrigerated. The lab freezes the samples within 48 hours of collection and the holding time is extended to 14 days from collection. The lab has the option to prepare the samples upon receipt by the addition of methanol to one vial and water or sodium bisulfate ($NaHSO_4$) and water to the remaining two vials. The samples are then refrigerated. The holding time for this latter preservation is 14 days from sampling.

8.2.2.3 Alternatively, the project team can request for each sample one tared vial containing methanol for medium level analysis and two tared vials containing water or sodium bisulfate and water, depending upon project requirements. An aliquot of the sample is extruded into each prepared vial while in the field and shipped on ice. The samples are refrigerated upon receipt at the laboratory. The holding time is 14 days from sampling for this field preservation technique.

8.2.3 Unpreserved Soils

8.2.3.1 At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the laboratory. This is the old procedure based on Method 5030A. It is no longer included in subsequent revisions of Method 5030 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.2.3.2 The maximum holding time is 14 days from sampling until the sample is analyzed. Unpreserved samples should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative.

8.2.4 An additional bottle of unpreserved soil for each sampling location must be shipped for percent moisture determination.

8.2.5 A second bottle of unpreserved soil is sent for screening.

8.2.6 Preservation and holding times for volatiles in soils are summarized in the following table, based on SW-846 Method 5035A. The "Coring Tool" listed in the container column may be the EnCore™ or Terra Core sampler.

Preservation and Holding Time for Volatiles in Soil
Method 5035A

Container/Contents[1]	Preservation	Holding time	Analysis
Empty Sealed Vial	Freeze on-site to -7°C (do not freeze below -20°C)	14 days	Low Level
Empty Sealed Vial	Cool to ≤ 6 °C	48 hours	Low Level
Empty Sealed Vial	Cool to ≤ 6 °C for no more than 48 hours Frozen upon receipt at lab (< -7 °C, do not freeze below -20 °C)	14 days	Low Level
Empty Sealed Vial	Cool to ≤ 6 °C for no more than 48 hours Preserved with methanol upon receipt at lab	14 days	Medium Level
Encore™ sampler used for transport	Cool to ≤ 6 °C or Freeze to < -7 °C in field	48 hours	Low or medium level
Encore™ sampler used for transport	Cool to ≤ 6°C or freeze to < -7°C in field and upon receipt at lab extruded to a sealed vial and either frozen to < -7°C or chemically preserved	14 days	Low or medium level
Vial containing reagent water	Sample is extruded into vial and frozen to < -7°C in field and maintained frozen upon receipt by the laboratory.	14 days	Low level
Vial containing reagent water	Sample is extruded into vial and cooled to ≤ 6°C in field then frozen to < -7°C upon laboratory receipt (within 48 hours of sampling).	14 days	Low level
Vial containing reagent water and 1 g $NaHSO_4$[2]	Sample is extruded into vial with preservative and cooled to ≤ 6°C. Stored at ≤ 6°C upon laboratory receipt.	14 days	Low Level
Vial containing methanol	Sample is extruded into vial with preservative, cooled to ≤ 6°C and frozen upon receipt at laboratory.	14 days	Medium Level

[1] For biologically active soils, immediate chemical or freezing preservation is necessary due to the rapid loss of BTEX compounds within the first 48 hours of sample collection.

[2] Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible

8.3 Trip blanks, consisting of laboratory prepared water samples with acid preservative, are also provided when bottles are supplied by the laboratory to the field. Trip blanks are used for both water and soil samples to monitor potential contamination from volatile compounds in transit and in the field.

8.4 A holding blank is stored in each refrigerator with the samples. This is analyzed every 7 – 14 days (see SOP DV-QA-0013).

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples (method blank, lab control sample, and matrix spike/matrix spike duplicate), processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. A method blank must be run on each instrument. See Policy DV-QA-003P for further details.

9.3 Method Blanks

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles in water, the method blank consists of reagent water. For low-level volatiles in soil, the blank medium is Ottawa sand. For medium-level volatiles, the method blank consists of 5.0 mL of methanol. Surrogates are added and the method blank is carried through the entire analytical procedure.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above one-half the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

The method blank must have acceptable surrogate recoveries. (See Section 9.4)

Corrective Actions: If the analyte is a common laboratory contaminant (i.e., methylene chloride, acetone, 2-butanone), the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the associated samples.

If there is no target analyte greater than one-half the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

If surrogate recoveries in the blank are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in the narrative to provide further documentation.

9.4 Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within

established limits. The compounds included in the surrogate spiking solutions are listed in Tables 8 and 8A.

Acceptance Criteria: Acceptance limits for surrogate recoveries are set at ± 3 standard deviations around the historical mean. Surrogate recovery limits are updated semi-annually and stored in the LIMS.

Corrective Actions: If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Re-prepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is necessary to re-prepare/reanalyze a sample only once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and re-preparation/reanalysis is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.5 Laboratory Control Samples (LCS)

An LCS is analyzed for each batch. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS spiking solution is prepared from a different source than are the calibration standards. The LCS contains a representative subset of the analytes of interest (See Table 10), and must contain the same analytes as the matrix spike. For low-level volatiles in water, the LCS matrix is reagent water. For low-level volatiles in soil, the LCS matrix is Ottawa sand.

Acceptance Criteria: The LCS recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. An LCS that is determined to be within acceptance criteria effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch. Recovery limits are updated semi-annually and stored in the LIMS

If there are a large number of analytes in the LCS, then a specified number of results may fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. Marginal exceedances are recognized and allowed by NELAC, AFCEE, and the DOE. DoD requires individual project approval for the use of marginal exceedances. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Note: Additional criteria are stated in the North Carolina QAS.

Note: Some programs (e.g., South Carolina) do not allow marginal exceedances. Please see the QSAS's in the public folders for the current requirements.

Corrective Actions: If any analyte or surrogate is outside established control limits as described above, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and reanalysis of the batch.

If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.

If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported,

all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 10. The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

Acceptance Criteria: The MS/MSD recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. The relative percent difference (RPD) between the MS and the MSD must be less than the established RPD limit, which is based on statistical analysis of historical data. MS/MSD recovery and RPD limits are updated semi-annually and stored in the LIMS.

Corrective Actions: If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.

If an MS/MSD is not possible due to limited sample, then an LCS duplicate should be analyzed. The RPD between the LCS and LCSD is compared to the established acceptance limit.

9.7 Acid Preservation or pH adjustment

The stability of 2-chloroethylvinylether, acrolein, and according to the regulations, acrylonitrile is reduced when subjected to low pH. It is therefore not recommended that these compounds be analyzed routinely from preserved VOA vials and since there is no reasonable way to achieve pH between 4 and 5, it is recommended that unpreserved vials be used for the analysis of these compounds.

Acceptance Criteria: To ensure detection of these compounds, samples must be processed correctly. Where Method 624 is being used for compliance purposes, the regulatory hold times take precedence.

Corrective Actions: If 624 data are not being generated for compliance purposes, the technical stability of the compounds may be considered. Where

method 8260 is the base method, it is allowable to qualify the results as estimated. To deviate from the regulatory hold times, the following documentation must be maintained:

- A NCM must be generated by the lab that the samples are for non-compliance.
- A NCM must be generated that results are not method compliant.

9.8 2012 MUR Required QC Elements

The May 2012 EPA Method Update Rule (MUR) to 40 CFR Part 136 for compliance testing under the Clean Water Act (CWA) requires laboratories to include 12 QC elements when performing the published or approved methods. See Work Instruction WI-DV-0060, QC Requirements for Methods Designated in 40 CFR Part 136, for list of approved test procedures performed by TestAmerica-Denver and the required QC elements in each of these methods.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preservation using EnCore™ Samplers.

10.3.1 Preservation in Methanol (Medium-Level Analysis)

10.3.1.1 Extrude the (nominal) 5 g sample from one of the Encore™ samplers into a tared 20 mL VOA vial. Obtain the weight of the soil added to the vial and record it on the label. Quickly add 5 mL of methanol and cap the vial.

10.3.1.2 If sufficient samplers are provided (or for the sample(s) designated by the client), prepare MS and MSD samples as above.

10.3.1.3 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials. Add 5 mL of methanol to the blank. For the LCS, the volume of methanol added is dependent upon the spike list. Add 4.95 mL methanol if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Cap tightly. Store with the samples.

10.3.1.4 Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.

10.3.2 Preservation in Water (Low-Level Analysis)

10.3.2.1 Extrude the (nominal) 5 g sample from one of the Encore™ samplers into a tared 20 mL VOA vial. Obtain the weight of the soil added to the vial and record it on the label. Quickly add 5 mL of water and a magnetic stirrer. Cap the vial. Repeat for the remaining aliquot.

10.3.2.2 If requested by the client, 1 g of sodium bisulfate is added to the second sample preserved with water.

10.3.2.3 If sufficient samplers are provided for a sample in the batch, or for any samples identified by the client, prepare MS and MSD samples as above.

10.3.2.4 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials. Add 5 mL of water to the blank. For the LCS, the volume of water added is dependent upon the spike list. Add 4.95 mL water if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Add a magnetic stirrer. Cap tightly. Store with the samples.

10.3.2.5 Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.

10.3.3 Screen the samples. (See Section 10.5.) If the screen indicates any samples will be analyzed as medium level, go to Section 10.5.1. If the screen indicates any samples will be analyzed as low level, go to Section 10.7.7.

10.4 Sample Storage for Field Preserved Samples

10.4.1 Obtain the weight of the soil added to each vial and record it in TALS.

10.4.1.1 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials for each analysis method (medium-level and low-level).

10.4.1.1.1 For the medium level method add 5 mL of methanol to the blank. For the LCS, the volume of methanol added is dependent upon the spike list. Add 4.95 mL methanol if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Cap tightly. Store with the samples.

10.4.1.1.2 For the low level method add water instead of methanol using the same volumes as in Section 10.4.1.1.1.

10.4.1.2 Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.

10.4.2 Screen the samples. (See Section 10.5) If the screen indicates any samples will be analyzed as medium level, go to Section 10.7.5. If the screen indicates any samples will be analyzed as low level, go to Section 10.7.7.

10.5 Sample Screening

10.5.1 Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. See SOP DV-MS-0009. Alternatively, an appropriate aliquot can be determined from sample histories.

10.6 Sample Preparation for Medium-Level Analysis – Field or Lab Preserved

10.6.1 For each of the samples that are determined to be Medium-Level samples by the screening procedure, add the correct amount of surrogate spiking mixture for a final concentration of 2 μg/mL. Example: 4 μL of 2500 μg/mL for a nominal 5 g sample or 20 μg/mL for a nominal 25 g sample. Cap the sample vial. Surrogates are added to all QC samples as well as field samples.

10.6.2 Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples for a final concentration of 2 μg/mL.

10.6.3 Add the correct amount of matrix spiking solution to the LCS sample for a final concentration of 2 μg/mL. If 25 g samples are being used, adjust the proportions for the LCS accordingly.

10.6.4 Shake the samples for two minutes to distribute the methanol throughout the soil.

10.6.5 Centrifuge the samples to clarify the extract.

10.6.6 Remove a portion of methanol and store in a clean Teflon-capped vial with no headspace refrigerated at $\leq 6\ ^{o}C$ until analysis. Duplicate aliquots of the methanol extract should be taken and stored.

10.7 Sample Analysis Procedure

10.7.1 All analysis conditions for samples must be the same as for the initial and continuing calibration standards (including purge volume, time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

10.7.2 All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a method blank, an LCS, and a MS/MSD.

10.7.2.1 If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. The

12-hour tuning requirements in Section 10.7.12.3 and 12-hour continuing calibration requirements in 10.7.14 must still be met. However, if any re-tuning or recalibration of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new QC batch must be started. For high-level soils the batch is defined at the sample preparation stage.

10.7.2.2 Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

10.7.2.3 Any reruns must be part of a valid batch. If dilutions of a sample are analyzed in the same calibration event they do not count towards the maximum batch count. (See DV-QA-003P.)

10.7.3 Water Samples

10.7.3.1 Purge-and-trap units that sample from a VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

10.7.3.2 All samples and standard solutions must be at ambient temperature before analysis.

NOTE: Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils.

10.7.3.3 To transfer a sample from its original container, fill a gas-tight syringe with the sample and adjust the sample volume based on the requested method. Place the measured sample into a clean VOA vial.

10.7.3.3.1 For Method 8260, 20 mL sample aliquots are used unless dilutions are performed. (See Section 10.7.4.) Sample aliquots are measured in 25 mL gas tight syringes. Separate syringes are used for each sample.

10.7.3.3.2 For Method 624, 5 or 20 mL sample aliquots are used unless dilutions are performed. (See Section 10.7.4.). Sample aliquots are measured in 5 or 25 mL gas-tight syringes. Separate syringes are used for each sample.

10.7.4 Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a Luerlok syringe.

10.7.4.1 For dilutions of aqueous samples which require less than 1 mL of sample the sample volume is added to 20 mL of reagent water in a VOA vial or in a gas-tight syringe.

10.7.4.2 For dilutions of aqueous samples which require more than 1 mL of sample, the volume of reagent water is adjusted so that the total volume of sample and reagent water is 20 mL. The dilution is made in the VOA vial by adding the appropriate amount of reagent water to the vial. The sample aliquot is then added to the closed vial by injecting below the surface of the water.

10.7.4.3 If the dilution required would use less than 5 µL of sample, then serial dilutions must be made in volumetric flasks.

10.7.4.4 Check and document the pH of the remaining sample. Document the pH value on the run log. If the pH is not as expected, based on the sample type and preservation, document in an NCM in the LIMS.

10.7.4.5 Sample remaining in the vial after sampling is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

10.7.4.6 For TCLP samples, use 2.0 mL of TCLP leachate and spike it with 2.5 µL of the 40 µg/mL TCLP spiking solution. Bring to a volume of 20 mL with reagent water.

10.7.4.7 Surrogates and internal standards are added to each sample at the instrument at the time of purging.

10.7.4.8 Calibration standards and spiking solutions are added to the CCVs, LCS and MS/MSD samples by the analyst prior to purging by inserting the syringe needle through the septum into the water. Surrogates and internal standards are added to these samples by the instrument.

10.7.4.9 Purge the sample for eleven minutes (the trap should be below 50 ºC).

10.7.4.10 After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 2-5 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

10.7.4.11 Desorb time, bake time, and temperature are optimized for the type of trap in use. Some programs or clients have special requirements for the desorb time. Method 624 requires a 4 minute desorb time.

NOTE: The same conditions must be used for samples and standards.

10.7.4.12 If foaming of the sample occurs, reanalyze the sample with the addition of 1 µL of an antifoaming agent such as Antifoam B (J. T. Baker). A method blank spiked with 1 µL of the Antifoam B must also be analyzed with the sample. Document in an NCM.

10.7.5 Methanol Extracts of Soils

10.7.5.1 Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic-free water as used in the calibrations (typically 5 mL).

10.7.5.2 Add no more than 25 µL of methanolic extract (from Section 10.3.1 or 10.5.1) to the syringe for each sample and QC sample. Add surrogates to each sample.

10.7.5.3 Calibration standards and spiking solutions are added to the CCVs, LCS and MS/MSD samples by the analyst prior to purging by inserting the syringe through the septum of the vial.

10.7.5.4 If less than 5 µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5 µL will be added to the water in the syringe.

10.7.5.5 Only internal standards are added at the instrument for methanol extracts.

10.7.5.6 Load the sample onto the purge and trap device and analyze as for aqueous samples. (See Section 10.7.3.)

10.7.6 Liquid Wastes that are Soluble in Methanol and Insoluble in Water

10.7.6.1 Pipette 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

10.7.6.2 Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly.

10.7.6.3 For an MS/MSD pair, add 6 mL of methanol to 2 mL of the sample in a tared vial. Add 1 mL of surrogate solution and 1 mL of matrix spike solution.

10.7.6.4 Prepare an LCS by adding 1 mL of surrogate solution and 1 mL of matrix spike solution to 8 mL of methanol.

10.7.6.5 Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic-free water as used in the calibrations.

10.7.6.6 Add no more than 25 µL of methanolic extract (Section 10.7.6.2) to the syringe. Add internal standard (if used).

10.7.6.7 Load the sample onto the purge and trap device and analyze as for aqueous samples using 5 mL reagent water.

10.7.6.8 If less than 5 µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5 µL will be added to the water in the syringe. (See Section 10.7.4.)

10.7.7 Low-Level Soil Sample Analysis following SW846 Method 5035

10.7.7.1 This technique is to be used when samples are collected utilizing SW-846 Method 5035. Pre-weighed vials are used to collect approximately a 5 gram aliquot of soil (see section 8.2).

10.7.7.2 Purge-and-trap units that sample from the VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

10.7.7.3 If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise, the internal and surrogate standards must be added to the vial.

10.7.7.4 The autosampler will heat and stir each sample as it is purged.

10.7.7.5 If any target analytes exceed the calibration range, analysis of the methanol preserved sample must be performed.

10.7.8 Low-Level Solids Analysis When Field Samples are Provided in a Jar

NOTE: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

10.7.8.1 This method is based on purging a heated sediment/soil sample mixed with water and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40 ºC.

10.7.8.2 Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

10.7.8.3 Weigh out 5 g (or other appropriate aliquot) of sample into a clean VOA vial. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium-level method described in Section 10.7.5 with preparation described in Section 10.5.1.

10.7.8.4 Rinse a 5 mL gas-tight syringe with organic-free water, and fill. Compress to 5 mL. Inject the spiked water into the VOA vial that contains the soil sample and add a stirring bar.

10.7.8.5 The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

10.7.8.6 Prepare a Method Blank and LCS using 5 g of Ottawa sand and 5 mL of water. Add a stirring bar to each. Prepare the MS/MSD (based on

the sample requested by the client. The LCS spiking solution is added via a syringe inserted through the septum of the vial to the LCS and MS/MSD samples.

10.7.8.7 Low level soil samples may be analyzed with a 1 g aliquot in place of the 5 g aliquot, mixed with water. If higher dilutions are required, the methanol extract (medium level) will be analyzed.

10.7.8.8 Surrogate and internal standards are added automatically to all samples at the instrument.

10.7.8.9 The autosampler will heat and stir each sample as it is purged.

10.7.8.10 Soil samples that have low internal standard recovery when analyzed (< 50%) should be reanalyzed once to confirm matrix effect.

10.7.9 Initial Review and Corrective Actions

10.7.9.1 If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minute from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.7.9.2 If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

Sample internal standard areas are compared to the mid-point of the supplemental initial calibration internal standard areas. Responses from 50% to 200% are acceptable. If a sample fails to meet these internal standard criteria, further investigation is necessary. If the change in sensitivity is a matrix effect confined to an individual sample, reanalysis is not necessary. If the change in sensitivity is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

10.7.9.3 The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

10.7.10 Dilutions

10.7.10.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the sample or extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.7.10.2 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment.

10.7.10.3 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.7.11 Instrument Set-up

Prior to the analysis of samples and blanks, the GC/MS system must be tuned and calibrated. Tuning is accomplished by analyzing 4-bromofluorobenzene (BFB) to establish that the GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations to determine the linearity of the response utilizing target calibration standards. The calibration must be verified each twelve-hour time period for each GC/MS system. The use of separate calibrations is required for water and low soil matrices.

10.7.12 Recommended Instrument Conditions

10.7.12.1 General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 amu
Scan Time:	to give at least 5 scans/peak, ≤ 2 second/scan
Injector Temperature:	200 – 250 ˚C
Source Temperature:	According to manufacturer's specifications
Transfer Line:	Temperature: 250 – 300 ˚C
Purge Flow:	40 mL/minute
Carrier Gas Flow:	1-15 mL/minute, dependent upon column specifications

10.7.12.2 Gas Chromatograph Suggested Temperature Program

The following temperature programs vary with the column type used.

BFB Analysis

Initial Temperature:	150 ˚C
Initial Hold Time:	0.00 minutes
1st Temperature Program:	50.00 ˚C/minute
Final Temperature:	220 ˚C
Final Time:	4.00 minutes
2nd Temperature Program:	OFF
Post Temperature:	0 ˚C
Post Time:	0.00 minutes
Run Time:	5.40 minutes

Sample Analysis

Initial Temperature:	40 ˚C
Initial Hold Time:	4 minutes
1st Temperature Program:	8 ˚C/minute
Final Temperature:	184 ˚C
2nd Temperature Program:	40 ˚C/minute
Final Temperature:	240 ˚C
Final Hold Time:	2.6 minutes

10.7.12.3 Instrument Tuning

Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 11 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.7.13 Initial Calibration

10.7.13.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and in the public folder *Arizona Calibration Training.*

10.7.13.2 A series of five or more initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Certain analytes are prepared at higher concentrations due to poor purge performance. The following calibration curves are maintained. Calibration levels for each analyte are given in the stated tables. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument or program requirements.

Initial Calibration by Matrix and Method

Method	Matrix	Purge Volume	Calibration Levels
624	Water	5 mL	Table A-3
8260	Water	20 mL	Tables 5 and 5A

Method	Matrix	Purge Volume	Calibration Levels
8260	Soil (low level)	5 mL	Tables 4 and 4A
8260	Soil (Methanol Extract)	5 mL reagent water + 25 µL Methanol	Tables 6 and 6A
Alaska	Soil	See Appendix B	See Appendix B

10.7.13.3 Calibration levels below the reporting limit may be removed provided that there is a minimum of five calibration points for linear regression and six calibration points for second order calibration. The lowest standard used in the calibration must be at or below the TestAmerica reporting limit.

10.7.13.4 The same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.7.13.5 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.

10.7.13.6 Internal standard calibration is used. The internal standards are listed in Tables 7 and 7A. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See Equation 1, Section 11.4.1, for calculation of response factor.

10.7.13.7 Evaluation of retention times

The relative retention time of each target analyte in each calibration standard should agree within 0.5 min.

10.7.13.8 The % RSD of each of the calibration check compounds (CCC) must be less than or equal to 30%. Refer to Table 13. See Table A-2 for Method 624 criteria.

10.7.13.9 The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 12 for the SPCC compounds and required minimum response factors.

10.7.13.10 If the software in use is capable of routinely reporting curve

coefficients for data validation purposes and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation. The correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990.

Note: Additional criteria are stated in the North Carolina QAS.

10.7.13.11 If the software in use is capable of routinely reporting curve coefficients for data, and if the average of all the %RSDs in the calibration is > 15%, then calibration on a curve must be used for all analytes with %RSD > 15%. The analyst should consider instrument maintenance to improve the linearity of response. Otherwise, the correlation coefficient, r (coefficient of determination, r^2 for non-linear curves) must be ≥ 0.990.

Note: Some states (like Arizona) and federal programs do not allow the use of grand mean. Refer to the Arizona QAS and SOP DV-QA-024P.

10.7.13.12 Once the initial calibration has been evaluated and determined to be valid, the calibration must be verified with an Initial Calibration Verification (ICV) using a standard prepared from an alternate source. All compounds in the ICV must be <35 % drift when compared to the initial calibration, except poor performers (see Table 16) which must be <55% drift. The ICV is generally run at the same concentration as the level 5 standard. See Table A-2 for method 624 criteria.

10.7.13.13 If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration, Section 10.7.14.

10.7.13.14 A separate five point calibration must be prepared for analysis of low-level soils. Low-level soils analysis requires the use of a closed vial autosampler. Each standard is prepared by spiking the methanol standard solution through the septum of a VOA vial containing 5 mL of water. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Methanol soil extracts should be analyzed using the methanol calibration curve.

10.7.13.15 Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. The primary ion for the single standard must generate a peak clearly visible over background noise (greater than three standard deviations at a minimum) and be free of spectral interferences. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s)

reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary. A footnote or narrative comment should describe the basis of the reported result.

10.7.14 Continuing Calibration

10.7.14.1 The initial calibration must be verified every twelve hours.

10.7.14.2 Continuing calibration begins with analysis of BFB as described in Section 10.7.12.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 4 calibration standard is used as the continuing calibration standard. See Table A-2 for method 624 criteria.

10.7.14.3 The RF data from the standards are compared with the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in equation 4, Section 11.4.4.

10.7.14.4 The % drift of the CCCs must be ≤ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 12. In addition, the % drift for most non-CCC analytes must be ≤ 35 %, and for poor performers ≤ 50 % (See Table 16), with allowance for up to six target analytes to have a % drift greater than the applicable limit. For agencies that require specific control limits for non-CCC compounds (i.e., State of Arizona) see Table 15. See Table A-2 for method 624 criteria.

Note: Additional criteria are stated in the North Carolina QAS.

10.7.14.4.1 If none of the CCCs are required analytes, project specific calibration specifications (which may include the use of the CCCs listed in Table 13) must be agreed to with the client.

10.7.14.4.2 Cyclohexanone is unstable in the calibration solution forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists.

10.7.14.5 The retention time of the internal standards in the continuing calibration standard cannot change by more than 30 seconds when compared to the most recent five-point calibration. The internal standard areas must not change by more than a factor of 2 (50 - 200 %) from the mid point standard of the most recent five-point calibration.

10.7.14.6 If the CCCs and/or the SPCCs do not meet the criteria in Sections 10.7.14.3 and 10.7.14.4, the system must be evaluated and corrective action must be taken. The BFB tune and continuing

calibration must be acceptable before analysis begins. Extensive corrective action, such as a different type of column, will require a new initial calibration.

10.7.14.7 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample desorbed less than or equal to 12 hours after the BFB is acceptable.)

10.7.14.8 Sodium Bisulfate must be added to the CCV when analyzing samples preserved with it.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate SOP CA-Q-S-005 "Calibration Curves" and in the public folder, *Arizona Calibration Training*.

11.2 Qualitative Identification

11.2.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library (same library as used for routine sample analysis). Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

11.2.1.1 The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

11.2.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

11.2.1.3 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%.)

11.2.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

11.2.3 All data are subject to two levels of technical review, as described in SOP DV-QA-0020.

11.3 Tentatively Identified Compounds (TICs)

11.3.1 If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. The following guidelines apply:

11.3.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

11.3.1.2 The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

11.3.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.3.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.3.1.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or co-eluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

11.3.1.6 Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst *assign a tentative identification.*

11.4 Calculations.

11.4.1 Response factor (RF):

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$ *Equation 1*

Where:

A_x = Area of the characteristic ion for the compound to be measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard, ng
C_x = Concentration of the compound being measured, ng.

11.4.2 Standard deviation (SD):

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(X_i - \overline{X}\right)^2}{n-1}} \qquad \textit{Equation 2}$$

Where:

X_i = Value of X at i through n.
n = Number of points.
$\overline{X}$ = Average value of X_i.

11.4.3 Percent relative standard deviation (%RSD):

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\% \qquad \textit{Equation 3}$$

Where $\overline{RF}$ is the mean of RF values for the calibration.

11.4.4 Percent drift between the initial calibration and the continuing calibration:

$$\%Drift = \frac{C_{\text{exp}ected} - C_{found}}{C_{\text{exp}ected}} \times 100\% \qquad \textit{Equation 4}$$

Where:

$C_{expected}$ = *Known concentration in standard.*
C_{found} = *Measured concentration using selected quantitation method.*

11.4.5 See SOP CA-Q-S-005 for more detailed calibration equations.

11.4.6 Target compound and surrogate concentrations:

Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is ≤ 15%.

11.4.6.1 Calculation of concentration using Average Response Factors:

$$\text{Concentration } (\mu\text{g/L}) = \frac{x}{\overline{RF}} \qquad \textit{Equation 5}$$

11.4.6.2 Calculation of concentration using Linear fit:

$$\text{Concentration } (\mu\text{g/L}) = A + Bx \qquad \textit{Equation 6}$$

11.4.6.3 Calculation of concentration using Quadratic fit:

$$Concentration(\mu g / L) = A + Bx + Cx^2$$ *Equation 7*

Where:

x = see equations 8, 9, and 10.
A = intercept of the calibration function.
B = slope of calibration function.
C = curvature of a second-order calibration function.

11.4.6.4 Calculation of x for Water and water-miscible waste:

$$x = \frac{A_x I_s D_f}{A_{is} V_0}$$ *Equation 8*

Where:

A_x = *Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion).*

A_{is} = *Area of the characteristic ion for the internal standard.*

I_s = *Amount of internal standard added in ng.*

$$D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = *Volume of water purged, mL.*

11.4.6.5 Calculation of x for High-level soils:

$$x = \frac{(A_x)(I_s)(V_t)(1000)D_f}{(A_{is})(V_a)(W_s)(D)}$$ *Equation 9*

Where:

A_x, I_s, D_f, A_{is}, = *same as used in equation 8 above.*

V_t = *Volume of total extract, mL (typically 25 mL).*

V_a = *Volume of extract added for purging, µL.*

W_s = *Weight of sample extracted, g.*

D = $\frac{100 - \%moisture}{100}$

11.4.6.6 Calculation of x for Low level soils:

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$ *Equation 10*

Where:

A_x, I_s, D_f, A_{is},	=	*same as used in equation 8 above.*
D	=	*same as in equation 9 above.*
W_s	=	*Weight of sample added to the purge vessel, g.*

11.4.6.7 Calculation of TICs

The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x	=	*Area in the total ion chromatogram for the compound being measured.*
A_{is}	=	*Area of the total ion chromatogram for the nearest internal standard without interference.*
RF	=	*1*

In other words, the concentration is equal to x as defined in equations 8, 9, and 10.

11.4.7 MS/MSD Recovery

$$\%\text{Recovery} = \frac{SSR - SR}{SA} \times 100\% \qquad \textit{Equation 11}$$

Where:

SSR	=	*Spike sample result.*
SR	=	*Sample result.*
SA	=	*Spike added.*

11.4.8 Relative % Difference calculation for the MS/MSD:

$$RPD = \frac{\left|MSR - MSDR\right|}{\frac{1}{2}\left(MSR + MSDR\right)} \times 100\% \qquad \textit{Equation 12}$$

Where:

RPD	=	*Relative percent difference.*
MSR	=	*Matrix spike result.*
MSDR	=	*Matrix spike duplicate result.*

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in TestAmerica Denver's Policy No. DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements (e.g., DoD) indicate a greater frequency.

12.2 Demonstration of Capabilities

12.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.2.1.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.1.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.1.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the

policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methanol Waste - Vial Waste and Flammable – Waste Streams A and C

14.2.2 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.3 Acidified Water – Waste Stream W

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 8260B, Volatile Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 2, December, 1996.

15.1.2 Method 5030B, Purge-and-Trap for Aqueous Samples, Revision 2, December, 1996.

15.1.3 Method 5035, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 0, December, 1996.

15.1.4 Method 5035A (R1-MIR), Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Draft Revision 1, July 2002.

15.1.5 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

15.1.6 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003..

15.2 40 CFR Part 136, Appendix A (Method 624, Method 603).

15.3 Method AK101 For the Determination of Gasoline Range Organics, Alaska DEC, Version 04/08/02.

16.0 Method Modifications:

Item	Method	Modification
1	SW-846 8260B	Ion 119 is used as the quantitation ion for chlorobenzene-d_5.
2	SW-846 8260B	The quantitation and qualifier ions for some compounds have been

Item	Method	Modification
		changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.
3	SW-846 8260B	This SOP has been written to allow for a 20 mL purge volume for waters. An additional 5 mL of DI water is added to all samples, QC and calibration standards. The final purge volume is 25 mL.
4	SW-846 8260B	Method 8260B recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.
5	SW-846 8260B	SW-846 recommends that a curve be used for any analytes with %RSD of the response factors > 15%. However, some industry standard data systems and forms generation software cannot report this data with the necessary information for data validation. In addition, most software available does not allow weighting of the curve. Unweighted curves may exhibit serious errors in quantitation at the low end, resulting in possible false positives or false negatives. Therefore, if the overall average is $\leq$ 15% then the ICAL is considered acceptable and any compounds that are not $\leq$15% will use linear regression.
6	EPA 624	Method 624 is required for demonstration of compliance with CWA permits, e.g., NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table A-1. If compounds are added to the analysis, all of the method criteria must be satisfied for the additional compounds.
7	EPA 624	The tune period for this method is defined as 24 hours, which is the maximum elapsed time before the tune check is performed. Calibration verifications are done at the same 24 hour frequency.
8	EPA 624	The initial calibration curve for this method requires at least three points, as shown in Table A-3.
9	EPA 624	Sample concentrations are calculated using the average RRF from the initial calibration curve.
10	EPA 624	Each target analyte is assigned to the closest eluting internal standard.
11	EPA 624	Initial demonstration of Proficiency • The spiking level for the four replicate initial demonstration of proficiency is 20 μg/L. The acceptance criteria are listed in Table A-2
12	EPA 624	Initial calibration curve requirements: • Target compounds must have RSD $\leq$ 35%.

Item	Method	Modification
		• If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.
13	EPA 624	Continuing calibration verification requirements: • The continuing calibration standard is from a different source than the initial calibration standard. The daily CCAL concentration is 20 ug/L. The acceptance criteria are listed in Table A-2. Matrix Spike and LCS Requirements • The matrix spike and LCS are spiked at 20 μg/L, prepared from the same source containing all analytes of interest. A matrix spike duplicate is not necessary for this method. The recovery limits for matrix spike and LCS recovery are listed in Table A-2.
14	EPA 624	Consistent with the other volatile methods, corrections for recovery are not allowed.
15	EPA 624	Qualitative Identification – The source method states that the relative intensities of ions should agree to within ±20% between the standard and sample spectra. This SOP uses ±30%. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)
16	EPA 624	Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely employs the OI #10 trap which consists of Tenax/Silica Gel/ Carbon Molecular Sieve or the Supelco Vocarb 3000 which consists of Carbopack B, Carbonxen1000 and 1001.
17	EPA 624	Section 5.3.2 of the source method describes a packed analytical column. TestAmerica routinely employs capillary columns when performing this method.
18	EPA 624	The source method provides a suggested list of compounds for internal and surrogate standards. Others are permitted by the method. TestAmerica uses three internal standards, including chlorobenzene-d_5 and 1,4-dichlorobenzene-d_4, which are not listed in Table 3 of the source method. Toluene-d_8 is used as a surrogate compound, which is also not listed in the source method.
19	EPA 624	The lab is preparing internal standards at 10 ug/L and applying the same criteria designed for 30 ug/L in the Method. The lower concentration is consistent with the greater sensitivity provided by capillary columns as compared to the older packed columns described in the method. It could only be more challenging for the lab to meet the acceptance criteria at 10 ug/L; it provides a higher

Item	Method	Modification
		level of data quality.
20	EPA 624	Method 624 describes a mass scan range of 25 to 260 amu. Table 13 lists all of the ions used for analysis. None of the ions are below 35 amu. Therefore, we scan from 35 to 300 and include all ions needed for analysis.
21	EPA 624	Method 624 describes dilutions "if response of any m/z" exceeds the response for the highest m/z in the ICAL. As the m/z ratio is always directly proportional to the concentration, evaluation based on dilution (per 11.10) is equivalent.
22	EPA 624	Method 624 has criteria for unresolved isomers. The problems of isomeric resolution for the routine analytes listed in this SOP were worked through when the laboratory developed its implementation of the method. For example, we know through experience that meta- and para-xylenes will not be resolved and it was not necessary to include an evaluation for the xylenes in each analysis. meta- and para-xylenes are reported as an isomeric pair. Any development work to add compounds would take this into account.
23	624	The source method recommends Method 603 as the preferred method for Acrolein and Acrylonitrile. Method 624 is recommended as a screening method (see section 1.2 of Methods 603 and 624). Calibration and quality control samples indicate that the conditions described in this SOP are suitable for the analysis of Acrolein and Acrylonitrile. EPA's Method Update Rule (MUR), May 18, 2012, allows the addition of acrolein and acrylonitrile to Method 624, using the preservation, holding time and QC acceptance criteria from Method 603. As states implement the MUR Method 624 becomes a determinative method for these two analytes. Until such time, Method 624 remains a screening method for regulatory compliance.
24	SW846 5035	The source method recommends adding approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~ 1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. TestAmerica Denver does not recommend the use of sodium bisulfate to preserve soil samples, but encourages clients to collect samples using other available methods. The use of this preservative has been shown to cause difficulties recovering more reactive analytes on the purge and trap system (e.g. 2-Chloroethyl vinyl ether, acrylamide).

17.0 Attachments

Table 1. TestAmerica Primary List Reporting Limits for 8260B
Table 2. TestAmerica 8260 Secondary List Reporting Limits
Table 3. TestAmerica Appendix IX List Reporting Limits
Table 4. TestAmerica Non-Standard Compound List Reporting Limits
Table 5. Soil Calibration Levels, 5-gram Purge (μg/Kg)

	(Standard Mixes: MV-Main & MV-Main GasKe)
Table 5A.	Soil Calibration Levels, 5 gram Purge (µg/Kg) (Standards: MV-Supp Std and MV-2 Cleve)
Table 6.	Water 8260 List Calibration Levels (µg/L) (Standards: MV-Main and MV-Main GasKe)
Table 6A	Water 8260 List Calibration Levels (µg/L) (Standards: MV-Supp Std and MV- 2 Cleve)
Table 7.	Medium Level Soil 8260 List Calibration Levels (µg/Kg) (Standards: MV-Main and MV-Main GasKe)
Table 7A.	Medium Level Soil 8260 List Calibration Levels (µg/Kg) (Standards: MV-Supp Std and MV- 2 Cleve)
Table8.	Manually added Internal Standards
Table 8A.	Automatically Added Internal Standards
Table 9.	Manually Added Surrogate Standards
Table 9A.	Automatically Added Surrogate Standards
Table 10.	Matrix Spike and LCS Standard
Table 11.	BFB Key Ion Abundance Criteria
Table 12.	SPCC Compounds and Minimum Response Factors
Table 13.	CCC Compounds
Table 14.	Characteristic Ions
Table 15.	State of Arizona ICV/CCV Quality Control Limits
Table 16.	List 1 Poorly performing Compounds
Table A-1.	Method 624 Analytes and Reporting Limits, 5-mL Purge
Table A-2.	Method 624 QC Acceptance Criteria
Table A-3.	Calibration Levels for 624, 5 mL Purge
Appendix A.	Modifications for Analysis of 1,4-Dioxane, 1,2,3-Ttrichloropropane, 1,2-Dibromo-3-chloropropane, and 1,2-Dibromoethane by Selected Ion Monitoring
Table Ap-1.	TAL Method 8260SIM Standard Reporting Limits
Table Ap-2.	Method 8260SIM Calibration Levels
Table Ap-3.	Method 8260SIM LCS Spike Concentrations
Table Ap-4.	8260SIM Surrogate Compounds
Table Ap-5.	8260SIM Internal Standard Compounds
Table Ap-6.	8260 Selected Masses
Table Ap-7.	Suggested Instrument Conditions for 8260SIM
Appendix B	Modifications for Analysis of Soils Collected for the State of Alaska
Table Bp-1:	TestAmerica 8260 Reporting Limits – AK Soils
Table Bp-2:	Calibration Levels for 8260, 5035FM_AK
Table Bp-3:	5035FM_AK Calibration Levels (µg/Kg) (Standards: MV-Supp Std and MV-2 Cleve)
Attachment 1.	Gas Standards Tracking Log

18.0 Changes from Previous Revision

- Revision 9, dated 04 January 2013
  - Added section 9.8 to address the 2012 MUR QC requirements
- Revision 8, dated 28 September 2012
  - Added to compounds to the reporting limit, characteristic ion and calibration tables

to match TALS.

- Revision 7, dated 27 July 2012
  - Added sodium bisulfate to Section 7.
  - Revised Section 8 to include Terra Core samplers and moved instructions on sample preparation and handling in the lab to Section 10. Reorganized sampling and preservation information into tables. Updated information including footnote on Holding Time and preservation table for water regarding Method Update Rule that approves use of Method 624 for analysis of acrolein and acrylonitrile.
  - Removed flowcharts from Section 8.
  - Revised Section 9.1
  - Revised Section 10.
  - Updated reference section to include Method 603, Method 5035A, and Method 8000B and 8000C.
  - Revised Method Modifications #23
  - Updated tables to reflect current practice.
  - Added Appendix B for the analysis of soils using the AK methanol extraction procedure.
  - Formatting and editorial changes throughout

- Revision 6.4, dated 28 December 2011
  - Changed the column ID and film thickness in section 6.1.8.1
  - Updated the calibration levels in Table AP-2

- Revision 6.3, dated 26 October 2011
  - Added Section 4.6 regarding interferences with toluene-d_8 surrogate when potassium permanganate may have been added to sample
  - Updated path to QAS folders in the public folders, section 9.7
  - Added J. T. Baker Antifoam B and reagent sand, sections 7.3, 7.4
  - Added description of procedure for use of antifoaming agent B, section 10.1.3.8
  - Formatting

- Revision 6.2, dated 25 August, 2011
  - Added requirements to section 9.4 for the use of Ottawa sand in soil LCS's.

- Revision 6.1, dated 31 January, 2011
  - Added details to Appendix A for the analysis of soils by SIM
  - Added Tables AP-1 through Ap-7
  - Added Attachment 1, Gas Standards Tracking Log
  - Added section 11.1 referencing corporate SOP CA-Q-S-005 "Calibration Curves"

- Revision 6, dated 02 November, 2010
  - Added analysis information concerning BFB

- Revision 4, dated May 5, 2010
  - Updated Tables to reflect current report limits.
  - Updated low level procedure to include water option for preservation.
  - Updated surrogate and spike amounts.

- Revision 3.1, dated 11 December 2009
  - Added Trichloroethene to Table 12.
  - Updated section 16 to describe the process of adding and additional 5 mL of DI

water to all samples and QC.
  - Added a note to section 9.4 that marginal exceedances are not allowed for some programs.
  - Updated the language in section 16 item 5 to describe the current practice.

- Revision 3.0, dated 21 January 2009
  - Added clarification of sample preservation requirements to section 8.
  - Adjusted Table 16 for South Carolina requirements to utilize default limits.
  - Added Table 8A for AFCEE water calibration levels.

- Revision 2.1, dated 16 July 2007
  - Add reference to North Carolina QAS for additional requirements to sections 9.6, 10.4.8, and 10.5.4.
  - Remove Nitrogen as an allowable substitution for Helium in section 6.8.
  - Added the current list of spike compounds to Table 12.
  - Updated references to include 5030B and 5035.
  - Removed EPA 524.2 references.

- Revision 2.0
  - The method blank acceptance criteria and corrective actions were updated in Section 9.4.

Table 1. TestAmerica Primary List Reporting Limits for 8260B

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
Dichlorodifluoromethane	75-71-8	2	10	500
Chloromethane	74-87-3	2	10	500
Bromomethane	74-83-9	2	10	500
Vinyl chloride	75-01-4	1	5	500
Chloroethane	75-00-3	2	10	500
Trichlorofluoromethane	75-69-4	2	10	500
Acrolein	107-02-8	20	50	5,000
Acetone	67-64-1	10	20	1,000
Trichlorotrifluoroethane	76-13-1	3	20	1,000
Ethanol	64-17-5	300	600	10,000
Iodomethane	74-88-4	1	5	250
Carbon disulfide	75-15-0	2	5	250
Methylene chloride	75-09-2	2	5	250
tert-Butyl alcohol	75-65-0	50	200	10,000
1,1-Dichloroethene	75-35-4	1	5	250
1,1-Dichloroethane	75-34-3	1	5	250
trans-1,2-Dichloroethene	156-60-5	1	2.5	125
Acrylonitrile	107-13-1	20	50	5,000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	5	20	250
Hexane	110-54-3	2	5	250
cis-1,2-Dichloroethene	156-59-2	1	2.5	125
1,2-Dichloroethene (Total)	540-59-0	1	5	250
Tetrahydrofuran	109-99-9	7	20	1,000
Chloroform	67-66-3	1	10	250
1,2-Dichloroethane	107-06-2	1	5	250
Dibromomethane	74-95-3	1	5	250
2-Butanone	78-93-3	6	20	1,000
1,4-Dioxane	123-91-1	200	500	25,000
1,1,1-Trichloroethane	71-55-6	1	5	250
Carbon tetrachloride	56-23-5	1	5	250
Bromodichloromethane	75-27-4	1	5	250
1,2-Dichloropropane	78-87-5	1	5	250

Table 1. TestAmerica Primary List Reporting Limits for 8260B

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
cis-1,3-Dichloropropene	10061-01-5	1	5	250
Trichloroethene	79-01-6	1	5	250
Dibromochloromethane	124-48-1	1	5	250
1,2-Dibromoethane	106-93-4	1	5	250
1,2,3-Trichloropropane	96-18-4	2.5	5	250
1,1,2-Trichloroethane	79-00-5	1	5	250
Benzene	71-43-2	1	5	250
Ethylmethacrylate	97-63-2	3	5	250
trans-1,3-Dichloropropene	10061-02-6	3	5	250
Bromoform	75-25-2	1	5	250
4-Methyl-2-pentanone	108-10-1	5	20	1,000
2-Hexanone	591-78-6	5	20	1,000
Tetrachloroethene	127-18-4	1	5	250
Toluene	108-88-3	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	1	5	250
2-Chloroethyl vinyl ether[2]	110-75-8	N/A[2]	50	2,500
Vinyl acetate	108-05-4	3	10	500
Chlorobenzene	108-90-7	1	5	250
Ethylbenzene	100-41-4	1	5	250
Styrene	100-42-5	1	5	250
trans-1,4-Dichloro-2-butene	110-57-6	3	5	250
m- and p-Xylenes	179601-23-1	2	3.5	250
o-xylene	95-47-6	1	2.5	125
Total xylenes	1330-20-7	2	10	250
1,3-Dichlorobenzene	541-73-1	1	5	250
1,4-Dichlorobenzene	106-46-7	1	5	250
1,2-Dichlorobenzene	95-50-1	1	5	250

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2. TestAmerica 8260 Secondary List Reporting Limits

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water µg/L	Low Soil µg/kg	Medium Soil µg/kg
2,2-Dichloropropane	590-20-7	1	5	250
Bromochloromethane	74-97-5	1	5	250
1,1-Dichloropropene	563-58-6	1	5	250
1,3-Dichloropropane	142-28-9	1	5	250
1-Chlorohexane	544-10-5	1	5	500
1,1,1,2-Tetrachloroethane	630-20-6	1	5	250
Isopropylbenzene	98-82-8	1	5	250
Bromobenzene	108-86-1	1	5	250
n-Propylbenzene	103-65-1	1	5	250
2-Chlorotoluene	95-49-8	1	5	250
4-Chlorotoluene	106-43-4	1	5	250
1,3,5-Trimethylbenzene	108-67-8	1	5	250
tert-Butylbenzene	98-06-6	1	5	250
1,2,4-Trimethylbenzene	95-63-6	1	5	250
sec-Butylbenzene	135-98-8	1	5	250
4-Isopropyltoluene	99-87-6	1	5	250
n-Butylbenzene	104-51-8	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	5	250
1,2,4-Trichlorobenzene	120-82-1	1	5	250
Naphthalene	91-20-3	1	5	500
Hexachlorobutadiene	87-68-3	1	5	250
1,2,3-Trichlorobenzene	87-61-6	1	5	250
2-Pentanone	107-87-9	5	10	500
cis-1,4-Dichloro-2-butene	1476-11-5	3	5	250
Ethylene oxide	75-21-8	600	3,000	150,000

Table 3. TestAmerica Appendix IX List Reporting Limits

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water µg/L	Low Soil µg/kg	Medium Soil µg/kg
Allyl Chloride	107-05-1	2	10	500
Acetonitrile	75-05-8	30	100	5,000
Dichlorofluoromethane	75-43-4	2	10	25,000
Isopropyl ether	108-20-3	10	50	2,500
Chloroprene	126-99-8	1	5	500
n-Butanol	71-36-3	60	200	10,000
Propionitrile	107-12-0	20	50	1,000
Methacrylonitrile	126-98-7	10	50	2,500
Isobutanol	78-83-1	110	200	10,000
Methyl methacrylate	80-62-6	4	5	250
1,1,1,2-Tetrachloroethane	630-20-6	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	10	500
Ethyl ether	60-29-7	2	10	500
Ethyl Acetate	141-78-6	5	10	500
2-Nitropropane	79-46-9	5	10	500
Cyclohexanone[2]	108-94-1	N/A[2]	N/A[2]	N/A[2]
Isopropylbenzene	98-82-8	1	5	250

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

Table 4. TestAmerica Non-Standard List Reporting Limits for 8260B

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
1,1,1-Trifluoro-2,2-Dichloroethane	306.83-2	2.0	5.0	1000
1,2,3-Trimethylbenzene	526-73-8	2.0	5.0	250
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	76-14-2	2.0	5.0	250
1,2-Dichloro-1,1,2-Trifluoroethane	354-23-4	2.0	5.0	250
1,3,5-Trichlorobenzene	108-70-3	5.00	***	***
2,2,3-Trimethylbutane	464-06-2	5.00	***	***
2,2-Dimethylpentane	590-35-2	5.00	***	***
2,3-Dimethylpentane	565-59-3	5.00	***	***
2,4-Dimethylpentane	108-08-7	5.00	***	***
2-Chloro-1,1,1-Trifluoroetane	75-88-7	5.00	5.0	250
2-Methylhexane	591-76-4	5.00	***	***
3,3-Dimethylpentane	562-49-2	5.00	***	***
3-Ethylpentane	617-78-7	5.00	***	***
3-Methylhexane	589-34-4	5.00	***	***
Chlorotrifluoroethene	79-38-9	5.00	5.0	250
Cyclohexane	110-82-7	2.0	5.0	250
Dimethyl Disulfide	624-92-0	5.00	***	***
Isopropyl Alcohol	67-63-0	40	200	10,000
Methyl Acetate	79-20-9	5.0	10	1000
Methylcyclohexane	108-87-2	1.0	5.0	250
n-Heptane	142-82-5	5.00	***	***
n-Nonyl Aldehyde	124-19-6	10.00	***	***
Propene Oxide	75-56-9	50	3000	250
Sec-Butyl Alcohol	78-92-2	***	200	***
Tert-amyl methyl ether	994-05-8	5	5.0	1000
Tert-butyl ethyl ether	637-92-3	5	5.0	1000
Tetrahydrothiophene	110-01-0	2.0	5.0	***

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table 5. Soil Calibration Levels, 5-gram Purge[1] (Standard Mixes: MV-Main & MV-Main GasKe)								
Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1,1,1,2-Tetrachloroethane	1	2	5	10	20	50	100	200
1,1,1-Trichloroethane	1	2	5	10	20	50	100	200
1,1,2,2-Tetrachloroethane	1	2	5	10	20	50	100	200
1,1,2-Trichloroethane	1	2	5	10	20	50	100	200
1,1-Dichloroethane	1	2	5	10	20	50	100	200
1,1-Dichloroethene	1	2	5	10	20	50	100	200
1,1-Dichloropropene	1	2	5	10	20	50	100	200
1,2,3-Trichlorobenzene	1	2	5	10	20	50	100	200
1,2,3-Trichloropropane	1	2	5	10	20	50	100	200
1,2,4-Trichlorobenzene	1	2	5	10	20	50	100	200
1,2,4-Trimethylbenzene	1	2	5	10	20	50	100	200
1,2-Dibromo-3-chloropropane	1	2	5	10	20	50	100	200
1,2-Dichlorobenzene	1	2	5	10	20	50	100	200
1,2-Dichloroethane	1	2	5	10	20	50	100	200
1,2-Dichloropropane	1	2	5	10	20	50	100	200
1,3,5-Trimethylbenzene	1	2	5	10	20	50	100	200
1,3-Dichlorobenzene	1	2	5	10	20	50	100	200
1,3-Dichloropropane	1	2	5	10	20	50	100	200
1,4-Dichlorobenzene	1	2	5	10	20	50	100	200
1,4-Dioxane	50	100	250	500	1,000	2,500	5,000	10,000
1-Chlorohexane	1	2	5	10	20	50	100	200
2,2-Dichloropropane	1	2	5	10	20	50	100	200
2-Butanone	4	8	20	40	80	200	400	800
2-Chloro-1,3-butadiene	1	2	5	10	20	50	100	200
2-Chlorotoluene	1	2	5	10	20	50	100	200
2-Hexanone	4	8	20	40	80	200	400	800
4-Chlorotoluene	1	2	5	10	20	50	100	200
4-Isopropyltoluene	1	2	5	10	20	50	100	200
4-Methyl-2-pentanone	4	8	20	40	80	200	400	800
Acetone	4	8	20	40	80	200	400	800
Acetonitrile	4	8	20	40	80	200	400	800
Acrolein	4	8	20	40	80	200	400	800
Acrylonitrile	4	2	5	10	20	50	100	200

Table 5. Soil Calibration Levels, 5-gram Purge[1]
(Standard Mixes: MV-Main & MV-Main GasKe)

Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Benzene	1	2	5	10	20	50	100	200
Bromobenzene	1	2	5	10	20	50	100	200
Bromoform	1	2	5	10	20	50	100	200
Bromomethane	1	2	5	10	20	50	100	200
Carbon tetrachloride	1	2	5	10	20	50	100	200
Chlorobenzene	1	2	5	10	20	50	100	200
Chlorobromomethane	1	2	5	10	20	50	100	200
Chloroethane	1	2	5	10	20	50	100	200
Chloroform	1	2	5	10	20	50	100	200
Chloromethane	1	2	5	10	20	50	100	200
cis-1,2-Dichloroethene	1	2	5	10	20	50	100	200
cis-1,3-Dichloropropene	1	2	5	10	20	50	100	200
Cyclohexanone	40	80	200	400	800	2,000	4,000	8,000
Chlorodibromomethane	1	2	5	10	20	50	100	200
Dibromomethane	1	2	5	10	20	50	100	200
Dichlorobromomethane	1	2	5	10	20	50	100	200
Dichlorodifluoromethane	1	2	5	10	20	50	100	200
Ethanol	50	100	250	500	1,000	2,500	5,000	10,000
Ethylbenzene	1	2	5	10	20	50	100	200
Ethylene dibromide	1	2	5	10	20	50	100	200
Hexachlorobutadiene	1	2	5	10	20	50	100	200
Iodomethane	1	2	5	10	20	50	100	200
Isobutyl alcohol	20	40	100	200	400	1,000	2,000	4,000
Isopropyl ether	5	10	25	50	100	250	500	1,000
Isopropylbenzene	1	2	5	10	20	50	100	200
m- and p-Xylenes	2	4	10	20	40	100	200	400
Methacrylonitrile	10	20	50	100	200	500	1,000	2,000
Methylene chloride	1	2	5	10	20	50	100	200
Naphthalene	1	2	5	10	20	50	100	200
n-Butanol	30	60	150	300	600	1,500	3,000	--
n-Butylbenzene	1	2	5	10	20	50	100	200
n-Propylbenzene	1	2	5	10	20	50	100	200
o-Xylene	1	2	5	10	20	50	100	200
Propionitrile	10	20	50	100	200	500	1,000	2,000

Table 5. Soil Calibration Levels, 5-gram Purge[1] (Standard Mixes: MV-Main & MV-Main GasKe)								
Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
sec-Butylbenzene	1	2	5	10	20	50	100	200
Styrene	1	2	5	10	20	50	100	200
2-Methyl-2-propanol (tert-Butyl alcohol)	20	40	100	200	400	1,000	2,000	4,000
tert-Butylbenzene	1	2	5	10	20	50	100	200
Tetrachloroethene	1	2	5	10	20	50	100	200
Toluene	1	2	5	10	20	50	100	200
trans-1,2-Dichloroethene	1	2	5	10	20	50	100	200
trans-1,3-Dichloropropene	1	2	5	10	20	50	100	200
Trichloroethene	1	2	5	10	20	50	100	200
Trichlorofluoroethane	1	2	5	10	20	50	100	200
Vinyl chloride	1	2	5	10	20	50	100	200

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 5A: Soil Calibration Levels, 5-gram Purge, µg/Kg[1] (Standards: MV-Supp Std and MV-2 Cleve)							
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1-Trifluoro-2,2-dichloro-ethane	2	5	10	20	50	100	200
1,1,2-Trichloro-1,2,2-trifluoroethane	2	5	10	20	50	100	200
1,2,3-Trimethylbenzene	2	5	10	20	50	100	200
1,2-Dichloro-1,1,2,2-tetrafluroethane	2	5	10	20	50	100	200
1,2-Dichloro-1,1,2-trifluoroethane	2	5	10	20	50	100	200
2-Chloroethyl vinyl ether	2	5	10	20	50	100	200
2-Nitropropane	2	5	10	20	50	100	200
2-Pentanone	8	20	40	80	200	400	800
3-Chloro-1-propene (Allyl Chloride)	2	5	10	20	50	100	200
Carbon disulfide	2	5	10	20	50	100	200
cis-1,4-Dichloro-2-butene	2	5	10	20	50	100	200
Cyclohexane	2	5	10	20	50	100	200

Table 5A: Soil Calibration Levels, 5-gram Purge, µg/Kg[1] (Standards: MV-Supp Std and MV-2 Cleve)							
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**
Dichlorofluoromethane	2	5	10	20	50	100	200
Ethyl Acetate	4	10	20	40	100	200	400
Ethyl ether	2	5	10	20	50	100	200
Ethyl methacrylate	4	10	20	40	100	200	400
Ethylene Oxide	250	625	1,250	2,500	6,250	12,500	25,000
Hexane	2	5	10	20	50	100	200
Isopropyl alcohol	40	100	200	400	1000	2000	4,000
Methyl acetate	10	25	50	100	250	500	1,000
Methyl methacrylate	4	10	20	40	100	200	400
Methyl *tert*-butyl ether (MTBE)	2	5	10	20	50	100	200
Methylcyclohexane	2	5	10	20	50	100	200
sec-Butyl alcohol	60	150	300	600	1500	3000	6,000
tert-Amyl methyl ether	10	25	50	100	250	500	1,000
tert-Butyl ethyl ether	10	25	50	100	250	500	1,000
Tetrahydrofuran	4	10	20	40	100	200	400
trans-1,4-Dichloro-2-butene	2	5	10	20	50	100	200
Trichlorofluoromethane	2	5	10	20	50	100	200
Vinyl acetate	4	10	20	40	100	200	400

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 6: Water 8260 List Calibration Levels (µg/L)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1,2-Tetrachloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1,1-Trichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1,2,2-Tetrachloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1,2-Trichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1-Dichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1-Dichloroethene	0.3	1.0	2.0	5.0	10	30	60
1,1-Dichloropropene	0.3	1.0	2.0	5.0	10	30	60
1,2,3-Trichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,2,3-Trichloropropane	0.3	1.0	2.0	5.0	10	30	60
1,2,4-Trichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,2,4-Trimethylbenzene	0.3	1.0	2.0	5.0	10	30	60
1,2-Dibromo-3-chloropropane	0.3	1.0	2.0	5.0	10	30	60
1,2-Dichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,2-Dichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,2-Dichloropropane	0.3	1.0	2.0	5.0	10	30	60
1,3,5-Trimethylbenzene	0.3	1.0	2.0	5.0	10	30	60
1,3-Dichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,3-Dichloropropane	0.3	1.0	2.0	5.0	10	30	60
1,4-Dichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,4-Dioxane	15	50	100	250	500	1,500	3,000
1-Chlorohexane	0.3	1.0	2.0	5.0	10	30	60
2,2-Dichloropropane	0.3	1.0	2.0	5.0	10	30	60
2-Butanone (MEK)	1.2	4.0	8.0	20	40	120	240
2-Chloro-1,3-butadiene (chloroprene)	0.3	1.0	2.0	5.0	10	30	60
2-Chlorotoluene	0.3	1.0	2.0	5.0	10	30	60
2-Hexanone	1.2	4.0	8.0	20	40	120	240
2-Methyl-2-propanol (tert-Butyl alcohol)	6	20	40	100	200	600	1,200
4-Chlorotoluene	0.3	1.0	2.0	5.0	10	30	60
4-Isopropyltoluene	0.3	1.0	2.0	5.0	10	30	60
4-Methyl-2-pentanone	1.2	4.0	8.0	20	40	120	240
Acetone	1.2	4.0	8.0	20	40	120	240
Acetonitrile	3	10	20	50	100	300	600
Acrolein	3	10	20	50	100	300	600
Acrylonitrile	3	10	20	50	100	300	600

Table 6: Water 8260 List Calibration Levels (µg/L)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Benzene	0.3	1.0	2.0	5.0	10	30	60
Bromobenzene	0.3	1.0	2.0	5.0	10	30	60
Bromoform	0.3	1.0	2.0	5.0	10	30	60
Bromomethane	0.3	1.0	2.0	5.0	10	30	60
Carbon tetrachloride	0.3	1.0	2.0	5.0	10	30	60
Chlorobenzene	0.3	1.0	2.0	5.0	10	30	60
Chlorobromomethane	0.3	1.0	2.0	5.0	10	30	60
Chlorodibromomethane	0.3	1.0	2.0	5.0	10	30	60
Chloroethane	0.3	1.0	2.0	5.0	10	30	60
Chloroform	0.3	1.0	2.0	5.0	10	30	60
Chloromethane	0.3	1.0	2.0	5.0	10	30	60
cis-1,2-Dichloroethene	0.3	1.0	2.0	5.0	10	30	60
cis-1,3-Dichloropropene	0.3	1.0	2.0	5.0	10	30	60
Cyclohexanone	12	40	80	200	400	1,200	2,400
Dibromomethane	0.3	1.0	2.0	5.0	10	30	60
Dichlorobromomethane	0.3	1.0	2.0	5.0	10	30	60
Dichlorodifluoromethane	0.3	1.0	2.0	5.0	10	30	60
Ethanol	15	50	100	250	500	1,500	3,000
Ethylbenzene	0.3	1.0	2.0	5.0	10	30	60
Ethylene dibromide (EDB)	0.3	1.0	2.0	5.0	10	30	60
Hexachlorobutadiene	0.3	1.0	2.0	5.0	10	30	60
Iodomethane	0.3	1.0	2.0	5.0	10	30	60
Isopropyl alcohol	6	20	40	100	200	600	1,200
Isopropyl ether	1.5	5.0	10	25	50	150	300
Isopropylbenzene	0.3	1.0	2.0	5.0	10	30	60
m and p Xylenes	0.6	2.0	4.0	10	20	60	120
Methacrylonitrile	3	10	20	50	100	300	600
Methylene chloride	0.3	1.0	2.0	5.0	10	30	60
Naphthalene	0.3	1.0	2.0	5.0	10	30	60
n-Butanol	9.0	30	60	150	300	900	1,800
n-Butylbenzene	0.3	1.0	2.0	5.0	10	30	60
n-Propylbenzene	0.3	1.0	2.0	5.0	10	30	60
o-Xylene	0.3	1.0	2.0	5.0	10	30	60
Propionitrile	3.0	10	20	50	100	300	600
sec-Butylbenzene	0.3	1.0	2.0	5.0	10	30	60

Table 6: Water 8260 List Calibration Levels (µg/L)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Styrene	0.3	1.0	2.0	5.0	10	30	60
tert-Butylbenzene	0.3	1.0	2.0	5.0	10	30	60
Tetrachloroethene	0.3	1.0	2.0	5.0	10	30	60
Tetrahydrothiophene	0.3	1.0	2.0	5.0	10	30	60
Toluene	0.3	1.0	2.0	5.0	10	30	60
trans-1,2-Dichloroethene	0.3	1.0	2.0	5.0	10	30	60
trans-1,3-Dichloropropene	0.3	1.0	2.0	5.0	10	30	60
Trichloroethene	0.3	1.0	2.0	5.0	10	30	60
Trichlorofluoromethane	0.3	1.0	2.0	5.0	10	30	60
Vinyl chloride	0.3	1.0	2.0	5.0	10	30	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 6A: Water 8260 List Calibration Levels (µg/L)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
1,1,1-Trifluoro-2,2-dichloroethane	1.0	2.0	5.0	10	30	60
1,1,2-Trichloro-1,2,2-trifluoroethane	1.0	2.0	5.0	10	30	60
1,2,3-Trimethylbenzene	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2,2-tetrafluoroethane	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2-trifluoroethane	1.0	2.0	5.0	10	30	60
2-Chloroethy vinyl ether	1.0	2.0	5.0	10	30	60
2-Nitropropane	1.0	2.0	5.0	10	30	60
2-Pentanone	4.0	8.0	20	40	120	240
3-Chloro-1-propene (Allyl chloride)	1.0	2.0	5.0	10	30	60
Carbon disulfide	1.0	2.0	5.0	10	30	60
cis-1,4-dichloro-2-butene	1.0	2.0	5.0	10	30	60
Cyclohexane	1.0	2.0	5.0	10	30	60
Dichlorofluoromethane	1.0	2.0	5.0	10	30	60
Ethyl acetate	2.0	4.0	10	20	60	120
Ethyl ether	1.0	2.0	5.0	10	30	60
Ethyl methacrylate	2.0	4.0	10	20	60	120

Table 6A: Water 8260 List Calibration Levels (µg/L)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Ethylene oxide	125	250	625	1,250	3,750	7,500
Hexane	1.0	2.0	5.0	10	30	60
Isobutyl alcohol	20	40	100	200	600	1,200
Methyl acetate	5.0	10	25	50	150	300
Methylcylcohexane	1.0	2.0	5.0	10	30	60
Methyl methacrylate	2.0	4.0	8.0	20	60	120
Methyl *tert*-butyl ether (MTBE)	1.0	2.0	5.0	10	30	60
Propene oxide	20	100	250	500	1,500	3,000
sec-Butyl alcohol	30	60	150	300	900	1,800
tert-Amyl methyl ether	5.0	10	25	50	150	300
tert-Butyl ethyl ether	5.0	10	25	50	150	300
Tetrahydrofuran	2.0	4.0	10	20	60	120
trans-1,4-dichloro-2-butene	1.0	2.0	5.0	10	30	60
Vinyl acetate	2.0	4.0	10	20	60	120

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 7: Medium Level Soil 8260 List Calibration Levels (µg/Kg)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,1-Trichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2-Trichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,2,4-Trichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2,4-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dibromo-3-chloropropane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,3,5-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,3-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,3-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,4-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,4-Dioxane	25	50	100	250	500	1,500	3,000
1-Chlorohexane	0.5	1.0	2.0	5.0	10	30	60
2,2-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
2-Butanone (MEK)	2.0	4.0	8.0	20	40	120	240
2-Chloro-1,3-butadiene (chloroprene)	0.5	1.0	2.0	5.0	10	30	60
2-Chlorotoluene	0.5	1.0	2.0	5.0	10	30	60
2-Hexanone	2.0	4.0	8.0	20	40	120	240
2-Methyl-2-propanol (tert-Butyl alcohol)	10	20	40	100	200	600	1,200
4-Chlorotoluene	0.5	1.0	2.0	5.0	10	30	60
4-Isopropyltoluene	0.5	1.0	2.0	5.0	10	30	60
4-Methyl-2-pentanone	2.0	4.0	8.0	20	40	120	240
Acetone	2.0	4.0	8.0	20	40	120	240
Acetonitrile	5	10	20	50	100	300	600
Acrolein	5	10	20	50	100	300	600
Acrylonitrile	5	10	20	50	100	300	600

Table 7: Medium Level Soil 8260 List Calibration Levels (µg/Kg)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Benzene	0.5	1.0	2.0	5.0	10	30	60
Bromobenzene	0.5	1.0	2.0	5.0	10	30	60
Bromoform	0.5	1.0	2.0	5.0	10	30	60
Bromomethane	0.5	1.0	2.0	5.0	10	30	60
Carbon tetrachloride	0.5	1.0	2.0	5.0	10	30	60
Chlorobenzene	0.5	1.0	2.0	5.0	10	30	60
Chlorobromomethane	0.5	1.0	2.0	5.0	10	30	60
Chlorodibromomethane	0.5	1.0	2.0	5.0	10	30	60
Chloroethane	0.5	1.0	2.0	5.0	10	30	60
Chloroform	0.5	1.0	2.0	5.0	10	30	60
Chloromethane	0.5	1.0	2.0	5.0	10	30	60
cis-1,2-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
cis-1,3-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
Cyclohexanone	20	40	80	200	400	1,200	2,400
Dibromomethane	0.5	1.0	2.0	5.0	10	30	60
Dichlorobromomethane	0.5	1.0	2.0	5.0	10	30	60
Dichlorodifluoromethane	0.5	1.0	2.0	5.0	10	30	60
Ethanol	25	50	100	250	500	1,500	3,000
Ethylbenzene	0.5	1.0	2.0	5.0	10	30	60
Ethylene dibromide (EDB)	0.5	1.0	2.0	5.0	10	30	60
Hexachlorobutadiene	0.5	1.0	2.0	5.0	10	30	60
Iodomethane	0.5	1.0	2.0	5.0	10	30	60
Isopropyl alcohol	10	20	40	100	200	600	1,200
Isopropyl ether	2.5	5.0	10	25	50	150	300
Isopropylbenzene	0.5	1.0	2.0	5.0	10	30	60
m and p Xylenes	1.0	2.0	4.0	10	20	60	120
Methacrylonitrile	5	10	20	50	100	300	600
Methylene chloride	0.5	1.0	2.0	5.0	10	30	60
Naphthalene	0.5	1.0	2.0	5.0	10	30	60
n-Butanol	15	30	60	150	300	900	1,800
n-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60
n-Propylbenzene	0.5	1.0	2.0	5.0	10	30	60
o-Xylene	0.5	1.0	2.0	5.0	10	30	60
Propionitrile	5.0	10	20	50	100	300	600
sec-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60

Table 7: Medium Level Soil 8260 List Calibration Levels (µg/Kg)[1]
(Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Styrene	0.5	1.0	2.0	5.0	10	30	60
tert-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60
Tetrachloroethene	0.5	1.0	2.0	5.0	10	30	60
Tetrahydrothiophene	0.5	1.0	2.0	5.0	10	30	60
Toluene	0.5	1.0	2.0	5.0	10	30	60
trans-1,2-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
trans-1,3-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
Trichloroethene	0.5	1.0	2.0	5.0	10	30	60
Trichlorofluoromethane	0.5	1.0	2.0	5.0	10	30	60
Vinyl chloride	0.5	1.0	2.0	5.0	10	30	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 7A: Medium Level Soil 8260 List Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1-Trifluoro-2,2-dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2-Trichloro-1,2,2-trifluoroethane	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2,2-tetrafluoroethane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2-trifluoroethane	0.5	1.0	2.0	5.0	10	30	60
2-Chloroethy vinyl ether	0.5	1.0	2.0	5.0	10	30	60
2-Nitropropane	0.5	1.0	2.0	5.0	10	30	60
2-Pentanone	2.0	4.0	8.0	20	40	120	240
3-Chloro-1-propene (Allyl chloride)	0.5	1.0	2.0	5.0	10	30	60
Carbon disulfide	0.5	1.0	2.0	5.0	10	30	60
cis-1,4-dichloro-2-butene	0.5	1.0	2.0	5.0	10	30	60
Cyclohexane	0.5	1.0	2.0	5.0	10	30	60
Dichlorofluoromethane	0.5	1.0	2.0	5.0	10	30	60
Ethyl acetate	1.0	2.0	4.0	10	20	60	120
Ethyl ether	0.5	1.0	2.0	5.0	10	30	60
Ethyl methacrylate	1.0	2.0	4.0	10	20	60	120

Table 7A: Medium Level Soil 8260 List Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Ethylene oxide	62.5	125	250	625	1,250	3,750	7,500
Hexane	0.5	1.0	2.0	5.0	10	30	60
Isobutyl alcohol	10	20	40	100	200	600	1,200
Methyl acetate	2.5	5.0	10	25	50	150	300
Methylcylcohexane	0.5	1.0	2.0	5.0	10	30	60
Methyl methacrylate	1.0	2.0	4.0	8.0	20	60	120
Methyl *tert*-butyl ether (MTBE)	0.5	1.0	2.0	5.0	10	30	60
Propene oxide	10	20	100	250	500	1,500	3,000
sec-Butyl alcohol	15	30	60	150	300	900	1,800
tert-Amyl methyl ether	2.5	5.0	10	25	50	150	300
tert-Butyl ethyl ether	2.5	5.0	10	25	50	150	300
Tetrahydrofuran	1.0	2.0	4.0	10	20	60	120
trans-1,4-dichloro-2-butene	0.5	1.0	2.0	5.0	10	30	60
Vinyl acetate	1.0	2.0	4.0	10	20	60	120

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 8. Manually Added Internal Standards

Internal Standard	Standard Concentration (µg/mL)	Quantitation Ion
Fluorobenzene	20	96
Chlorobenzene-d_5	20	119
1,4-Dichlorobenzene-d_4	20	152

NOTES:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal standard in the sample at 10 µg/L for a 20 mL purge.

2) Except for high-level soils, the surrogate and internal standards may be combined in one solution.

Table 8A. Automatically Added Internal Standards

Internal Standard	Standard Concentration (µg/mL)	Quantitation Ion
Fluorobenzene	250	96
Chlorobenzene-d_5	250	119
1,4-Dichlorobenzene-d_4	250	152

NOTES:

1) 1 µL of the internal standard is added to the sample. This results in a concentration of each internal standard in the sample at 10 µg/L for a 20 mL purge.

2) There may be some variability in the size of the internal standard loop from one instrument to the next. This is compensated for on the day of initial calibration by comparing the manually added and automatically added internal standard concentrations.

Table 9. Manually Added Surrogate Standards

Surrogate Compounds	Standard Concentration (µg/mL)
1,2-Dichloroethane-d_4	20
Dibromofluoromethane	20
Toluene-d_8	20
4-Bromofluorobenzene	20

NOTES:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample at 10 µg/L for a 20 mL purge.

2) Except for high-level soils, the surrogate and internal standards may be combined in one solution.

3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 9A. Surrogate Standards

Surrogate Compounds	Standard Concentration (µg/mL)
1,2-Dichloroethane-d_4	250
Dibromofluoromethane	250
Toluene-d_8	250
4-Bromofluorobenzene	250

NOTES:

1) 1 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample at 10 µg/L for a 20 mL purge.

2) There may be some variability in the size of the surrogate standard loop from one instrument to the next. This is compensated for on the day of initial calibration by comparing the manually added and automatically added surrogate standard concentrations.

3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 10. Matrix Spike and LCS Standard

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	40
Methylene Chloride	40
Trans-1,2-Dichloroethene	40
1,1-Dichloroethane	40
1111-Trichloroethane	40
Carbon Tetrachloride	40
Benzene	40
Trichloroethene	40
1,2-Dichloropropane	40
Bromodichloromethane	40
Toluene	40
Tetrachloroethene	40
Chlorobenzene	40
Ethylbenzene	40
1,4-Dichlorobenzene	40
1,3-Dichlorobenzene	40

NOTES:

1) 2.5 µL of the standard is added to the LCS or matrix spike sample. This results in a concentration of each spike analyte in the sample of 5 µg/L for a 20 mL purge.
2) Recovery and precision limits for the LCS, MS, and MSD are generated from historical data and are maintained by the QA department.
3) Full analyte spikes or different compounds may also be used at the laboratory's option or at client request.

Table 11. BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40 % of Mass 95
75	30 to 60 % of Mass 95
95	Base Peak, 100 % Relative Abundance
96	5 to 9 % of Mass 95
173	Less than 2 % of Mass 174
174	Greater than 50 % of Mass 95
175	5 to 9 % of Mass 174
176	Greater than 95 %, but less than 101 % of Mass 174
177	5 to 9 % of Mass 176

Table 12. SPCC Compounds and Minimum Response Factors

Compound	8260B Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	> 0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 13. CCC Compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	≤ 30.0	≤ 20.0
1,1-Dichloroethene	≤ 30.0	≤ 20.0
Chloroform	≤ 30.0	≤ 20.0
1,2-Dichloropropane	≤ 30.0	≤ 20.0
Toluene	≤ 30.0	≤ 20.0
Ethylbenzene	≤ 30.0	≤ 20.0

Table 14. Characteristic Ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	--
Dichlorodifluoromethane	85	87	50, 101,103
Dibromofluoromethane	111	113	--
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	--
Trichlorotrifluoroethane	151	101	153
Ethanol	45	46	--
Acetone	43	58	--
Methylene chloride	84	49	51, 86
Tert-Butyl alcohol	59	74	--
Trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		--
Hexane	57	43	--
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	--
Tetrahydrofuran	42	71	--
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	--
Vinyl acetate	43	86	--
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121

Table 14. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
Benzene	78	52	77
Trichloroethene	95	130***	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	--
Xylenes	106	91	--
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
Trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	41	76	78
Acetonitrile	41	40	--
Dichlorofluoromethane	67	69	--
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74

Table 14. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	--
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	--
2,2-Dichloropropane	77	97	--
Bromochloromethane	128	49	130
1,1-Dichloropropene	75	39	110
1,3-Dichloropropane	76	41	78
1-Chlorohexane	91	55	41
1,1,1,2-Tetrachloroethane	131	133	--
Bromobenzene	156	158	77
n-Propylbenzene	120	91	65
2-Chlorotoluene	126	91	65
1,3,5-Trimethylbenzene	105	120	77
4-Chlorobenzene	126	91	89
t-Butylbenzene	119	134	91
sec-Butylbenzene	134	105	--
4-Isopropyltoluene	119	134	91
n-Butylbenzene	91	92	134
1,2,4-Trichlorobenzene	180	182	--
Hexachlorobutadiene	225	227	223
Naphthalene	128	127	--
1,2,3-Trichlorobenzene	180	182	--
1,1,1-Trifluoro-2,2-Dichloroethane	83	133	--
1,2,3-Trimethylbenzene	105	120	91
1,2,4-Trimethylbenzene	105	120	119
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	85	87	--
1,2-Dichloro-1,1,2-Trifluoroethane	117	67	85
1,3,5-Trichlorobenzene	180	182	184
2,2,3-Trimethylbutane	57	43	85
2,2-Dimethylpentane	57	43	85
2,3-Dimethylpentane	56	71	73

Table 14. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
2,4-Dimethylpentane	43	57	85
2-Chloro-1,1,1-Trifluoroetane	118	83	69
2-Methylhexane	43	85	57
3,3-Dimethylpentane	43	71	--
3-Ethylpentane	43	70	71
3-Methylhexane	43	57	71
4-Chlorotoluene	126	91	89
2-Pentanone	43	86	--
Chlorotrifluoroethene	116	66	97
Cis-1,4-Dichloro-2-butene	53	75	89
Cyclohexane	56	84	55
Dimethyl Disulfide	94	79	45
Ethylene Dibromide	107	109	--
Ethylene Oxide	43	44	--
Isopropyl Alcohol	45	43	--
Methyl Acetate	43	74	59
Methylcyclohexane	55	83	41
m-Xylene & p-Xylene	91	106	77
n-Heptane	43	100	71
n-Nonyl Aldehyde	46	44	207
O-Xylene	106	91	--
Propene Oxide	58	43	57
Sec-Butyl Alcohol	45	59	--
Tert-amyl methyl ether	73	55	87
Tert-butyl ethyl ether	59	87	57
Tetrahydrothiophene	60	88	45

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-butanone, but m/z 72 must be present for positive identification.

*** Used as quantitation ion for method 624.

Table 15. State of Arizona ICV/CCV Quality Control Limits

QC Limits not specified in method	Default QC (method specified or laboratory historical if not specified)
CCV Non-CCC compounds	CCC limits (≤30%)
ICV	Same as CCV (≤30%)
Reporting Limit	Must be supported by low level initial calibration standard
LCS/LCSD	Lab historical
MS/MSD	Lab historical

NOTES:

1) Based on ADHS Rule A.A.C.R9-14-615.C.8. Director approved on June 29, 2005 for the labs to use default limits as an alternative to developing statistically derived limits.

Table 16. List 1 Poorly Performing Compounds

The laboratory's GC/MS group identified this list of compounds based on current and historical performance. The recovery performance was reviewed against full spike recovery data and method performance data, where available, to validate each compound as a "poor performer."

Acetone	1,2-Dichloro-1,1,2,2-tetrafluoroethane
Acetonitrile	Ethanol
Acrolein	Ethyl acetate
Acrylonitrile	Ethylene oxide
n-Butanol	2-Hexanone
2-Butanone (MEK)	Isobutyl alcohol
tert-Butyl alcohol	Isopropanol
Carbon disulfide	Methacrylonitrile
2-Chloroethyl vinyl ether	Methyl acetate
2-Chloro-1,1,1-trifluoroethane	4-Methyl-2-pentanone
Chlorotrifluoroethene	2-Nitropropane
cis-1,4-Dichloro-2-butene	2-Pentanone
trans-1,4-Dichloro-2-butene	2-Propanol
Dichlorodifluoromethane	Propionitrile
Dichlorofluoromethane	Tetrahydrofuran
1,2-Dibromo-3-chloropropane (DBCP)	Tetrahydrothiophene
1,2-Dichlorotetrafluoroethane	1,1,2-Trichloro-1,2,2-trifluoroethane
1,2-Dichloro-1,1,2-trifluoroethane (Freon 123a)	Trichlorofluoromethane
2,2-Dichloro-1,1,1-trifluoroethane	Vinyl acetate
1,4-Dioxane	

Table A-1. Method 624 Analytes and Reporting Limits, 5-mL Purge

Analytes	µg/L
Acrolein[1]	100
Acrylonitrile[1]	100
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	10
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	10
2-Chloroethyl vinyl ether	5
Chloroform	5
Chloromethane	10
Dibromochloromethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	10
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	15
Vinyl chloride	10

[1] Acrolein and Acrylonitrile have been added to the 624 analyte list in the EPA Method Update Rule, May 18, 2012. Analysis of these analytes by Method 624 as being regulatory compliant is dependent upon individual state approval of the MUR. Verify state status before analysis.

Table A-2. Method 624 QC Acceptance Criteria

Analytes[1]	Daily QC Check (CCV) Acceptance Criteria (20 µg/L spike)	Mean Recovery, Initial Demonstration Acceptance Criteria (IDOC) (20 µg/L spike)	Std Dev, Initial Demonstration Acceptance Criteria (IDOC) (20 µg/L spike)	Matrix Spike and LCS Acceptance Criteria (% Recovery)
Acrolein[2]	45.9-54.1	42.9-60.1	4.6	88-118
Acrylonitrile[2]	41.2-58.8	33.1-66.9	9.9	71-135
Benzene	12.8 - 27.2	15.2 - 26.0	6.9	37 - 151
Bromodichloromethane	13.1 - 26.9	10.1 - 28.0	6.4	35 - 155
Bromoform	14.2 - 25.8	11.4 - 31.1	5.4	45 - 169
Bromomethane	2.8 - 37.2	D - 41.2	17.9	D - 242
Carbon tetrachloride	14.6 - 25.4	17.2 - 23.5	5.2	70 - 140
Chlorobenzene	13.2 - 26.8	16.4 - 27.4	6.3	37 - 160
Chloroethane	7.6 - 32.4	8.4 - 40.4	11.4	14 - 230
2-Chloroethyl vinyl ether	D - 44.8	D - 50.4	25.9	D - 305
Chloroform	13.5 - 26.5	13.7 - 24.2	6.1	51 - 138
Chloromethane	D - 40.8	D - 45.9	19.8	D - 273
Dibromochloromethane	13.5 - 26.5	13.8 - 26.6	6.1	53 - 149
1,2-Dichlorobenzene	12.6 - 27.4	11.8 - 34.7	7.1	18 - 190
1,3-Dichlorobenzene	14.6 - 25.4	17.0 - 28.8	5.5	59 - 156
1,4-Dichlorobenzene	12.6 - 27.4	11.8 - 34.7	7.1	18 - 190
1,1-Dichloroethane	14.5 - 25.5	14.2 - 28.5	5.1	59 - 155
1,2-Dichloroethane	13.6 - 26.4	14.3 - 27.4	6.0	49 - 155
1,1-Dichloroethene	10.1 - 29.9	3.7 - 42.3	9.1	D - 234
trans-1,2-Dichloroethene	13.9 - 26.1	13.6 - 28.5	5.7	54 - 156
1,2-Dichloropropane	6.8 - 33.2	3.8 - 36.2	13.8	D - 210
cis-1,3-Dichloropropene	4.8 - 35.2	1.0 - 39.0	15.8	D- 227
trans-1,3-Dichloropropene	10.0 - 30.0	7.6 - 32.4	10.4	17- 183
Ethylbenzene	11.8 - 28.2	17.4 - 26.7	7.5	37 - 162
Methylene chloride	12.1 - 27.9	D - 41.0	7.4	D - 221
1,1,2,2-Tetrachloroethane	12.1 - 27.9	13.5 - 27.2	7.4	46 - 157
Tetrachloroethene	14.7 - 25.3	17.0 - 26.6	5.0	64 - 148
Toluene	14.9 - 25.1	16.6 - 26.7	4.8	47 - 150
1,1,1-Trichloroethane	15.0 - 25.0	13.7 - 30.1	4.6	52 - 162
1,1,2-Trichloroethane	14.2 - 25.8	14.3 - 27.1	5.5	52 - 150
Trichloroethene	13.3 - 26.7	18.6 - 27.6	6.6	71 - 157
Trichlorofluoromethane	9.6 – 30.4	8.9- 31.5	10.0	17 - 181
Vinyl chloride	0.8 – 39.2	D - 43.5	20.0	D - 251

[1] Analytes not listed on the table must meet a CCV drift criteria of ± 30%. Method 624 does not specify second source (ICV) criteria. The laboratory has adopted criteria of ± 30% difference for the ICV. The LIMS requires a minimum value of 10% for the lower limit when D is noted in the reference table.

[2] Acrolein and Acrylonitrile have been added to the 624 analyte list in the EPA Method Update Rule, May 18, 2012. Analysis of these analytes by Method 624 as being regulatory compliant is dependent upon individual state approval of the MUR. Verify state status before analysis. Per the MUR, QC criteria from Method 603 are to be applied and are presented here.

Table A-3. Calibration Levels for 624, 5 mL Purge

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Acetone	20	40	80	200	400
Acrolein	50	100	200	500	1000
Acrylonitrile	50	100	200	500	1000
Benzene	5.0	10	20	50	100
Bromoform	5.0	10	20	50	100
Bromomethane	5.0	10	20	50	100
2-Butanone	20	40	80	200	400
Carbon disulfide	5.0	10	20	50	100
Carbon tetrachloride	5.0	10	20	50	100
Chlorobenzene	5.0	10	20	50	100
Chlorodibromomethane	5.0	10	20	50	100
Chloroethane	5.0	10	20	50	100
2-Chloroethyl vinyl ether	5.0	10	20	50	100
Chloroform	5.0	10	20	50	100
Chloromethane	5.0	10	20	50	100
1,2-Dibromo-3-chloropropane	5.0	10	20	50	100
1,2-Dibromoethane (EDB)	5.0	10	20	50	100
Dibromomethane	5.0	10	20	50	100
1,2-Dichlorobenzene	5.0	10	20	50	100
1,3-Dichlorobenzene	5.0	10	20	50	100
1,4-Dichlorobenzene	5.0	10	20	50	100
Dichlorobromomethane	5.0	10	20	50	100
Dichlorodifluoromethane	5.0	10	20	50	100
1,1-Dichloroethane	5.0	10	20	50	100
1,2-Dichloroethane	5.0	10	20	50	100
cis-1,2-Dichloroethene	5.0	10	20	50	100
trans-1,2-Dichloroethene	5.0	10	20	50	100
1,1-Dichloroethene	5.0	10	20	50	100
1,2-Dichloropropane	5.0	10	20	50	100
cis-1,3-Dichloropropene	5.0	10	20	50	100
trans-1,3-Dichloropropene	5.0	10	20	50	100
1,4-Dioxane	250	500	1000	2500	5000

Table A-3. Calibration Levels for 624, 5 mL Purge (cont.)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Ethylbenzene	5.0	10	20	50	100
Hexane	5.0	10	20	50	100
2-Hexanone	20	40	80	200	400
Methylene chloride	5.0	10	20	50	100
4-Methyl-2-pentanone (MIBK)	20	40	80	200	400
Methyl *tert*-butyl ether (MTBE)	5.0	10	20	50	100
Styrene	5.0	10	20	50	100
1,1,1,2-Tetrachloroethane	5.0	10	20	50	100
1,1,2,2-Tetrachloroethane	5.0	10	20	50	100
Tetrachloroethene	5.0	10	20	50	100
Toluene	5.0	10	20	50	100
1,1,1-Trichloroethane	5.0	10	20	50	100
1,1,2-Trichloroethane	5.0	10	20	50	100
Trichloroethene	5.0	10	20	50	100
Trichlorofluoromethane	5.0	10	20	50	100
1,2,3-Trichloropropane	5.0	10	20	50	100
Vinyl acetate	5.0	10	20	50	100
Vinyl chloride	5.0	10	20	50	100
m- and p-Xylenes	10	20	40	100	200
o-Xylene	5.0	10	20	50	100

If the response factor (RF) is constant over the working range (<35% RSD), the average RF may used for calculations. Alternatively, a calibration curve may be used if the correlation coefficient is ≥ 0.99.

APPENDIX A

Modifications for Analysis of 1,4-Dioxane, 1,2,3-Trichloropropane, 1,2-Dibromo-3-chloropropane, and 1,2-Dibromoethane by Selected Ion Monitoring

1.0 REQUIREMENTS FOR METHOD 8260 SELECTED ION MONITORING (SIM)

1.1 The gas chromatograph/mass spectrometer (GCMS) is utilized in the SIM mode to obtain lower reporting limits. The standard analyte list and reporting limits are listed in Table Ap-1.

1.2 This method can be applied to aqueous and solid matrices.

1.3 The sample preparation is the same as defined in section 10.1.1 through 10.1.3 in this SOP, DV-MS-0010.

1.4 The tune period for this method is defined as 12 hours. Instrument tuning is described in section 10.1.11.3 above.

1.5 Initial calibration curve requirements are as follows:

1.5.1 Same as for 8260 detailed in Section 10.1.12 of this SOP.

1.5.2 The calibrations levels are shown in Table Ap-2.

1.6 Continuing calibration verification requirements are as follows:

1.6.1 The %drift for 1,4-dioxane must be $\leq$ 25% for the continuing calibration to be valid.

1.6.2 In addition, the %drift for the surrogate compounds should be $\leq$ 25%.

1.7 Matrix Spike and LCS requirements are as follows:

1.7.1 The spike levels are listed in Table Ap-3.

1.8 Internal Standards: The internal standard concentrations are listed in Table Ap-5.

1.9 Surrogates: The surrogate concentrations are listed in Table Ap-4.

1.10 Instrument Conditions are shown in Table Ap-7.

Table Ap-1.

TAL Method 8260SIM Standard Reporting Limits

Analytes	CAS Number	Aqueous, µg/L	Solid, µg/Kg
1,4-Dioxane	123-91-1	2.0	5.0
1,2-Dibromo-3-chloropropane	96-12-8	0.02	1.0
1,2-Dibromoethane	106-93-4	0.02	1.0
1,2,3-Trichloropropane	96-18-4	0.02	1.0

Table Ap-2.

Method 8260SIM Calibration Levels

Calibration Level	1,4-Dioxane Aqueous Calibration Concentration, µg/L	EDB,DBCP,TCP Aqueous Calibration Concentration, µg/L	1,4-Dioxane Solid Calibration Concentration, µg/Kg	EDB,DBCP,TCP Solid Calibration Concentration, µg/Kg
1	NA	0.02	1.0	1.0
2	NA	0.05	2.0	2.0
3	0.2	0.2	4.0	4.0
4	1.0	1.0	8.0	8.0
5	2.0	2.0	16.0	16.0
6	5.0	5.0	32.0	32.0
7	10.0	10.0	48.0	48.0
8	20.0	20.0	NA	NA
SSV	5.0	5.0	16.0	16.0

Table Ap-3.

Method 8260SIM LCS Spike Concentrations

LCS Compounds	Aqueous Spiking Level, µg/L	Solid Spiking Level, µg/Kg
1,4-Dioxane	5.0	20
1,2-Dibromo-3-chloropropane	1.0	8
1,2-Dibromoethane	1.0	8
1,2,3-Trichloropropane	1.0	8

Table Ap-4.

8260SIM Surrogate Compounds

Surrogate Compounds	Aqueous Spiking Level, µg/L[1]	Solid Spiking Level, µg/Kg[1]
Dibromofluoromethane	12.5	50
1,2-Dichloroethane-d_4	12.5	50
Toluene-d_8	12.5	50
4-Bromofluorobenzene	12.5	50

[1] – Exact spike levels are dependent upon the calibration of the autosampler loop used for the addition of the surrogate spike solution.

Table Ap-5.

8260SIM Internal Standard Compounds

Surrogate Compounds	Aqueous Spiking Level, µg/L	Solid Spiking Level, µg/Kg
Fluorobenzene	12.5	50
Chlorobenzene-d_5	12.5	50
1,4-Dichlorobenzene-d_4	12.5	50

Table Ap-6.

8260 Selected Masses

Compound	Quant	Qualifier Ion
1,4-Dioxane	88	58
Fluorobenzene	96	70
Chlorobenzene-d_5	119	117
1,4-Dichlorobenzene-d_4	152	150
Dibromofluoromethane	111	113
1,2-Dichloroethane-d_4	65	102
Toluene-d_8	98	70
4-Bromofluorobenzene	95	174
1,2-Dibromo-3-chloropropane	157	155
1,2-Dibromoethane	107	109
1,2,3-Trichloropropane	110	75

Table Ap-7.

Suggested Instrument Conditions for 8260SIM

Selected Masses:	See Table Ap-6
Dwell Time:	≥ 30 milliseconds
Initial Column Temperature/Hold Time:	50 °C for 2 minutes
Column Temperature Program:	50 - 160 °C at 30°C/min, 160 - 220 °C at 60°C/min .
Final Column Temperature/Hold Time:	220 °C/4.3 min hold
Injector Temperature:	220 °C
Transfer Line Temperature:	260 °C
Source Temperature:	240 °C
Trap Desorb Temperature:	270 °C
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 1.3mL/min.
Column:	DB-624 Capillary 60m x 0.25mm x 1.8 um film thickness, or equivalent

APPENDIX B

Modifications for Analysis of Soils Collected for the State of Alaska

1. Collection and Preservation Requirements

Preservation and Holding Time for Volatiles in Soil
Method 5035A for Alaska

Container/Contents[1]	Preservation	Holding time	Analysis
Vial containing methanol and TFT surrogate	Sample is extruded into pre-tared 4 oz jar, containing 25 mL of methanol spiked with 2.5 ppm (ug/mL) of α, α, α-trifluorotoluene, cooled to ≤ 6°C and frozen upon receipt at laboratory.	14 days	Medium Level

Sample weights are calculated in the laboratory by adding the received weight of the sample into the AK Methanol Volume Correction spreadsheet stored on G:\QA\Edit\FORMS\GCMS.

2. Sample Preparation for Medium-Level Analysis – Field Preserved, AK method

a. Fill a 40 mL VOA vial with reagent water ~ 42 mL (no head space), and remove 1000 µL of water using a volumetric pipette or syringe.

b. Add 1050 µL of methanol extract to the vial and immediately cap. Invert the vial to ensure that there is no air bubble larger than 4 mm present. If a > 4 mm air bubble is present, re-prepare the sample.

c. Load the sample in the auto sampler and proceed to analyze against the methanol calibration curve.

d. As with water samples, surrogate and internal standard solutions are added by the autosampler (see Tables 7 and 7A in the main body of this SOP). The surrogate α, α, α-trifluorotoluene is added to the samples at the time of sampling. Recoveries for this surrogate will be reported in addition to recoveries for the surrogate compounds added at the time of analysis.

e. Prepare laboratory control samples by filling a 40 mL VOA vial with reagent water, and remove 1000 µL of water using a volumetric pipette or syringe. Add reagents as needed plus sufficient methanol for a total methanol volume of 1050 µL. The recommended concentration for the LCS is the same as the Level 5 of the initial calibration curve.

f. Remove a portion of the methanol extract for each sample and store in a clean Teflon-capped vial with no headspace at ≤ 6 °C until analysis. Duplicate aliquots of the methanol extract should be taken and stored.

3. Percent Moisture Correction for Soils from the State of Alaska

A percent moisture correction is required for soil samples submitted from the state of AK to adjust the extraction final volume in order to allow for the miscible solvent effects. The following formula is used to determine the corrected final volume. This calculation is performed in the AK Methanol Volume Correction spreadsheet stored on G:\QA\Edit\FORMS\GCMS.

a. $$V_t = [V_m + (M * W_s/100)]$$

Where:

V_t = final extract volume, corrected for moisture (mL)

V_m = volume methanol used for extraction (mL)

M = moisture content of the sample (%)

W_s = aliquot of sample extracted (g)

Table Bp-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
Dichlorodifluoromethane	75-71-8	80
Chloromethane	74-87-3	40
Bromomethane	74-83-9	40
Vinyl chloride	75-01-4	40
Chloroethane	75-00-3	40
n-Butanol	71-36-3	800
Trichlorofluoromethane	75-69-4	40
Acrolein	107-02-8	200
Acetone	67-64-1	400
Trichlorotrifluoroethane	76-13-1	400
Iodomethane	74-88-4	500
Carbon disulfide	75-15-0	40
Methylene chloride	75-09-2	40
tert-Butyl alcohol	75-65-0	800
1,1-Dichloroethene	75-35-4	40
1,1-Dichloroethane	75-34-3	40
trans-1,2-Dichloroethene	156-60-5	40
Acrylonitrile	107-13-1	400
Methyl *tert*-butyl ether (MTBE)	1634-04-4	200
Hexane	110-54-3	400
cis-1,2-Dichloroethene	156-59-2	40
1,2-Dichloroethene (Total)	540-59-0	40
Tetrahydrofuran	109-99-9	80
Chloroform	67-66-3	40
1,2-Dichloroethane	107-06-2	40
Dibromomethane	74-95-3	40
2-Butanone	78-93-3	160
1,4-Dioxane	123-91-1	2,000
1,1,1-Trichloroethane	71-55-6	40
Carbon tetrachloride	56-23-5	40
Bromodichloromethane	75-27-4	40
1,2-Dichloropropane	78-87-5	40
Isopropyl Alcohol	67-63-0	1,000
Isopropyl ether	108-20-3	200

Table Bp-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
cis-1,3-Dichloropropene	10061-01-5	40
Trichloroethene	79-01-6	40
Dibromochloromethane	124-48-1	40
1,2-Dibromoethane	106-93-4	40
1,2,3-Trichloropropane	96-18-4	40
1,1,2-Trichloroethane	79-00-5	40
Benzene	71-43-2	16
Ethylmethacrylate	97-63-2	80
trans-1,3-Dichloropropene	10061-02-6	40
Bromoform	75-25-2	40
4-Methyl-2-pentanone	108-10-1	160
2-Hexanone	591-78-6	160
Tetrachloroethene	127-18-4	40
Toluene	108-88-3	40
1,1,2,2-Tetrachloroethane	79-34-5	40
2-Chloroethyl vinyl ether	110-75-8	80
Vinyl acetate	108-05-4	80
Chlorobenzene	108-90-7	40
Ethylbenzene	100-41-4	40
Styrene	100-42-5	40
trans-1,4-Dichloro-2-butene	110-57-6	400
m- and p-Xylenes	179601-23-1	80
o-Xylene	95-47-6	40
Total xylenes	1330-20-7	80
1,3-Dichlorobenzene	541-73-1	40
1,4-Dichlorobenzene	106-46-7	40
1,2-Dichlorobenzene	95-50-1	40
2,2-Dichloropropane	590-20-7	40
Bromochloromethane	74-97-5	40
1,1-Dichloropropene	563-58-6	40
1,3-Dichloropropane	142-28-9	40
1-Chlorohexane	544-10-5	80
1,1,1,2-Tetrachloroethane	630-20-6	40

Table Bp-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
Isopropylbenzene	98-82-8	40
Bromobenzene	108-86-1	40
n-Propylbenzene	103-65-1	40
2-Chlorotoluene	95-49-8	40
4-Chlorotoluene	106-43-4	40
1,3,5-Trimethylbenzene	108-67-8	40
tert-Butylbenzene	98-06-6	40
1,2,4-Trimethylbenzene	95-63-6	40
sec-Butylbenzene	135-98-8	40
4-Isopropyltoluene	99-87-6	40
n-Butylbenzene	104-51-8	40
1,2-Dibromo-3-chloropropane	96-12-8	200
1,2,4-Trichlorobenzene	120-82-1	40
Naphthalene	91-20-3	40
Hexachlorobutadiene	87-68-3	40
1,2,3-Trichlorobenzene	87-61-6	40
Propionitrile	107-12-0	400
Cylcohexanone	108-94-1	1,600
Methyl methacrylate	80-62-6	80
Acetonitrile	75-05-8	400
Methacrylonitrile	126-98-7	400
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	76-14-2	160
1,2-Dichloro-1,1,2-trifluoroethane	354-23-4	160
2-Pentanone	107-87-9	600
cis-1,4-Dichloro-2-butene	1476-11-5	400
Cyclohexane	110-82-7	40
Methyl acetate	79-20-9	200
Methylcyclohexane	108-87-2	160
2-Chloro-1,3-butadiene	126-99-8	80
2-Methyl-2-propanol	75-65-0	800

Table Bp-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
tert-Butyl ethyl ether	637-92-3	80
1,2,3-Trimethylbenzene	526-73-8	40
Ethyl acetate	141-78-6	80
Ethyl ether	60-29-7	200
Isobutyl alcohol	78-83-1	800
Dichlorofluoromethane	75-43-4	120
Tetrahydrothiophene	110-01-0	40

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table Bp-2								
Calibration Levels for 8260, 5035FM_AK (ug/Kg)								
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	**Level 8**
1,1,1,2-Tetrachloroethane	20	40	80	200	600	2000	4000	8000
1,1,1-Trichloroethane	20	40	80	200	600	2000	4000	8000
1,1,2,2-Tetrachloroethane	20	40	80	200	600	2000	4000	8000
1,1,2-Trichloroethane	20	40	80	200	600	2000	4000	8000
1,1-Dichloroethane	20	40	80	200	600	2000	4000	8000
1,1-Dichloroethene	20	40	80	200	600	2000	4000	8000
1,1-Dichloropropene	20	40	80	200	600	2000	4000	8000
1,2,3-Trichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2,3-Trichloropropane	20	40	80	200	600	2000	4000	8000
1,2,4-Trichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2,4-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,2-Dibromo-3-chloropropane	20	40	80	200	600	2000	4000	8000
1,2-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2-Dichloroethane	20	40	80	200	600	2000	4000	8000
1,2-Dichloropropane	20	40	80	200	600	2000	4000	8000
1,3,5-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,3-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,3-Dichloropropane	20	40	80	200	600	2000	4000	8000
1,4-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,4-Dioxane	1000	2000	4000	10000	30000	100000	200000	400000
1-Chlorohexane	20	40	80	200	600	2000	4000	8000
2,2-Dichloropropane	20	40	80	200	600	2000	4000	8000
2-Butanone (MEK)	80	160	320	800	2400	8000	16000	32000
2-Chloro-1,3-butadiene (chloroprene)	20	40	80	200	600	2000	4000	8000
2-Chlorotoluene	20	40	80	200	600	2000	4000	8000
2-Hexanone	80	160	320	800	2400	8000	16000	32000
2-Methyl-2-propanol (tert-Butyl alcohol)	400	800	1600	4000	12000	40000	80000	160000
4-Chlorotoluene	20	40	80	200	600	2000	4000	8000
4-Isopropyltoluene	20	40	80	200	600	2000	4000	8000
4-Methyl-2-pentanone	80	160	320	800	2400	8000	16000	32000
Acetone	80	160	320	800	2400	8000	16000	32000
Acetonitrile	200	400	800	2000	6000	20000	40000	80000
Acrolein	200	400	800	2000	6000	20000	40000	80000
Acrylonitrile	200	400	800	2000	6000	20000	40000	80000

Table Bp-2								
Calibration Levels for 8260, 5035FM_AK (ug/Kg)								
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	**Level 8**
Benzene	20	40	80	200	600	2000	4000	8000
Bromobenzene	20	40	80	200	600	2000	4000	8000
Bromoform	20	40	80	200	600	2000	4000	8000
Bromomethane	20	40	80	200	600	2000	4000	8000
Carbon tetrachloride	20	40	80	200	600	2000	4000	8000
Chlorobenzene	20	40	80	200	600	2000	4000	8000
Chlorobromomethane	20	40	80	200	600	2000	4000	8000
Chlorodibromomethane	20	40	80	200	600	2000	4000	8000
Chloroethane	20	40	80	200	600	2000	4000	8000
Chloroform	20	40	80	200	600	2000	4000	8000
Chloromethane	20	40	80	200	600	2000	4000	8000
cis-1,2-Dichloroethene	20	40	80	200	600	2000	4000	8000
cis-1,3-Dichloropropene	20	40	80	200	600	2000	4000	8000
Cyclohexanone	20	40	80	200	300	1000	2000	4000
Dibromomethane	20	40	80	200	600	2000	4000	8000
Dichlorobromomethane	20	40	80	200	600	2000	4000	8000
Dichlorodifluoromethane	20	40	80	200	600	2000	4000	8000
Ethanol	1000	2000	4000	10000	30000	100000	200000	400000
Ethylbenzene	20	40	80	200	600	2000	4000	8000
Ethylene dibromide (EDB)	20	40	80	200	600	2000	4000	8000
Hexachlorobutadiene	20	40	80	200	600	2000	4000	8000
Iodomethane	20	40	80	200	600	2000	4000	8000
Isopropyl alcohol	400	800	1600	4000	12000	40000	80000	160000
Isopropyl ether	100	200	400	1000	3000	10000	20000	40000
Isopropylbenzene	20	40	80	200	600	2000	4000	8000
m- and p-Xylenes	40	80	160	400	1200	4000	8000	16000
Methacrylonitrile	200	400	800	2000	6000	20000	40000	80000
Methylene chloride	20	40	80	200	600	2000	4000	8000
Naphthalene	20	40	80	200	600	2000	4000	8000
n-Butanol	600	1200	2400	6000	18000	60000	120000	240000
n-Butylbenzene	20	40	80	200	600	2000	4000	8000
n-Propylbenzene	20	40	80	200	600	2000	4000	8000
o-Xylene	20	40	80	200	600	2000	4000	8000
Propionitrile	200	400	800	2000	6000	20000	40000	80000
sec-Butylbenzene	20	40	80	200	600	2000	4000	8000
Styrene	20	40	80	200	600	2000	4000	8000

Table Bp-2								
Calibration Levels for 8260, 5035FM_AK (ug/Kg)								
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	**Level 8**
tert-Butylbenzene	20	40	80	200	600	2000	4000	8000
Tetrachloroethene	20	40	80	200	600	2000	4000	8000
Tetrahydrothiophene	20	40	80	200	600	2000	4000	8000
Toluene	20	40	80	200	600	2000	4000	8000
trans-1,2-Dichloroethene	20	40	80	200	600	2000	4000	8000
trans-1,3-Dichloropropene	20	40	80	200	600	2000	4000	8000
Trichloroethene	20	40	80	200	600	2000	4000	8000
Trichlorofluoromethane	20	40	80	200	600	2000	4000	8000
Vinyl chloride	20	40	80	200	600	2000	4000	8000

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table Bp-3: 5035FM_AK Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1,1,1-Trifluoro-2,2-dichloroethane	20	40	80	200	600	2000	4000	8000
1,1,2-Trichloro-1,2,2-trifluoroethane	20	40	80	200	600	2000	4000	8000
1,2,3-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,2-Dichloro-1,1,2,2-tetrafluoroethane	20	40	80	200	600	2000	4000	8000
1,2-Dichloro-1,1,2-trifluoroethane	20	40	80	200	600	2000	4000	8000
2-Chloroethy vinyl ether	20	40	80	200	600	2000	4000	8000
2-Nitropropane	20	40	80	200	600	2000	4000	8000
2-Pentanone	80	160	320	800	2400	8000	16000	32000
3-Chloro-1-propene (Allyl chloride)	20	40	80	200	600	2000	4000	8000
Carbon disulfide	20	40	80	200	600	2000	4000	8000
cis-1,4-dichloro-2-butene	20	40	80	200	600	2000	4000	8000
Cyclohexane	20	40	80	200	600	2000	4000	8000
Dichlorofluoromethane	20	40	80	200	600	2000	4000	8000
Ethyl acetate	40	80	160	400	1200	4000	8000	16000
Ethyl ether	20	40	80	200	600	2000	4000	8000

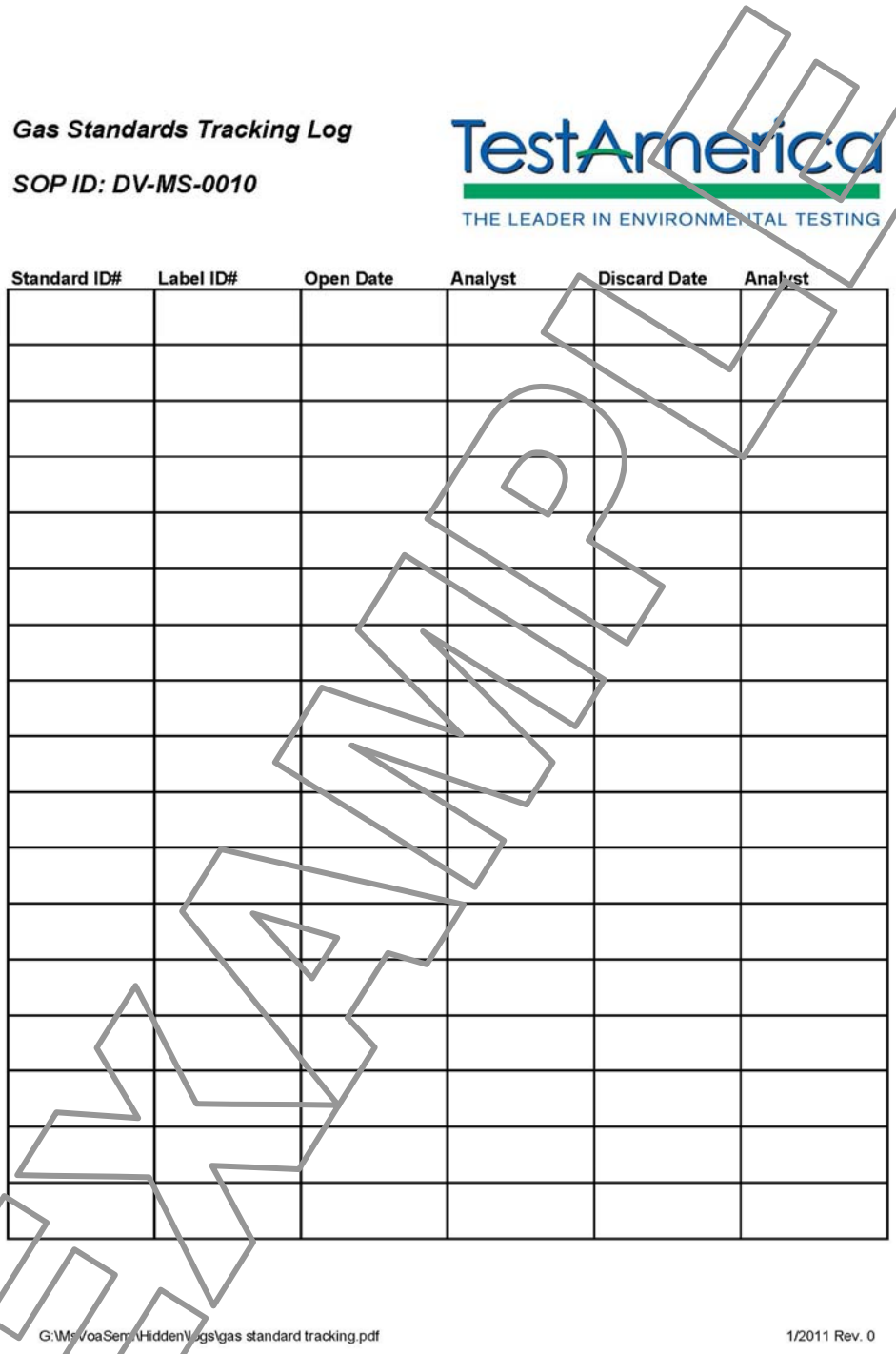
Table Bp-3: 5035FM_AK Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Ethyl methacrylate	40	80	160	400	1200	4000	8000	16000
Ethylene oxide	2500	5000	10000	25000	75000	250000	500000	1000000
Hexane	20	40	80	200	600	2000	4000	8000
Isobutyl alcohol	400	800	1600	4000	12000	40000	80000	160000
Methyl acetate	100	200	400	1000	3000	10000	20000	40000
Methylcylcohexane	20	40	80	200	600	2000	4000	8000
Methyl methacrylate	40	80	160	400	1200	4000	8000	16000
Methyl *tert*-butyl ether (MTBE)	20	40	80	200	600	2000	4000	8000
Propene oxide	400	800	1600	4000	12000	40000	80000	160000
sec-Butyl alcohol	600	1200	2400	6000	18000	60000	120000	240000
tert-Amyl methyl ether	100	200	400	1000	3000	10000	20000	40000
tert-Butyl ethyl ether	100	200	400	1000	3000	10000	20000	40000
Tetrahydrofuran	40	80	160	400	1200	4000	8000	16000
trans-1,4-dichloro-2-butene	20	40	80	200	600	2000	4000	8000
Vinyl acetate	40	80	160	400	1200	4000	8000	16000

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Attachment 1

Gas Standards Tracking Log



Gas Standards Tracking Log

SOP ID: DV-MS-0010

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Standard ID#	Label ID#	Open Date	Analyst	Discard Date	Analyst

G:\MS\VoaSemi\Hidden\Logs\gas standard tracking.pdf 1/2011 Rev. 0

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: GC/MS Analysis Based On Methods 8270C and 625

Approvals (Signature/Date):

William Rhoades 4/26/10
William Rhoades Date
Technical Manager

Adam W. Alban 27 Apr 10
Adam Alban Date
Health & Safety Manager / Coordinator

Karen Kuoppala 4.24.10
Karen Kuoppala Date
Quality Assurance Manager

Robert C Hanisch 4/27/10
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This method is based upon standard method SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices.

1.1.1 The modifications presented in Appendix A may be followed for analysis of wastewater following method 625.

1.1.2 The modifications presented in Appendix B may be followed for analysis of wastewater following method 8270 (best practices).

1.1.3 Direct injection of a sample may be used in limited applications.

1.1.4 Refer to Tables 1 and 2 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. This method may be amenable to additional compounds. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as ¾-methylphenol.
- Hexachlorophene and famphur analysis are not quantitatively reliable by this method.
- Kepone should be analyzed by GC/ECD.

1.3 The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

2.0 Summary of Method

2.1 Aqueous samples are extracted with methylene chloride using a continuous extractor or a separatory funnel.

2.2 Solid samples are extracted with methylene chloride / acetone using sonication or Soxhlet extraction. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS.

2.3 Waste dilution is used for samples that are miscible with the solvent.

2.4 Extraction procedures are detailed in the following SOPs:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series
DV-OP-0007	Concentration of Organic Extracts, SW846 3510C, 3520C, 3540C, 3550B, and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0016	Ultrasonic Extraction of Solid Samples, SW846 3550
DV-OP-0010	Soxhlet Extraction of Solid Samples, SW846 3540C

2.5 Qualitative identification of the analytes in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3.0 Definitions

3.1 CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCCs is specified for calibration acceptance.

3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / matrix spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.

3.4 Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica QC Program document (DV-QA-003P) for further details of the batch definition.

3.5 Method Blank (MB) - An analytical control consisting of all reagents, internal standards and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.6 Laboratory Control Sample (LCS) - A blank matrix (reagent water or Ottawa Sand) spiked with the analytes of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked analytes demonstrates that the laboratory techniques for this method are acceptable.

3.7 Matrix Spike (MS) – An aliquot of a matrix (water or soil) fortified (spiked) with known amounts of specific analytes and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.8 Matrix Spike Duplicate (MSD) - A second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method by measuring the relative percent difference (RPD) between the MS and MSD results.

3.9 Surrogates - Organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, MS, and MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

4.0 Interferences

4.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample. Cleanup procedures may help to eliminate select interferences, as follows:

- Method 3640A, Gel-Permeation Chromatography - Removes higher molecular weight hydrocarbons by size exclusion chromatography, which is most frequently used for biological samples
- Method 3660B, Sulfur Cleanup – If a sulfur peak is detected, copper or mercury can be used to treat the extract and remove the sulfur
- Other, more aggressive cleanup procedures listed in SW-846 may be used for select compounds listed in this procedure, but may cause degradation of some of the more reactive compounds. Consult with a technical expert in the laboratory for more difficult interference problems.

Details concerning cleanup steps are described in the organic extraction SOPs (see Section 2.4).

4.2 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section (Section

9.3). Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.3 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

NOTE: Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.

5.1.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power

before performing any maintenance.

5.1.5 The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and must be done either in a hood with the sash down to chest level or while wearing a face shield over safety glasses.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Significant or Serious Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Sodium Hydroxide	Corrosive	2 mg/m3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system: an analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Column: 30 m x 0.25 mm I.D., 0.5-µm film thickness fused-silica capillary column coated with 5% diphenyl/95% dimethyl polysiloxane(Restek Rtx®-5MS or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 u (previously "amu") every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) that meets all of the criteria in Table 4 when 25 ng of the GC/MS tuning standard is injected through the GC.

6.4 Autosampler: LEAP Technologies CTC A200S, HP7683 Autosampler or equivalent.

6.5 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.6 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.7 Syringe: 10 µL or 5µL Hamilton Laboratory grade syringes or equivalent. The 5 µL syringe is used for the Agilent ALS to be able to inject 0.5 µL.

6.8 Carrier gas: Ultra high-purity helium.

6.9 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

7.1 A minimum five-point calibration curve is prepared when average response factors or linear regression curve fitting is used. Six calibration points are required for second-order curve fits. The low point should be at or below the reporting limit. Refer to tables 11 and 12 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2 An internal standard (IS) solution is prepared. Compounds in the IS Mix are acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.2.1 Internal standards are added to all standards and extracts to result in a final concentration of 40 µg/mL. For example, if the volume of an extract aliquot used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added to the aliquot. See Appendix B for the levels used for the 8270 best practice method.\

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). Surrogate compounds and levels are listed in Table 9.

Acid Surrogates	Base Surrogates
2-Fluorophenol	2-Fluorobiphenyl
2,4,6-tribromophenol	Terphenyl-d4
Phenol-d5	Nitrobenzene-d5
2-chlorophenol-d4	1,2,-Dichlorobenzene-d4

7.4 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT should also be included in the Tuning Standard at 50 µg/mL.

7.5 Laboratory Control Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). LCS compounds and levels are listed in Table 7.

7.6 Matrix Spike Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). The matrix spike compounds and levels are the same as the LCS compounds.

7.7 The standards listed in sections 7.1 to 7.6 must be refrigerated at -10°C to -20°C if it can be demonstrated that analytes do not fall out of solution at these temperatures. If not stable, the standards should be stored at 4 ± 2 °C. The standard stock solutions expire after one year from preparation date or at the earliest expiration date assigned by the vendor to any parent standard, whichever is earlier. The continuing calibration standard should be replaced every week, when there are visible signs of degradation, or when the standard fails to meet QC criteria. The continuing calibration standard is stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	1 liter amber	1 Liter	Cool 4 ± 2°C	7 Days	40 Days from extraction	40 CFR Part 136.3
Soils	4oz Jar	30 grams	Cool 4 ± 2°C	14 Days	40 Days from extraction	N/A

9.0 Quality Control

9.1 Initial Performance Studies

9.1.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL). See Section 13 for a discussion of detection limit studies.

9.1.2 In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument they will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details.

9.2 Control Limits

9.2.1 In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits are determined

every 6 months. The recovery limits are the mean recovery ±3 standard deviations for surrogates, MS, and LCS. Precision limits for the MS/MSD pair results is the absolute value of the mean relative percent difference (RPD) +3 standard deviations.

9.2.2 These limits do not apply to dilutions, but surrogate and matrix spike recoveries will be reported unless the dilution is 4x or more.

9.2.3 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into the LIMS or other database so that accurate historical control limits can be generated. For multiple dilutions reported from the same extract, surrogates will be reported for all dilutions of less than 4x. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.4 Refer to the Quality Assurance Program document, DV-QA-003P, for further details of control limits.

9.3 Method Blank (MB)

For aqueous sample batches, the method blank is reagent water; for solid sample batches, the method blank is clean sand. In either case, the method blank is free of the analytes of interest and is spiked with the surrogates. At least one method blank must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than ½ of the reporting limit or less than 10% of the analyte concentration found in the associated samples, whichever is higher. When a compound is above ½ the reporting limit a NCM needs to be completed.

NOTE: All programs require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: Re-preparation and reanalysis of all samples associated with an unacceptable method blank. If the analyte was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.4 Instrument Blank

Instruments must be evaluated for contamination during each 12-hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.5 Laboratory Control Sample (LCS)

The LCS is prepared using reagent water for aqueous methods and Ottawa sand for solid sample methods. A laboratory control sample (LCS) is prepared and analyzed with every

batch of samples. The LCS is spiked with the compounds listed in Table 8 unless specified by a client or agency. The compounds must be spiked at a concentration equivalent to 100 or 150 µg/L, depending on the analyte, unless a special QAS states a specific level. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Acceptance Criteria: All analytes must be within established control limits. See Quality Assurance Program DV-QA-003P for details on establishing control limits.

Corrective Action: If any analyte in the LCS is outside the laboratory-established historical control limits or project-specific control limits, as applicable, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.
- If re-extraction and reanalysis of the batch are not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The matrix spike is a second aliquot of one of the samples in the batch. The matrix spike duplicate is a third aliquot of the same sample. The MS and MSD are spiked with the same analytes as the LCS (See Tables 9 and 10). An MS/MSD pair is prepared and analyzed with every batch of samples.

Acceptance Criteria: The percent recovery (%R) must fall within either historical limits or project-specific limits, as applicable. The relative percent difference (RPD) between the MS and MSD results must be less than or equal to the established historical or project-specific limit. See Quality Assurance Program Policy DV-QA-003P for details on establishing control limits

Corrective Action: If any individual recovery or RPD fails the acceptance criteria, then corrective action must occur. Initially check the recovery of the analyte in question in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is considered to be in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any analyte fails acceptance criteria for the MS, MSD, and the LCS, the laboratory operation is considered to be out of out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.
- The MS/MSD pair must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted to concentrations below the calibration range.

9.7 Surrogates

9.7.1 Each sample, blank, and QC sample is spiked with the surrogate standards. Surrogate compounds must be spiked at either 100 or 150 ug/L, depending on the surrogate. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 9. For the Best Practice method, see table B-4 in Appendix B.

Acceptance Criteria: Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

Corrective Action: If any surrogates are outside of the limits, then the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

NOTE: The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.7.2 If the sample with failed surrogate recoveries was a sample used for an MS/MSD pair and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample and the MS and the MSD do not require reanalysis. This phenomenon indicates a possible matrix problem.

9.7.3 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only

the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate).

9.7.4 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effects.

9.8 Nonconformance and Corrective Action

9.8.1 Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The nonconformance shall be addressed in the case narrative, and the NCM shall be filed in the project file. The NCM process is described in more detail in SOP DV-QA-0031.

9.8.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. and approved by a supervisor and QA Manager.

9.8.3 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.9 Quality Assurance Summaries (QAS) or Program Distillations

Certain clients may require specific project or program QC that may supersede the requirements presented in this section. Quality Assurance Summaries (also known as Program Distillations) should be developed to address these requirements.

9.10 TestAmerica Quality Assurance Program

Details of the TestAmerica Denver Quality Assurance Program, including corrective action guidelines, are presented in DV-QA-003P, Quality Assurance Program. Refer to this document if in doubt regarding corrective actions.

10.0 Procedure

10.1 Sample Preparation

Samples are prepared according to the following organic preparation SOPs, as applicable:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series

DV-OP-0007	Concentration of Organic Extracts, SW846 3510C, 3520C, 3540C, 3550B, and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0016	Ultrasonic Extraction of Solid Samples, SW846 3550

10.2 Sample Analysis Procedure

10.2.1 Calibrate the instrument as described in Section 11. Depending on the target compounds required by the client, it may be necessary to use more than one set of calibration standards.

10.2.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration verification (CCV) standard.

10.2.3 Add internal standard to an aliquot of the extract to result in a 40-ng/µL concentration (for example, 20 µL of internal standard solution at, 400 µg/mL in 200 µL of extract). Mix thoroughly before injection into the instrument.

10.2.4 Inject the aliquot into the GC/MS system using the same injection technique as used for the standards.

10.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration verification.

10.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst (see DV-QA-0033, Acceptable Manual Integration Practices) or automatically by the data system. The minimum documentation required includes a hard copy of original data system peak integration and a similarly scaled hard copy showing the manual integration with analyst initials and date.

10.2.7 Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

10.2.8 Library searches of peaks present in the chromatogram that are not target compounds, i.e., Tentatively Identified Compounds (TIC), may be performed if required by the client. They are evaluated using the criteria in Section 12.2.

10.3 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.3.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are

significantly less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

10.3.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, freeze them at <-10°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

10.5 Retention Time Criteria for Samples

10.5.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.5.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

10.6 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP DV-WC-0023 for determination of percent moisture.

10.7 Procedural Variations

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described

10.8 Troubleshooting Guide

10.8.1 Daily Instrument Maintenance

In addition to the checks listed in Appendix D, the following daily maintenance should be performed.

- Clip Column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Install new or cleaned gold seal and washer as necessary.

- Perform mass calibration as necessary.

10.8.2 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the repeller, cleaning the source, replacing the multiplier, and replacing the "top board" or RF-related electronics. Refer to the manufacturer's manual for specific guidance.

11.0 Calibration

11.1 Summary

The instrument is tuned for DFTPP, calibrated initially with a minimum of a five levels, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 3.

11.2 All standards and extracts are allowed to warm to room temperature before injecting.

11.3 Instrument Tuning

At the beginning of every twelve-hour shift when analyses are to be performed, the GC/MS system must be checked to see if the acceptance criteria are achieved for DFTPP (decafluorotriphenylphosphine). See Table 4.

11.3.1 Inject 25 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 4 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

11.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. The acceptance criteria for the peak tailing factor for benzidine is < 3.0 and pentachlorophenol is < 5.0. DDT breakdown must be <20%. Refer to section 12 for the appropriate calculations.

11.4 Initial Calibration

11.4.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)*.

11.4.2 Internal Standard (IS) Calibration Procedure: Internal standards are listed in Table 5. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

11.4.3 Compounds are assigned to the IS with the closest retention time.

11.4.4 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest when average response factors or linear

regression curve fits are used. Six standards must be used for a quadratic least-squares calibration. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response.

11.4.5 For AFCEE projects, the five calibration levels will be those shown in Table 10. The table also lists a sixth calibration level that is used if a second-order regression fit is needed. The only exceptions would be for the AFCEE projects requiring special reporting limits, i.e., reporting limits different than those in the AFCEE program QAPP. Additional calibration points may be required for special projects.

11.4.6 Rejection of Calibration Points

11.4.6.1 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

11.4.6.2 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious misinjection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

- The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and
- The lowest remaining calibration point is still at or below the project reporting limit; and
- The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and
- The calibration must still have the minimum number of calibration levels required by the method, i.e. five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

11.4.7 Add the internal standard mixture to result in a 40-ng/µL final concentration. (For example, if the volume of the calibration standard used is 0.5 mL, add 50 µL of the 400 µg/mL internal standard). The concentrations of all analytes are listed in Tables 11 and 12. For the Best Practice method, see Table B-5 in Appendix B.

11.4.8 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against the concentration for each compound and internal standard. Calculate the response factors (RF), average response

factors, and the percent RSD of the response factors for each compound using the equations in section 12. Verify that the CCC and SPCC criteria, which are specified in Sections 11.4.9 and 11.4.10 are met. No sample analysis may be performed unless these criteria are met.

11.4.9 System Performance Check Compounds (SPCCs)

The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

11.4.9.1 SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

11.4.10 Calibration Check Compounds (CCCs)

The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this check.

11.4.10.1 If none of the CCCs are required analytes, then project-specific calibration specifications (which may include the use of the CCCs listed in Section 11.4.10.2) must be implemented with concurrence from the client.

11.4.10.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

11.4.11 If the average of all RSDs in the initial calibration is < 15%, then all analytes may use average response factor for calibration.

NOTE: Some states (like Arizona) and federal programs do not allow the use of grand mean. Refer to the Arizona QAS and SOP DV-QA-024P.

11.4.11.1 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary

calibration reports can be generated, then the analyst should evaluate analytes with RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve fit, then the appropriate curve should be used for quantitation.

11.4.11.2 If the average of all the RSDs in the initial calibration is > 15%, then calibration using a curve fit, must be used for those analytes with RSD > 15%. Linear or quadratic curve fits may be used. Use of 1/Concentration2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

11.4.11.3 If a linear regression equation is used, the correlation coefficient r must be greater than 0.990, and r square (r2) greater than 0.9801 Use of second-order regression equations may be used on rare occasions. In these cases, the intercept and degree of curvature should be examined to be sure that results will be reliable throughout the working range, and the coefficient of determination must be greater than 0.990.

Note: South Carolina can only be analyzed using linear calibration.

11.4.11.4 An initial calibration verification containing all components from a second source (an alternate vendor, or, a unique lot from the same vendor, or, the same source but prepared by an alternate analyst) must be analyzed after the initial calibration. Acceptance criteria for ICV percent recovery (%R) are 75-125% for DoD projects (e.g., AFCEE); 65-135% for non-DoD projects (e.g., 625/8270C HSL components); and 45-155% for poor performers (e.g., 8270C AP9, Custom, Refinery, DBP, benzaldehyde).

11.4.12 Weighting of Calibration Data Points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/Concentration2 weighting (often called 1/X2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability. Because the data system does not indicate the type of weighting used, the analyst must make a notation on the initial calibration form as to the weighting used (e.g. $1/x$ or $1/x^2$).

11.4.13 If time remains in the 12-hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration, Section 11.5.

NOTE: Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

11.5 Continuing Calibration Verification (CCV)

11.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 25-ng injection of DFTPP must result in a mass spectrum for DFTPP, which meets the criteria given in Table 4.

11.5.2 Following a successful DFTPP analysis, the continuing calibration verification (CCV) standard(s) are analyzed. The standard(s) must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the CCV.

11.5.3 The following criteria must be met for the CCV to be acceptable:

- The SPCC compounds must have a response factor ≥ 0.050.
- The percent difference or drift (%D) of the CCC compounds must be ≤ 20%. (See Section 12 for calculations.)
- For compounds of interest, reliably performing compounds (see Table 14, List 1 Reliably Performing Compounds) should have a %D ≤ 35%. Poorly performing compounds (see Table 15, List 2 Poorly Performing Compounds) should have a %D ≤ 50%, with allowance for up to 6 target analytes to have %D values greater than the applicable limit. Any compound of interest that does not meet the applicable criteria will be narrated.
- The internal standard response of the CCV must be within 50 - 200% of the response in the same level of the corresponding calibration.
- If any internal standard retention time in the CCV changes by more than 30 seconds from that of the same level of the corresponding initial calibration, the chromatographic system must be inspected for malfunctions and corrections made, as required.

11.5.3.1 If none of the CCCs are required analytes, project-specific calibration requirements (which may include the use of the CCCs listed in Section 11.4.10.2) must be implemented with concurrence from the client.

11.5.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample injected less than or equal to 12 hours after the DFTPP is acceptable.)

NOTE: Some states (like Arizona) have special requirements. Please refer to the posted QAS.

12.0 Calculations / Data Reduction

12.1 Qualitative Identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention

time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

12.1.1 The sample component relative retention time must compare to within ± 0.06 RRT units of the relative retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

12.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.1.3 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.1.4 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.5 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Following are guidelines for making tentative identification:

12.2.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

12.2.2 The relative intensities of the major ions should agree to within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)

12.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or the presence of co-eluting compounds.

12.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.2.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.3 Isomers with identical mass spectra and close elution times pose problems for definitive identification. The following compounds fall into this category:

Aniline and bis(2-chloroethyl) ether
Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Identification of these compounds requires both experience and extra precautions on the part of the analyst. Specifically, the analyst must more closely scrutinize the comparison of retention times between the unknown and the calibration standard. The analyst must also check that all isomers have distinct retention times.

12.4 A second category of problem compounds consist of the poor responders or compounds that chromatograph poorly. The integrations for these types of compounds should be checked manually. The following compounds are included in this category:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol
Ariazine
Famphur
Benzidine

12.5 Calculating the Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{RF} \times 100\%$$

Where:

RF = Mean of RFs from the initial calibration for a compound

SD = Standard deviation for the mean RF from the initial calibration for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}}$$

RFi = RF for each of the calibration levels

n = Number of RF values

12.6 Calculating the Continuing Calibration Percent Drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

Where:

Cactual = Known concentration in standard

Cfound = Measured concentration using selected quantitation method

12.7 Calculating the Concentration in the Extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.7.1 Average Response Factor Calibration

If the average of all the RSDs of the response factors in the initial calibration is ≤15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

Where:

Cex = Concentration in the extract, µg/mL

Rx = Response for the analyte

Ris = Response for the internal standard

Cis = Concentration of the internal standard

$\overline{RF}$ = Average response factor

12.7.2 Linear Fit Calibration

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

Where:

Cex	=	Concentration in the extract, µg/mL
Rx	=	Response for the analyte
Ris	=	Response for the internal standard
Cis	=	Concentration of the internal standard
A	=	Intercept of linear calibration line
B	=	Slope of linear calibration line

12.7.3 Quadratic Fit Calibration

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

Where:

Cex	=	Concentration in the extract, µg/mL
Rx	=	Response for the analyte
Ris	=	Response for the internal standard
Cis	=	Concentration of the internal standard
A	=	Intercept
B	=	Factor for the linear term of the quadratic calibration function
C	=	Factor for the curvature term of the quadratic calibration function

12.8 Calculating the Concentration in the Sample

12.8.1 Calculation for Aqueous Samples

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where:

Cex	=	Concentration in the extract
Vt	=	Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that Vt = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then Vt = 2,000.)
Vo	=	Volume of the sample that was extracted (mL)

12.8.2 Calculation for Sediment, Soil, Sludge, and Waste Samples

Results for sediments, sludges, and soils are usually calculated on a dry-weight basis, and for waste, on a wet-weight basis.

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

Where:

Cex = Concentration in the extract

Vt = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that Vt = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then Vt = 2,000.)

Ws = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry-weight basis or 1 for a wet-weight basis

12.9 MS/MSD Percent Recovery Calculation

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

SSR = Spike sample result

SR = Sample result

SA = Spike added

12.10 Calculating the Relative Percent Difference (RPD) MS/MSD Pair

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

12.11 Relative Response Factor Calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

Ax = Area of the characteristic ion for the compound being measured

Ais = Area of the characteristic ion for the specific internal standard

Cx = Concentration of the compound being measured (µg/L)

Cis = Concentration of the specific internal standard (µg/L)

12.12 Calculation of TICs

The calculation of TICs (tentatively identified compounds) is identical to the above calculation (12.11) with the following exceptions:

Ax = Area of the total ion chromatogram for the compound being measured

Ais = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

12.13 Calculating Percent DDT Breakdown

$$\%\ \text{DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDDarea}}$$

The areas for the 235 ion are used for this calculation.

12.14 Calculating the Peak Tailing Factor

$$TailingFactor = \frac{BC}{AB}$$

Where:

Peak width (AC) is measured at 10% peak height, and divided into two line segments at the peak centroid, so that .

AC = AB + BC, with

AB = left-hand segment

BC = right-hand segment

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

An initial MDL study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows

13.1.1 Prepare seven replicates at three to five times the estimated MDL concentration.

13.1.2 Extract and analyze the MDL standards as described in Section 10.

13.1.3 Calculate the mean concentration found (X) in µg/L, and the standard deviation of the mean concentration in µg/L, for each analyte. Then calculate the MDL (single-tailed, 99% confidence level, as described in Policy DV-QA-005P) for each analyte.

13.1.4 MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy DV-QA-005P for further details concerning MDL studies.

13.1.5 The current MDL value is maintained in the TestAmerica Denver LIMS.

13.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard prepared at 1-2 times the calculated MDL concentration. An MDLV standard is analyzed immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

Acceptance Criteria:	The calculated MDL is verified if the MDLV standard is detected, nominally signal to noise ratio ≥ 3, under routine instrument conditions.
Corrective Actions:	If the first MDLV is not detected, the MDLV standard will be reprepared and analyzed at twice the original concentration. The lowest concentration that produces a detectable signal will then be reported as the MDL.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

13.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.3.2 Calculate the mean recovery and standard deviation for each analyte of interest.

13.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

13.4.1 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

13.4.2 Non-standard Analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration should include the analysis of an extracted standard at the reporting limit and a single point calibration.

14.0 Pollution Control

14.1 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Methylene Chloride- B

15.2.3 Flammable Solvent- Waste Stream C

15.2.4 Used vials- Waste Stream A

NOTE: Radioactive, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.2 40CFR, part 136, Appendix A, "Base/Neutrals and Acids", Method 625.

17.0 Method Modifications:

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.3 This procedure includes the option for weighted linear regression curves using 1/concentration2 weighting factors. Section 7.5.2 of Method 8000B discusses the use of weighted least square regression based on 1/standard deviation2 weighting factors, which would require multiple analyses of each standard to determine the standard deviation. IAETL has presented information to the EPA Office of Solid Waste demonstrating that the variance (standard deviation2) is proportional to the standard concentration. EPA accepted this argument and issued a letter in July 1998, which authorizes the use of 1/concentration2 weighting factors.

18.0 Attachments

Table 1. TestAmerica Primary Standard and Standard Reporting Limits
Table 2. TestAmerica Appendix IX Standard Reporting Limits
Table 3. Suggested Instrument Conditions
Table 4. DFTPP Key Ions and Ion Abundance Criteria
Table 5. Characteristic Ions, Primary Standard (in approximate retention time order)
Table 6. Characteristic Ions, Appendix IX Standard (in approximate retention time order)
Table 7. 8270C LCS Compounds
Table 8. TCLP LCS Compounds
Table 9. 8270C Surrogate Compounds
Table 10. Calibration Levels for AFCEE Projects, µg/mL
Table 11. Calibration Levels, Primary Standard, µg/mL
Table 12. Calibration Levels, Appendix IX Standard, µg/mL
Table 13. Initial Demonstration Recovery and Precision Limits
Table 14. List 1 Reliably Performing Compounds
Table 15. List 2 Poorly Performing Compounds
APPENDIX A. Modifications Required for Analysis of Wastewater Following Method 625
Table A-1. TestAmerica Method 625 Standard Reporting List and Reporting Limits
Table A-2. Method 625 LCS and MS Compounds and Spike Concentrations
APPENDIX B. Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (8270BP)
Table B-1. TestAmerica Method 8270BP Standard Reporting Limits
Table B-2. Method 8270BP Calibration Levels
Table B-3. Method 8270BP LCS Spike Concentrations
Table B-4. 8270BP Surrogate Compounds
Table B-5 8270BP Internal Standard Compounds
Table B-6. Suggested Instrument Conditions for 8270BP

APPENDIX C. Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

19.0 Revision History

- Revision 5.2, dated 04 May 2010
  - Annual Review
  - Added section 6.9.

- Revision 5.1, dated 17 April 2009
  - Updated Table 8 to contain a longer list of LCS compounds.
  - Corrected several references to incorrect sections.
  - Removed all references to the isotope dilution method.
  -
- Revision 5, dated 20 March 2008
  - Integration for TestAmerica and STL operations.
  - Revised Tables 1 and 2 to reflect current reporting limits.
  - Removed the use of average average from the calibration section 11.4.10.

Changes fromPrevious Major Revision

- Removed the modifications for 1,4-dioxane by isotope dilution, and included this compound in Appendix B, 8270 Best Practice.

Table 1.

TAL Primary Standard and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	4	330
1,4-Dichlorobenzene	106-46-7	4	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	4	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)2	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	4	330
2-Nitroaniline	88-74-4	10	1600
Dimethyl phthalate	131-11-3	4	330
Acenaphthylene	208-96-8	4	330
3-Nitroaniline	99-09-2	10	1600
Acenaphthene	83-32-9	4	330
2,4-Dinitrophenol	51-28-5	30	1600
4-Nitrophenol	100-02-7	10	1600
Dibenzofuran	132-64-9	4	330
2,4-Dinitrotoluene	121-14-2	10	330

Table 1.

TAL Primary Standard and Standard Reporting Limits (cont.)

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	4	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	4	330
4-Nitroaniline	100-01-6	10	1600
4,6-Dinitro-2-methylphenol	534-52-1	20	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	4	330
Anthracene	120-12-7	4	330
Carbazole	86-74-8	4	330
Di-n-butyl phthalate	84-74-2	4	330
Fluoranthene	206-44-0	4	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	4	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	4	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
4,4-Methylenebis(2-chloroaniline)	101-14-4	100	330
Chrysene	218-01-9	4	330
Di-n-octylphthalate	117-84-0	4	330
Benzo(b)fluoranthene	205-99-2	4	330
Benzo(k)fluoranthene	207-08-9	4	330
Benzo(a)pyrene	50-32-8	4	330
Indeno(1,2,3-cd)pyrene	193-39-5	4	330
Diethyl phthalate	84-66-2	4	660
Dibenz(a,h)anthracene	53-70-3	4	330
Benzo(g,h,i)perylene	191-24-2	4	330
Acetophenone	98-86-2	10	330
3/4-Methylphenol	108-39-4	10	330
1,4-Dioxane	54841-74-6	20	660

1. The TAL primary standard is the standard normally used at TAL. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.
2. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether.

Table 2.

TAL Appendix IX Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	10	660
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	1600
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	50	1600
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin	297-97-2	10	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp	3689-24-5	50	1000
Phorate	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate	2303-16-4	20	660
Dimethoate	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	10	660
Methyl Parathion	298-00-0	50	1600
1-chloronaphthalene	90-13-1	10	330
Biphenyl	92-51-3	10	330

Table 2.

TAL Appendix IX Standard Reporting Limits (cont.)

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
Isodrin	465-73-6	10	330
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	20	660
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	10	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660
Diphenylamine	122-39-4	10	330

1. The Appendix IX standard contains additional analytes required for the Appendix IX list. The TAL primary standard must also be analyzed to include all of the Appendix IX list.
2. May also be analyzed by method 8141, which can achieve lower reporting limits.
3. May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits.

Table 3.

Suggested Instrument Conditions

Mass Range:	35 - 500 amu
Scan Time:	≤ 1 second/scan
Initial Column Temperature/Hold Time:	40 °C for 1 minute
Column Temperature Program:	40 - 325 °C at 25 °C/min.
Final Column Temperature/Hold Time:	325 °C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature:	250 °C
Transfer Line Temperature:	290 °C
Source Temperature:	According to manufacturer's specifications
Injector:	Grob-type, split / splitless
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 3.4 mL/min.

Table 4.

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	40 - 100% of mass 198
443	17 - 23% of mass 442

Table 5.

Characteristic Ions, Primary Standard (in approximate retention time order)

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
1,4-Dioxane	88	58	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	77
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	271
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153

Table 5.

Characteristic Ions, Primary Standard (in approximate retention time order) (cont.)

Analyte	Primary	Secondary	Tertiary
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	105	51
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Famphur	218	93	125
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
4,4-Methylenebis(2-Chloroanaline)	231	266	-
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 6.

Characteristic Ions, Appendix IX Standard (in approximate retention time order)

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	77
3/4-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	54
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	50	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169	168	115
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160

Table 6.

Characteristic Ions, Appendix IX Standard (in approximate retention time order) (cont.)

Analyte	Primary	Secondary	Tertiary
Parathion	109	97	291
Isodrin	193	66	195
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 7.

8270C LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
1,2,4-Trichlorobenzene	100
Acenaphthene	100
2,4-Dinitrotoluene	100
Pyrene	100
N-Nitroso-di-n-propylamine	100
1,4-Dichlorobenzene	100
2-Methylnaphthalene	100
Carbazole	100
Anthracene	100
Pentachlorophenol	150
Phenol	150
2-Chlorophenol	150
4-Chloro-3-methylphenol	150
4-Nitrophenol	150
2,4,6-Trichlorophenol	150
2-Methylphenol	150

Table 8.

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
1,4-Dichlorobenzene	50
2,4-Dinitrotoluene	50
Hexachlorobenzene	50
Hexachlorobutadiene	50
Hexachloroethane	50
2-Methylphenol	50
3/4-Methylphenol	100
Nitrobenzene	50
Pentachlorophenol	100
Pyridine	50
2,4,5-Trichlorophenol	50
2,4,6-Trichlorophenol	50

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA group.

Table 9.

8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d5	100
2-Fluorobiphenyl	100
Terphenyl-d14	100
1,2-Dichlorobenzene-d4[1]	100
Phenol-d5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d4[1]	150

1. Included in standard mix, but not routinely evaluated for method 8270C

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 10.

Calibration Levels for AFCEE Projects, µg/mL

Analyte	Std Conc 1	Std Conc 2	Std Conc 3	Std Conc 4	Std Conc 5	Std Conc 6	Addnl Conc for 2nd Order ICALs
Pyridine	20	50	80	120	200	---	160
N-nitrosodimethylamine	10	20	50	80	120	200	---
Aniline	10	20	50	80	120	200	---
Phenol	10	20	50	80	120	200	---
Bis(2-chloroethyl)ether	10	20	50	80	120	200	---
2-Chlorophenol	10	20	50	80	120	200	---
1,3-Dichlorobenzene	10	20	50	80	120	200	---
1,4-Dichlorobenzene	10	20	50	80	120	200	---
Benzyl alcohol	10	20	50	80	120	200	---
1,2-Dichlorobenzene	10	20	50	80	120	200	---
2-Methylphenol	10	20	50	80	120	200	---
2,2'-oxybis(1-chloropropane)[1]	10	20	50	80	120	200	---
4-Methylphenol	10	20	50	80	120	200	---
N-Nitroso-di-n-propylamine	10	20	50	80	120	200	---
Hexachloroethane	10	20	50	80	120	200	---
Nitrobenzene	10	20	50	80	120	200	---
Isophorone	10	20	50	80	120	200	---
2-Nitrophenol	10	20	50	80	120	200	---
2,4-Dimethylphenol	10	20	50	80	120	200	---
Benzoic acid	50	50	80	120	200	---	160
Bis(2-chloroethoxy)methane	10	20	50	80	120	200	---
2,4-Dichlorophenol	10	20	50	80	120	200	---
1,2,4-Trichlorobenzene	10	20	50	80	120	200	---
Naphthalene	10	20	50	80	120	200	---
4-Chloroaniline	10	20	50	80	120	200	---
Hexachlorobutadiene	20	20	50	80	120	200	---
4-Chloro-3-methylphenol	10	20	50	80	120	200	---
2-Methylnaphthalene	10	20	50	80	120	200	---
Hexachlorocyclopentadiene	20	50	80	120	200	---	160
2,4,6-Trichlorophenol	10	20	50	80	120	200	---
2,4,5-Trichlorophenol	10	20	50	80	120	200	---
2-Chloronaphthalene	10	20	50	80	120	200	---
2-Nitroaniline	20	50	80	120	200	---	160
Dimethyl phthalate	10	20	50	80	120	200	---
Acenaphthylene	10	20	50	80	120	200	---
3-Nitroaniline	20	50	80	120	200	---	160
Acenaphthene	10	20	50	80	120	200	---
2,4-Dinitrophenol	20	50	80	120	200	---	160
4-Nitrophenol	20	50	80	120	200	---	160
Dibenzofuran	10	20	50	80	120	200	---
2,4-Dinitrotoluene	10	20	50	80	120	200	---
2,6-Dinitrotoluene	10	20	50	80	120	200	---
Diethylphthalate	10	20	50	80	120	200	---

Table 10.

Calibration Levels for AFCEE Projects, µg/mL (cont.)

Analyte	Std Conc 1	Std Conc 2	Std Conc 3	Std Conc 4	Std Conc 5	Std Conc 6	Addnl Conc for 2nd Order ICALs
4-Chlorophenyl phenyl ether	10	20	50	80	120	200	---
Fluorene	10	20	50	80	200	200	---
4-Nitroaniline	20	50	80	120	200	200	---
4,6-Dinitro-2-methylphenol	20	50	80	120	200	200	---
N-Nitrosodiphenylamine	10	20	50	80	200	200	---
Azobenzene[2]	10	20	50	80	200	200	---
4-Bromophenyl phenyl ether	10	20	50	80	200	200	---
Hexachlorobenzene	10	20	50	80	200	200	---
Pentachlorophenol	20	50	80	120	200	200	---
Phenanthrene	10	20	50	80	200	200	---
Anthracene	10	20	50	80	200	200	---
Carbazole	10	20	50	80	200	200	---
Di-n-butyl phthalate	10	20	50	80	200	200	---
Fluoranthene	10	20	50	80	200	200	---
Benzidine	50	50	80	120	200	200	---
Pyrene	10	20	50	80	200	200	---
Butyl benzyl phthalate	10	20	50	80	200	200	---
3,3'-Dichlorobenzidine	10	50	80	120	200	200	---
Benzo(a)anthracene	10	20	50	80	200	200	---
Bis(2-ethylhexyl)phthalate	10	20	50	80	200	200	---
Chrysene	10	20	50	80	200	200	---
Di-n-octylphthalate	10	20	50	80	200	200	---
Benzo(b)fluoranthene	10	20	50	80	200	200	---
Benzo(k)fluoranthene	10	20	50	80	200	200	---
Benzo(a)pyrene	10	20	50	80	200	200	---
Indeno(1,2,3-cd)pyrene	10	20	50	80	200	200	---
Dibenz(a,h)anthracene	10	20	50	80	200	200	---
Diethyl phthalate	10	20	50	80	200	200	---
Benzo(g,h,i)perylene	10	20	50	80	200	200	---

1. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether.
2. Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 11.

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Pyridine	--	20	50	80	120	160	200
N-nitrosodimethylamine	10	20	50	80	120	160	200
Aniline	10	20	50	80	120	160	200
Phenol	10	20	50	80	120	160	200
Bis(2-chloroethyl)ether	10	20	50	80	120	160	200
2-Chlorophenol	10	20	50	80	120	160	200
1,3-Dichlorobenzene	10	20	50	80	120	160	200
1,4-Dichlorobenzene	10	20	50	80	120	160	200
Benzyl alcohol	10	20	50	80	120	160	200
1,2-Dichlorobenzene	10	20	50	80	120	160	200
2-Methylphenol	10	20	50	80	120	160	200
2,2'-oxybis(1-chloropropane)[1]	10	20	50	80	120	160	200
4-Methylphenol	10	20	50	80	120	160	200
N-Nitroso-di-n-propylamine	10	20	50	80	120	160	200
Hexachloroethane	10	20	50	80	120	160	200
Nitrobenzene	10	20	50	80	120	160	200
Isophorone	10	20	50	80	120	160	200
2-Nitrophenol	10	20	50	80	120	160	200
2,4-Dimethylphenol	10	20	50	80	120	160	200
Benzoic acid	--	20	50	80	120	160	200
Bis(2-chloroethoxy)methane	10	20	50	80	120	160	200
2,4-Dichlorophenol	10	20	50	80	120	160	200
1,2,4-Trichlorobenzene	10	20	50	80	120	160	200
Naphthalene	10	20	50	80	120	160	200
4-Chloroaniline	10	20	50	80	120	160	200
Hexachlorobutadiene	10	20	50	80	120	160	200
4-Chloro-3-methylphenol	10	20	50	80	120	160	200
2-Methylnaphthalene	10	20	50	80	120	160	200
Hexachlorocyclopentadiene	--	20	50	80	120	160	200
2,4,6-Trichlorophenol	10	20	50	80	120	160	200
2,4,5-Trichlorophenol	10	20	50	80	120	160	200
2-Chloronaphthalene	10	20	50	80	120	160	200
2-Nitroaniline	--	20	50	80	120	160	200
Dimethyl phthalate	10	20	50	80	120	160	200
Acenaphthylene	10	20	50	80	120	160	200
3-Nitroaniline	--	20	50	80	120	160	200
Acenaphthene	10	20	50	80	120	160	200
2,4-Dinitrophenol	--	20	50	80	120	160	200
4-Nitrophenol	--	20	50	80	120	160	200
Dibenzofuran	10	20	50	80	120	160	200
2,4-Dinitrotoluene	10	20	50	80	120	160	200
2,6-Dinitrotoluene	10	20	50	80	120	160	200
Diethylphthalate	10	20	50	80	120	160	200
4-Chlorophenyl phenyl ether	10	20	50	80	120	160	200

Table 11.

Calibration Levels, Primary Standard, µg/mL (cont.)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Fluorene	10	20	50	80	120	160	200
4-Nitroaniline	--	20	50	80	120	160	200
4,6-Dinitro-2-methylphenol	--	20	50	80	120	160	200
N-Nitrosodiphenylamine	10	20	50	80	120	160	200
Azobenzene[2]	10	20	50	80	120	160	200
4-Bromophenyl phenyl ether	10	20	50	80	120	160	200
Hexachlorobenzene	10	20	50	80	120	160	200
Pentachlorophenol	--	20	50	80	120	160	200
Phenanthrene	10	20	50	80	120	160	200
Anthracene	10	20	50	80	120	160	200
Carbazole	10	20	50	80	120	160	200
Di-n-butyl phthalate	10	20	50	80	120	160	200
Fluoranthene	10	20	50	80	120	160	200
Benzidine	--	20	50	80	120	160	200
Pyrene	10	20	50	80	120	160	200
Butyl benzyl phthalate	10	20	50	80	120	160	200
3,3'-Dichlorobenzidine	--	20	50	80	120	160	200
Benzo(a)anthracene	10	20	50	80	120	160	200
Bis(2-ethylhexyl)phthalate	10	20	50	80	120	160	200
4,4-Methylenebis(2-chloroaniline)	10	20	50	80	120	160	200
Chrysene	10	20	50	80	120	160	200
Di-n-octylphthalate	10	20	50	80	120	160	200
Benzo(b)fluoranthene	10	20	50	80	120	160	200
Benzo(k)fluoranthene	10	20	50	80	120	160	200
Benzo(a)pyrene	10	20	50	80	120	160	200
Indeno(1,2,3-cd)pyrene	10	20	50	80	120	160	200
Dibenz(a,h)anthracene	10	20	50	80	120	160	200
Diethyl phthalate	10	20	50	80	120	160	200
Benzo(g,h,i)perylene	10	20	50	80	120	160	200

1. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether
2. Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 12.
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2-Picoline	10	20	50	80	120	160	200
N-Nitrosomethylethylamine	10	20	50	80	120	160	200
Methyl methanesulfonate	10	20	50	80	120	160	200
N-Nitrosodiethylamine	10	20	50	80	120	160	200
Ethyl methanesulfonate	10	20	50	80	120	160	200
Pentachloroethane	--	20	50	80	120	160	200
Acetophenone	10	20	50	80	120	160	200
N-Nitrosopyrrolidine	10	20	50	80	120	160	200
N-Nitrosomorpholine	10	20	50	80	120	160	200
o-Toluidine	10	20	50	80	120	160	200
3-Methylphenol	10	20	50	80	120	160	200
N-Nitrosopiperidine	10	20	50	80	120	160	200
o,o,o-Triethyl-Phosphorothioate	--	20	50	80	120	160	200
a,a-Dimethyl-phenethylamine	--	20	50	80	120	160	200
2,6-Dichlorophenol	10	20	50	80	120	160	200
Hexachloropropene	--	20	50	80	120	160	200
p-Phenylenediamine	--	20	50	80	120	160	200
n-Nitrosodi-n-butylamine	10	20	50	80	120	160	200
Safrole	--	20	50	80	120	160	200
1,2,4,5-Tetrachlorobenzene	10	20	50	80	120	160	200
Isosafrole 1 + 2	10	20	50	80	120	160	200
1,4-Dinitrobenzene	10	20	50	80	120	160	200
1,4-Naphthoquinone	--	20	50	80	120	160	200
1,3-Dinitrobenzene	10	20	50	80	120	160	200
Pentachlorobenzene	10	20	50	80	120	160	200
1-Naphthylamine	10	20	50	80	120	160	200
2-Naphthylamine	10	20	50	80	120	160	200
2,3,4,6-Tetrachlorophenol	--	20	50	80	120	160	200
5-Nitro-o-toluidine	10	20	50	80	120	160	200
Thionazin	10	20	50	80	120	160	200
1,3,5-Trinitrobenzene	--	20	50	80	120	160	200
Sulfotepp	--	20	50	80	120	160	200
Phorate	--	20	50	80	120	160	200
Phenacetin	10	20	50	80	120	160	200
Diallate 1 + 2	10	20	50	80	120	160	200
Dimethoate	10	20	50	80	120	160	200
4-Aminobiphenyl	--	20	50	80	120	160	200
Pentachloronitrobenzene	--	20	50	80	120	160	200
Pronamide	10	20	50	80	120	160	200
Disulfoton	--	20	50	80	120	160	200
2-secbutyl-4,6-dinitrophenol (Dinoseb)	10	20	50	80	120	160	200
Methyl parathion	--	20	50	80	120	160	200
4-Nitroquinoline-1-oxide	--	20	50	80	120	160	200
Parathion	--	20	50	80	120	160	200

Table 12.

Calibration Levels, Appendix IX Standard, µg/mL (cont.)

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Isodrin	10	20	50	80	120	160	200
Methapyrilene	--	20	50	80	120	160	200
Aramite 1 and 2	10	20	50	80	120	160	200
p-(Dimethylamino) azobenzene	10	20	50	80	120	160	200
p-Chlorobenzilate	10	20	50	80	120	160	200
3,3'-Dimethylbenzidine	10	20	50	80	120	160	200
2-Acetylaminofluorene	--	20	50	80	120	160	200
Dibenz (a,j)acridine	10	20	50	80	120	160	200
7,12-Dimethylbenz(a) anthracene	10	20	50	80	120	160	200
3-Methylcholanthrene	10	20	50	80	120	160	200

Table 13.

Initial Demonstration Recovery and Precision Limits

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7

Table 13.

Initial Demonstration Recovery and Precision Limits (cont.)

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

1. Organochlorine pesticides and PCBs project DQOs generally require better sensitivity than is provided by 8270C, so methods 8081 and 8082 are used instead. These compounds will not be included in the initial demonstration of capability for method 8270C.

Table 14.

List 1 Reliably Performing Compounds

Acenaphthene	Dibenzofuran	1H-Indene
Acenaphthylene	1,4-Dioxane	Indeno(1,2,3-cd)pyrene
Acetophenone	n-Dodecane	Isophorone
Alachlor	n-Docosane	1-Methylnaphthalene
Aniline	1,2-Dichlorobenzene	2-Methylnaphthalene
Anthracene	1,3-Dichlorobenzene	2-Methylphenol
Atrazine	1,4-Dichlorobenzene	4-Methylphenol
Benzo(a)anthracene	2,3-Dichlorobenzeneamine	Methylstyrene
Benzo(a)pyrene	3,3'-Dichlorobenzidine	Naphthalene
Benzo(b)fluoranthene	2,4-Dichlorophenol	2-Nitroaniline
Benzo(k)fluoranthene	Diethyl phthalate	3-Nitroaniline
Benzo(g,h,i)perylene	2,4-Dimethylphenol	4-Nitroaniline
Benzoic acid	Dimethyl phthalate	Nitrobenzene
Benzyl alcohol	Di-n-butyl phthalate	2-Nitrophenol
Bis(2-chloroethoxy)methane	4,6-Dinitro-2-methylphenol	4-Nitrophenol
Bis(2-chloroethyl)ether	2,4-Dinitrophenol	N-Nitrosodimethylamine
Bis(2-ethylhexyl)phthalate	2,4-Dinitrotoluene	N-Nitroso-di-n-propylamine
4-Bromophenyl phenyl ether	2,6-Dinitrotoluene	N-Nitrosodiphenylamine
Butyl benzyl phthalate	1,2-Diphenylhydrazine (as Azobenzene)	2,2'-Oxybis(1-chloropropane) aka "bis(2-chloroisopropyl) ether"
Caprolactam	Di-n-octyl phthalate	n-Octadecane
Carbazole	n-Eicosane	Pentachlorophenol
4-Chloroaniline	Famphur	Phenanthrene
4-Chloro-3-methylphenol	Fluoranthene	Phenol
2-Chloronaphthalene	Fluorene	Pyrene
2-Chlorophenol	Hexachlorobenzene	Pyridine
4-Chlorophenyl phenyl ether	Hexachlorocyclopentadiene	n-Tetradecane
Chrysene	Hexachlorobutadiene	1,2,4-Trichlorobenzene
n-Decane	Hexachloroethane	2,4,5-Trichlorophenol
Dibenz(a,h)anthracene	n-Hexadecane	2,4,6-Trichlorophenol

Table 15.

List 2 Poorly Performing Compounds

2-Acetylaminofluorene	Diphenylamine	N-Nitrosopyrrolidine
Acrylamide	Disulfoton	Parathion
4-Aminobiphenyl	2-Ethoxyethanol	Pentachlorobenzene
Aramite (#1)	Ethyl methanesulfonate	Pentachloroethane
Aramite (#2)	Hexachlorophene	Pentachloronitrobenzene
Benzenethiol	Hexachloropropene	Perylene
Benzidine	Isosafrole (#1)	Phenacetin
Benzyl chloride	Isosafrole (#2)	p-Phenylenediamine
Biphenyl	Isodrin	Phorate
Carbofuran phenol	Methapyrilene	Phthalic anhydride
Chlorobenzilate	Methomyl	2-Picoline
Diallate (#1)	3-Methylcholanthrene	Pronamide
Diallate (#2)	6-Methylchrysene	Quinoline
Dibenz(a,h)acridine	4,4"-Methylenebis(2-chloroaniline)	Safrole
Dibenz(a,j)acridine	Methyl methanesulfonate	2-secbutyl-4,6-dinitrophenol (Dinoseb)
Dibenzo(a,e)pyrene	Methyl Parathion	Sulfotepp
Tris(2,3-Dibromopropyl) phosphate	1-Naphthylamine	1,2,4,5-Tetrachlorobenzene
2,6-Dichlorophenol	2-Naphthylamine	2,3,4,6-Tetrachlorophenol
Dimethoate	1,4-Naphthoquinone	Thionazin
p-(Dimethylamino)azobenzene	5-Nitro-o-toluidine	o-Toluidine
7,12-Dimethylbenz(a)anthracene	4-Nitroquinoline-1-oxide	2,4- and 2,6-Toluenediamine
3,3'-Dimethylbenzidine	N-Nitrosodiethylamine	Triethylamine
N,N-Dimtheylformamide	n-Nitrosodi-n-butylamine	Triethylphosphate
a,a-Dimethyl-phenethylamine	N-Nitrosomethylethylamine	o,o,o-Triethylphosphorothioate
1,3-Dinitrobenzene	N-Nitrosomorpholine	1,3,5-Trinitrobenzene
1,4-Dinitrobenzene	N-Nitrosopiperidine	

APPENDIX A

Modifications Required for Analysis of Wastewater Following Method 625

REQUIREMENTS FOR METHOD 625

- Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits or other CWA compliance situations. The standard analyte list and reporting limits are listed in Table A-1.
- This method can be applied to only aqueous matrices.
- The tune period for this method is defined as 24 hours.
- Initial calibration curve requirements are as follows:
  - The initial calibration curve for this method requires at least three points.
  - Target compounds must have RSD ≤ 35%.
  - If this requirement cannot be met, a regression curve must be constructed for the non-compliant compounds.
- Continuing calibration verification requirements are as follows:
  - All target compounds must have %D ≤ 20%.
- Matrix Spike and LCS requirements are as follows:
- A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

Table A-1.

TAL Method 625 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	20
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50

Table A-1.

TAL Method 625 Standard Reporting List and Reporting Limits (cont.)

Analytes	CAS Number	Aqueous, µg/L
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10
N-Nitrosodimethylamine	62-75-9	10

Table A-2.

Method 625 LCS and MS Compounds and Spike Concentrations

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	100
Bis(2-chloroethyl)ether	100
2-Chlorophenol	100
1,3-Dichlorobenzene	100
1,4-Dichlorobenzene	100
1,2-Dichlorobenzene	100
2,2'-oxybis(1-chloropropane)	100
N-Nitroso-di-n-propylamine	100
Hexachloroethane	100
Nitrobenzene	100
Isophorone	100
2-Nitrophenol	100
2,4-Dimethylphenol	100
Bis(2-chloroethoxy)methane	100
2,4-Dichlorophenol	100
1,2,4-Trichlorobenzene	100
Naphthalene	100
Hexachlorobutadiene	100
4-Chloro-3-methylphenol	100
Hexachlorocyclopentadiene	100
2,4,6-Trichlorophenol	100
2-Chloronaphthalene	100
Dimethyl phthalate	100
Acenaphthylene	100
Acenaphthene	100
2,4-Dinitrophenol	100
4-Nitrophenol	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Diethylphthalate	100
4-Chlorophenyl phenyl ether	100
Fluorene	100
4,6-Dinitro-2-methylphenol	100
N-Nitrosodiphenylamine	100
4-Bromophenyl phenyl ether	100
Hexachlorobenzene	100
Pentachlorophenol	100

Table A-2.

Method 625 LCS and MS Compounds and Spike Concentrations (cont.)

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenanthrene	100
Anthracene	100
Di-n-butyl phthalate	100
Fluoranthene	100
Benzidine	100
Pyrene	100
Butyl benzyl phthalate	100
3,3'-Dichlorobenzidine	100
Benzo(a)anthracene	100
Bis(2-ethylhexyl)phthalate	100
Chrysene	100
Di-n-octylphthalate	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Indeno(1,2,3-cd)pyrene	100
Dibenz(a,h)anthracene	100
Benzo(g,h,i)perylene	100
N-Nitrosodimethylamine	100

APPENDIX B

Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (8270BP)

REQUIREMENTS FOR METHOD 8270 BEST PRACTICE (8270BP)

- Method Best Practice is utilized to obtain lower reporting limits while still providing full scan data.. The standard analyte list and reporting limits are listed in Table B-1.
- This method can be applied to only aqueous matrices.
- The extraction is the same with one exception. The final volume of the extract is 2 mL.
- The tune period for this method is defined as 12 hours.
- Initial calibration curve requirements are as follows:
  - Same as for 8270 detailed in Section 11.4 of this SOP.
  - The calibrations levels are shown in Table B-2.
- Continuing calibration verification requirements are as follows:
  - Same as for 8270 detailed in Section 11.5 of this SOP, except the level 7 calibration point is used.
- Matrix Spike and LCS requirements are as follows:
  - The spike levels are listed in Table B-3.
- Internal Standards: The internal standard concentrations are listed in Table B-5.
- Surrogates: The surrogate concentrations are listed in Table B-4.
- Instrument Conditions are shown in Table B-6.

Table B-1.

TAL Method 8270BP Standard Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Pyridine	110-86-1	20
N-nitrosodimethylamine	62-75-9	5
Aniline	62-53-3	5
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	1
2-Chlorophenol	95-57-8	5
Benzyl alcohol	100-51-6	5
2-Methylphenol	95-48-7	5
2,2'-oxybis(1-chloropropane)[2]	108-60-1	5
4-Methylphenol	106-44-5	5
N-Nitroso-di-n-propylamine	621-64-7	5
Hexachloroethane	67-72-1	5
Nitrobenzene	98-95-3	5
Isophorone	78-59-1	5
2-Nitrophenol	88-75-5	5
Benzoic acid	65-85-0	10
Bis(2-chloroethoxy)methane	111-91-1	5
2,4-Dichlorophenol	120-83-2	5
1,2,4-Trichlorobenzene	120-82-1	5
Naphthalene	91-20-3	5
4-Chloroaniline	106-47-8	5
Hexachlorobutadiene	87-68-3	5
4-Chloro-3-methylphenol	59-50-7	5
2-Methylnaphthalene	91-57-6	5
Hexachlorocyclopentadiene	77-47-4	5
2,4,6-Trichlorophenol	88-06-2	5
2,4,5-Trichlorophenol	95-95-4	5
2-Chloronaphthalene	91-58-7	5
2-Nitroaniline	88-74-4	5
Dimethyl phthalate	131-11-3	5
Acenaphthylene	208-96-8	5
3-Nitroaniline	99-09-2	5
Acenaphthene	83-32-9	5
2,4-Dinitrophenol	51-28-5	5
4-Nitrophenol	100-02-7	5
Dibenzofuran	132-64-9	5
2,4-Dinitrotoluene	121-14-2	5
2,6-Dinitrotoluene	606-20-2	5
4-Chlorophenyl phenyl ether	7005-72-3	5
Fluorene	86-73-7	5
4-Nitroaniline	100-01-6	5
4,6-Dinitro-2-methylphenol	534-52-1	10
N-Nitrosodiphenylamine	86-30-6	5
Azobenzene	103-33-3	5
4-Bromophenyl phenyl ether	101-55-3	5

Table B-1.

TAL Method 8270BP Standard Reporting Limits (cont.)

Analytes	CAS Number	Aqueous, µg/L
Hexachlorobenzene	118-74-1	1
Pentachlorophenol	87-86-5	10
Phenanthrene	85-01-8	1
Anthracene	120-12-7	5
Carbazole	86-74-8	5
Di-n-butyl phthalate	84-74-2	5
Fluoranthene	206-44-0	1
Benzidine	92-87-5	1
Pyrene	129-00-0	5
Butyl benzyl phthalate	85-68-7	5
3,3'-Dichlorobenzidine	91-94-1	5
Benzo(a)anthracene	56-55-3	1
Bis(2-ethylhexyl)phthalate	117-81-7	5
Chrysene	218-01-9	1
Di-n-octylphthalate	117-84-0	5
Benzo(b)fluoranthene	205-99-2	5
Benzo(k)fluoranthene	207-08-9	5
Benzo(a)pyrene	50-32-8	5
Indeno(1,2,3-cd)pyrene	193-39-5	5
Diethyl phthalate	84-66-2	5
Dibenz(a,h)anthracene	53-70-3	5
Benzo(g,h,i)perylene	191-24-2	5
1,4-Dioxane	123-91-2	1

Table B-2.

Method 8270BP Calibration Levels

Calibration Level	Calibration Concentration, µg/mL
1	0.25
2	0.40
3	1.00
4	2.50
5	5.00
6	7.50
7	10.
8	12.5
9	20.0
10	40.0
SSV	5.0

Table B-3.

Method 8270BP LCS Spike Concentrations

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	10
Bis(2-chloroethyl)ether	10
2-Chlorophenol	10
1,3-Dichlorobenzene	10
1,4-Dichlorobenzene	10
1,2-Dichlorobenzene	10
2,2'-oxybis(1-chloropropane)	10
N-Nitroso-di-n-propylamine	10
Hexachloroethane	10
Nitrobenzene	10
Isophorone	10
2-Nitrophenol	10
2,4-Dimethylphenol	10
Bis(2-chloroethoxy)methane	10
2,4-Dichlorophenol	10
1,2,4-Trichlorobenzene	10
Naphthalene	10
Hexachlorobutadiene	10
4-Chloro-3-methylphenol	10
Hexachlorocyclopentadiene	10
2,4,6-Trichlorophenol	10
2-Chloronaphthalene	10
Dimethyl phthalate	10
Acenaphthylene	10
Acenaphthene	10
2,4-Dinitrophenol	10
4-Nitrophenol	10
2,4-Dinitrotoluene	10
2,6-Dinitrotoluene	10
Diethylphthalate	10
4-Chlorophenyl phenyl ether	10
Fluorene	10
4,6-Dinitro-2-methylphenol	10
N-Nitrosodiphenylamine	10
4-Bromophenyl phenyl ether	10
Hexachlorobenzene	10
Pentachlorophenol	10
Phenanthrene	10
Anthracene	10
Di-n-butyl phthalate	10
Fluoranthene	10
Benzidine	10
Pyrene	10
Butyl benzyl phthalate	10
3,3'-Dichlorobenzidine	10
Benzo(a)anthracene	10

Table B-3.

Method 8270BP LCS Spike Concentrations (cont.)

LCS Compounds	Spiking Level, ng/µL in extract[1]
Bis(2-ethylhexyl)phthalate	10
Chrysene	10
Di-n-octylphthalate	10
Benzo(b)fluoranthene	10
Benzo(k)fluoranthene	10
Benzo(a)pyrene	10
Indeno(1,2,3-cd)pyrene	10
Dibenz(a,h)anthracene	10
Benzo(g,h,i)perylene	10
N-Nitrosodimethylamine	10
1,4-Dioxane	10

Table B-4.

8270BP Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d5	5
2-Fluorobiphenyl	5
Terphenyl-d14	5
1,2-Dichlorobenzene-d4[1]	5
Phenol-d5	7.5
2-Fluorophenol	7.5
2,4,6-Tribromophenol	7.5
2-Chlorophenol-d4[1]	7.5

Table B-5.

8270BP Internal Standard Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d5	5
2-Fluorobiphenyl	5
Terphenyl-d14	5
1,2-Dichlorobenzene-d4[1]	5
Phenol-d5	7.5
2-Fluorophenol	7.5
2,4,6-Tribromophenol	7.5
2-Chlorophenol-d4[1]	7.5

Table B-6.

Suggested Instrument Conditions for 8270BP

Mass Range:	35 - 500 amu
Scan Time:	≤1 second/scan
Initial Column Temperature/Hold Time:	50 °C for 1 minutes
Column Temperature Program:	50 - 320 °C at 35°C/min.
Final Column Temperature/Hold Time:	325 °C/4 min hold
Injector Temperature:	275 °C
Transfer Line Temperature:	290 °C
Source Temperature:	230 °C
Injector:	Single Taper Direct Connect Liner /splitless
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 1.0mL/min.
Column:	DB-5 Capillary 20m x 0.18mm x 0.36 um film thickness

APPENDIX C

Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

MASS SPECTROMETER Instrument Maintenance Schedule				
Daily	Weekly	As Needed	Quarterly	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure	**Check mass calibration (PFTBA or FC-43).**	**Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.**	**Check vacuum, relays, gas pressures, and flows.**	**Replace the exhaust filters on the mechanical rough pump every 1 to 2 years.**
Check temperatures of injector, detector. Verify temperature programs.		**Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.**		**Change the oil in the mechanical rough pump.**
Check inlets, septa.		**Clean source, including all ceramics and lenses. Source cleaning is indicated by a variety of symptoms, including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.**		**Relubricate the turbomolecular pump-bearing wick.**
Check baseline level.		**Repair/replace jet separator.**		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		**Replace filaments when both filaments burn out or performance indicates the need for replacement.**		

APPENDIX C

Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph (cont.)

GAS CHROMATOGRAPH Instrument Maintenance Schedule (For GC/MS only.)	
Daily	*As Needed*
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	***Replace front portion of column packing or guard column or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance indicates it is required (e.g., peak tailing, poor resolution, high backgrounds, etc.).***
Check temperatures of injectors and detectors. Verify temperature programs.	***Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.***
Check inlets, septa. ***Clean injector port.***	***Replace septa.***
Check baseline level.	***Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).***
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	***Repair or replace flow controller if constant gas flow cannot be maintained.***
	Reactivate flow controller filter dryers when the presence of moisture is suspected.
	Autosampler: Replace syringe, fill wash bottle, dispose of waste bottle contents.

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Title: GC/MS Analysis Based On Methods 8270D

Approvals (Signature/Date):

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Technical Specialist — Date

Adam Alban 1/1/13
Health & Safety Manager / Coordinator — Date

John P. Morris 1/3/13
Quality Assurance Manager — Date

William S. Cicero 1/3/13
Laboratory Director — Date

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This method is based upon standard method SW846 8270D, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices.

1.1.1 The modifications presented in Appendix A may be followed for analysis of samples following method 8270 (best practices).

NOTE: The 8270 Best Practice method is NOT applicable for the analysis of South Carolina regulatory compliance samples.

1.1.2 Direct injection of a sample may be used in limited applications.

1.1.3 Refer to Tables 1 and 2 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. This method may be amenable to additional compounds. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as 3/4-methylphenol.
- Hexachlorophene and famphur analysis are not quantitatively reliable by this method.
- Kepone should be analyzed by GC/ECD.
- Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

1.3 The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

2.0 Summary of Method

2.1 Aqueous samples are extracted with methylene chloride using a continuous extractor or a separatory funnel.

2.2 Solid samples are extracted with methylene chloride / acetone using sonication. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS.

2.3 Waste dilution is used for samples that are miscible with the solvent.

2.4 Extraction procedures are detailed in the following SOPs:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series
DV-OP-0007	Concentration of Organic Extracts, SW846 3510C, 3520C, 3540C, 3550B, 3550C, 3660B, 3665A and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0016	Ultrasonic Extraction of Solid Samples, SW846 3550C

2.5 Qualitative identification of the analytes in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3.0 Definitions

3.1 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / matrix spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.

3.2 Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TAL QC Program document (DV-QA-003P) for further details of the batch definition.

3.3 Method Blank (MB) - An analytical control consisting of all reagents, internal standards and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.4 Laboratory Control Sample (LCS) - A blank matrix (reagent water or Ottawa Sand) spiked with the analytes of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked analytes demonstrates that the laboratory techniques for this method are acceptable.

3.5 Matrix Spike (MS) – An aliquot of a matrix (water or soil) fortified (spiked) with known amounts of specific analytes and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.6 Matrix Spike Duplicate (MSD) - A second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method by measuring the relative percent difference (RPD) between the MS and MSD results.

3.7 Surrogates - Organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, MS, and MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

4.0 Interferences

4.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample. Cleanup procedures may help to eliminate select interferences, as follows:

- Method 3640A, Gel-Permeation Chromatography (GPC) - Removes higher molecular weight hydrocarbons by size exclusion chromatography, which is most frequently used for biological samples (TestAmerica Denver does not have a GPC unit).

- Method 3660B, Sulfur Cleanup – If a sulfur peak is detected, copper or mercury can be used to treat the extract and remove the sulfur

- Other, more aggressive cleanup procedures listed in SW-846 may be used for select compounds listed in this procedure, but may cause degradation of some of the more reactive compounds. Consult with a technical expert in the laboratory for more difficult interference problems.

Details concerning cleanup steps are described in the organic extraction SOP DV-OP-0007.

4.2 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section (Section 9.3). Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.3 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

NOTE: Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.

5.1.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power before performing any maintenance.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Significant or Serious Hazard Rating

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system: an analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Column: 30 m x 0.25 mm I.D., 0.5-µm film thickness fused-silica capillary column coated with 5% diphenyl/95% dimethyl polysiloxane(Restek Rtx®-5MS or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 u (previously “amu”) every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) that meets all of the criteria in Table 4 when 25 ng of the GC/MS tuning standard is injected through the GC.

6.4 Autosampler: LEAP Technologies CTC A200S, HP7683 Autosampler or equivalent.

6.5 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.6 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or

scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.7 Syringe: 10 µL or 5µL Hamilton Laboratory grade syringes or equivalent. The 5 µL syringe is used for the Agilent ALS to be able to inject 0.5 µL.

6.8 Carrier gas: Ultra high-purity helium.

6.9 Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\|Master List of Documents, Software and Hardware.xls for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 A minimum five-point calibration curve is prepared when average response factors or linear regression curve fitting is used. Six calibration points are required for second-order curve fits. The low point should be at or below the reporting limit. Refer to tables 11 and 12 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2 An internal standard (IS) solution is prepared. Compounds in the IS Mix are acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.2.1 Internal standards are added to all standards and extracts to result in a final concentration of 40 µg/mL. For example, if the volume of an extract aliquot used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added to the aliquot. See Appendix B for the levels used for the 8270 best practice method.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). Surrogate compounds and levels are listed in Table 9.

Acid Surrogates	Base Surrogates
2-Fluorophenol	2-Fluorobiphenyl
2,4,6-tribromophenol	Terphenyl-d_4
Phenol-d_5	Nitrobenzene-d_5
2-chlorophenol-d_4	1,2,-Dichlorobenzene-d_4

7.4 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT should also be included in the Tuning Standard at 50 µg/mL.

7.5 Laboratory Control Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). LCS compounds and levels are listed in Table 7.

7.6 Matrix Spike Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). The matrix spike compounds and levels are the same as the LCS compounds.

7.7 The standards are stored away from any light source at 6 °C (-10 °C recommended). The

standard stock solutions expire after one year from preparation date or at the earliest expiration date assigned by the vendor to any parent standard, whichever is earlier. The continuing calibration standard should be replaced every week, when there are visible signs of degradation, or when the standard fails to meet QC criteria.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	1 liter amber	1 Liter	Cool 4 ± 2°C	7 Days	40 Days from extraction	40 CFR Part 136.3 and SW846 Chapter 4
Soils	4oz Jar	30 grams	Cool 4 ± 2°C	14 Days	40 Days from extraction	SW846 Chapter 4

9.0 Quality Control

9.1 Initial Performance Studies

9.1.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL). See Section 13 for a discussion of detection limit studies.

9.1.2 In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument they will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details.

9.2 Control Limits

9.2.1 In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits are determined every 6 months. The recovery limits are the mean recovery ±3 standard deviations for surrogates, MS, and LCS. Precision limits for the MS/MSD pair results is the absolute value of the mean relative percent difference (RPD) +3 standard deviations.

9.2.2 These limits do not apply to dilutions, but surrogate and matrix spike recoveries will be reported unless the dilution is 4x or more.

9.2.3 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into the LIMS or other database so that accurate historical control limits can be generated. For multiple dilutions reported from the same extract, surrogates will be reported for all dilutions of less than 4x. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.4 Refer to the QC program document, DV-QA-003P, for further details of control limits.

9.3 Method Blank (MB)

For aqueous sample batches, the method blank is reagent water; for solid sample batches, the method blank is clean sand. In either case, the method blank is free of the analytes of interest and is spiked with the surrogates. At least one method blank must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than ½ of the reporting limit or less than 10% of the analyte concentration found in the associated samples, whichever is higher. When a compound is above ½ the reporting limit a NCM needs to be completed.

NOTE: All programs require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: Re-preparation and reanalysis of all samples associated with an unacceptable method blank. If the analyte was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.4 Instrument Blank

Instruments must be evaluated for contamination during each 12-hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.5 Laboratory Control Sample (LCS)

The LCS is prepared using reagent water for aqueous methods and Ottawa sand for solid sample methods. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. The LCS is spiked with the compounds listed in Tables 7 and 8 unless specified by a client or agency. The compounds must be spiked at a concentration equivalent to 80 or 120 µg/L, depending on the analyte, unless a special QAS states a specific level. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Acceptance Criteria: All analytes must be within established control limits. See QC Policy DV-QA-003P for details on establishing control limits.

Corrective Action: If any analyte in the LCS is outside the laboratory-established historical control limits or project-specific control limits, as applicable, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the

problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.

- If re-extraction and reanalysis of the batch are not possible due to limited sample volume or other constraints, the failed LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The matrix spike is a second aliquot of one of the samples in the batch. The matrix spike duplicate is a third aliquot of the same sample. The MS and MSD are spiked with the same analytes as the LCS (See Tables 7 and 8). An MS/MSD pair is prepared and analyzed with every batch of samples.

Acceptance Criteria: The percent recovery (%R) must fall within either historical limits or project-specific limits, as applicable. The relative percent difference (RPD) between the MS and MSD results must be less than or equal to the established historical or project-specific limit. See QC Policy DV-QA-003P for details on establishing control limits

Corrective Action: If any individual recovery or RPD fails the acceptance criteria, then corrective action must occur. Initially check the recovery of the analyte in question in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is considered to be in control and analysis may proceed unless project specific requirements indicate alternative corrective actions. The reasons for accepting the batch must be documented. The sample results must be flagged and the nonconformance described in the case narrative.

NOTE: South Carolina requires reanalysis to confirm matrix interference.

- If the recovery for any analyte fails acceptance criteria for the MS, MSD, and the LCS, the laboratory operation is considered to be out of out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.
- The MS/MSD pair must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted to concentrations below the calibration range.

9.7 Surrogates

9.7.1 Each sample, blank, and QC sample is spiked with the surrogate standards. Surrogate compounds must be spiked at either 100 or 150 ug/L, depending on the surrogate. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 9. For the Best Practice method, see table B-4 in Appendix B.

Acceptance Criteria: Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

Corrective Action: If any surrogates are outside of the limits, then the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

NOTE: The decision to reanalyze or flag the data for failed QC should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.7.2 If the sample with failed surrogate recoveries was a sample used for an MS/MSD pair and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample and the MS and the MSD do not require reanalysis. This phenomenon indicates a possible matrix problem.

NOTE: In these circumstances, South Carolina requires re-extraction and reanalysis of the sample and MS/MSD to confirm matrix interference.

9.7.3 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (If the re-analysis was outside holding times, both sets of results may be reported, with appropriate flags and discussion in the case narrative. Consult client and/or program specifications for reporting requirements.)

9.7.4 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effects.

9.8 Nonconformance and Corrective Action

9.8.1 Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry of the sample, sample size, or other parameters. Any variation in procedure shall be completely documented

using a Nonconformance Memo (NCM). The NCM is a then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The nonconformance shall be addressed in the case narrative, and the NCM shall be filed in the project file. The NCM process is described in more detail in SOP DV-QA-0031.

9.8.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents and approved by a supervisor and QA Manager. Unless the client requests more stringent criteria than the requirements in this SOP, the deviations must be clearly documented in the report narrative and the samples flagged accordingly.

9.8.3 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.9 Quality Assurance Summaries (QAS) or Program Distillations

Certain clients may require specific project or program QC that may supersede the requirements presented in this section. Quality Assurance Summaries (also known as Program Distillations) should be developed to address these requirements.

9.10 TestAmerica QC Program

Details of the Denver Quality Control Program, including corrective action guidelines, are presented in SOP DV-QA-003P, *Quality Control Program*. Refer to this document if in doubt regarding corrective actions.

10.0 Procedure

10.1 Sample Preparation

Samples are prepared according to the following organic preparation SOPs, as applicable:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series
DV-OP-0007	Concentration of Organic Extracts, SW846 3510C, 3520C, 3540C, 3550B, 3550C, 3660B, 3665A and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0016	Ultrasonic Extraction of Solid Samples, SW846 3550C

10.2 Sample Analysis Procedure

10.2.1 Calibrate the instrument as described in Section 11. Depending on the target compounds required by the client, it may be necessary to use more than one set of calibration standards.

10.2.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration verification (CCV) standard.

10.2.3 Add internal standard to an aliquot of the extract to result in a 40-ng/µL concentration (for example, 20 µL of internal standard solution at, 400 µg/mL in 200 µL of extract). Mix thoroughly before injection into the instrument.

10.2.4 Inject the aliquot into the GC/MS system using the same injection technique as used for the standards.

10.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration verification.

10.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst (see DV-QA-0033, Acceptable Manual Integration Practices) or automatically by the data system. The minimum documentation required includes a hard copy of original data system peak integration and a similarly scaled hard copy showing the manual integration with analyst initials and date.

10.2.7 Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

10.2.8 Library searches of peaks present in the chromatogram that are not target compounds, i.e., Tentatively Identified Compounds (TIC), may be performed if required by the client. They are evaluated using the criteria in Section 12.2.

10.2.9 The internal standard response in the sample must be within 50 - 200% of the response in the CCV.

10.2.10 Structural isomers that produce very similar mass spectra should be quantitated as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights.

10.3 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.3.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are significantly less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

10.3.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at ≤ 6°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

10.5 Retention Time Criteria for Samples

10.5.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.5.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

10.6 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP DV-WC-0023 for determination of percent moisture.

10.7 Procedural Variations

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry of the sample, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.8 Troubleshooting Guide

10.8.1 Daily Instrument Maintenance

In addition to the checks listed in Appendix B, the following daily maintenance should be performed.

- Clip column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Install new or cleaned gold seal and washer as necessary.
- Perform mass calibration as necessary.

10.8.2 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the repeller, cleaning the source, replacing the multiplier, and replacing the “top board” or RF-related electronics. Refer to the manufacturer's manual for specific guidance.

11.0 Calibration

11.1 Summary

The instrument is tuned for DFTPP, calibrated initially with a minimum of a five levels, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 3.

11.2 All standards and extracts are allowed to warm to room temperature before injecting.

11.3 Instrument Tuning

At the beginning of every twelve-hour shift when analyses are to be performed, the GC/MS system must be checked to see if the acceptance criteria are achieved for DFTPP (decafluorotriphenylphosphine), see Table 4. The mass spectrum is acquired with three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of DFTPP. The background subtraction should be designated only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or any other discrete peak that does not co-elute with DFTPP.

11.3.1 Inject 25 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 4 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

11.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. The acceptance criteria for the peak tailing factor for benzidine is < 2.0 and pentachlorophenol is < 2.0. DDT breakdown must be <20%. Refer to section 12 for the appropriate calculations.

11.3.3 Degradation of DDE and DDD must not exceed 20%.

11.4 Initial Calibration

11.4.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, Calibration Curves *(General)*.

11.4.2 Internal Standard (IS) Calibration Procedure: Internal standards are listed in Table 5. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

11.4.3 Compounds are typically assigned to the IS with the closest retention time. The laboratory tries to maintain consistent internal standard references across instruments. As a result, there may be a few cases where compounds are very close to two different internal standards that this is not true.

11.4.4 Evaluation of retention times – The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units.

11.4.5 Prepare calibration standards at a minimum of five concentration levels for

each parameter of interest when average response factors or linear regression curve fits are used. Six standards must be used for a quadratic least-squares calibration. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response.

11.4.6 For AFCEE projects, the six calibration levels will be those shown in Table 10. The table also lists a seventh calibration level that is used if a second-order regression fit is needed. The only exceptions would be for the AFCEE projects requiring special reporting limits, i.e., reporting limits different than those in the AFCEE program QAPP. Additional calibration points may be required for special projects.

11.4.7 Rejection of Calibration Points

11.4.7.1 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

11.4.7.2 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious misinjection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

- The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and
- The lowest remaining calibration point is still at or below the project reporting limit; and
- The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and
- The calibration must still have the minimum number of calibration levels required by the method, i.e. five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

11.4.8 Add the internal standard mixture to result in a 40-ng/µL final concentration. (For example, if the volume of the calibration standard used is 0.5 mL, add 50 µL of the 400 µg/mL internal standard). The concentrations of all analytes are listed in Tables 11 and 12. For the Best Practice method, see Table A-1 in Appendix A.

11.4.9 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against the concentration for each compound and internal standard. Calculate the response factors (RF), average response factors, and the percent RSD of the response factors for each compound

using the equations in section 12. No sample analysis may be performed unless the following criteria are met.

11.4.10 The RSD must be < 20% for each compound of interest.

11.4.10.1 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with RSD > 20% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve fit, then the appropriate curve should be used for quantitation.

11.4.10.2 If the RSD in the initial calibration is > 20%, then calibration using a curve fit must be used for those analytes with RSD > 20%. Linear or quadratic curve fits may be used. Use of a weighted regression is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

11.4.10.3 If a linear regression equation is used, the correlation coefficient r must be greater than 0.99. Use of second-order regression equations may be used on rare occasions. In these cases, the intercept and degree of curvature should be examined to be sure that results will be reliable throughout the working range, and the coefficient of determination must be greater than 0.990. When linear regression is used, the first point of the calibration is recalculated under the new calibration, with the values agreeing within 30% of the true values.

Note: South Carolina can only be analyzed using linear calibration.

11.4.10.4 An initial calibration verification containing all components from a second source (an alternate vendor, or, a unique lot from the same vendor) must be analyzed after the initial calibration. Acceptance criteria for ICV percent recovery (%R) are 75-125% for DoD projects (e.g., AFCEE) and 70-130% for non-DoD projects (e.g., 8270C HSL components).

Note: Several states (Arizona) and/or federal programs have special requirements. Be sure to review state QAS summaries and SOP DV-QA-024P for special requirements.

11.4.11 If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Clean or replace the injector liner and/or column, then repeat the calibration procedure.

11.4.12 The minimum response factor for the most common target analytes from Table 16 must be met.

11.4.13 Weighting of Calibration Data Points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/x^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability. Because the data system does not indicate the type of weighting used, the analyst must make a notation on the initial calibration form as to the weighting used (e.g. 1/x or 1/x^2).

11.4.14 If time remains in the 12-hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed.

NOTE: Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration. For additional information on calibrations see SOP CA-Q-S-005.

11.5 Continuing Calibration Verification (CCV)

11.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 25-ng injection of DFTPP must result in a mass spectrum for DFTPP, which meets the criteria given in Table 4.

11.5.2 Following a successful DFTPP analysis, the continuing calibration verification (CCV) standard(s) are analyzed. The standard(s) must contain all semivolatile analytes, including all required surrogates. A mid-level calibration standard is used for the CCV.

11.5.3 The following criteria must be met for the CCV to be acceptable:

- The percent difference or drift (%D) of each compound must be ≤ 20%. (See Section 12 for calculations.)

NOTE: Some states (Wisconsin) have special continuing calibration requirements when initial calibration is performed using a quadratic curve. Please refer to state specific QAS.

- Due to the large numbers of compounds that may be analyzed by this method, it is expected that some compounds will fail to meet the criterion. If the criterion is not met for more than 20% of the compounds included in the calibration, then corrective action must take place prior to the analysis for samples. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values.
- The internal standard response of the CCV must be within 50 - 200% of the response in the same level of the corresponding calibration.
- If any internal standard retention time in the CCV changes by more than 30 seconds from that of the same level of the corresponding initial

calibration, the chromatographic system must be inspected for malfunctions and corrections made, as required.

11.5.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample injected less than or equal to 12 hours after the DFTPP is acceptable.)

11.5.5 Each of the most common target analytes in the CCV must meet the minimum response factors listed in table 16. If they are not met, the system is evaluated, and corrective action takes place before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

12.0 Calculations / Data Reduction

12.1 Qualitative Identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

12.1.1 The sample component relative retention time must compare to within ± 0.06 RRT units of the relative retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

12.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.1.3 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.1.4 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.5 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Following are guidelines for making tentative identification:

12.2.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

12.2.2 The relative intensities of the major ions should agree to within ±30%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 20%and 80%.)

12.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or the presence of co-eluting compounds.

12.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.2.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.3 Isomers with identical mass spectra and close elution times pose problems for definitive identification. The following compounds fall into this category:

Aniline and bis(2-chloroethyl) ether
Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Identification of these compounds requires both experience and extra precautions on the part of the analyst. Specifically, the analyst must more closely scrutinize the comparison of retention times between the unknown and the calibration standard. The analyst must also check that all isomers have distinct retention times.

12.4 A second category of problem compounds consist of the poor responders or compounds that chromatograph poorly. The integrations for these types of compounds should be checked manually. The following compounds are included in this category:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol
Atrazine
Famphur
Benzidine

12.5 Calculating the Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{RF} \times 100\%$$

Where:

RF = Mean of RFs from the initial calibration for a compound

SD = Standard deviation for the mean RF from the initial calibration for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}}$$

RF_i = RF for each of the calibration levels

n = Number of RF values

12.6 Calculating the Continuing Calibration Percent Drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

Where:

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.7 Calculating the Concentration in the Extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.7.1 Average Response Factor Calibration

If the RSD of the response factors for each compound of interest in the initial calibration is ≤20%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

Where:

C_{ex}	=	Concentration in the extract, µg/mL
R_x	=	Response for the analyte
R_{is}	=	Response for the internal standard
C_{is}	=	Concentration of the internal standard
$\overline{RF}$	=	Average response factor

12.7.2 Linear Fit Calibration

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

Where:

C_{ex}	=	Concentration in the extract, µg/mL
R_x	=	Response for the analyte
R_{is}	=	Response for the internal standard
C_{is}	=	Concentration of the internal standard
A	=	Intercept of linear calibration line
B	=	Slope of linear calibration line

12.7.3 Quadratic Fit Calibration

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

Where:

C_{ex}	=	Concentration in the extract, µg/mL
R_x	=	Response for the analyte
R_{is}	=	Response for the internal standard
C_{is}	=	Concentration of the internal standard
A	=	Intercept
B	=	Factor for the linear term of the quadratic calibration function
C	=	Factor for the curvature term of the quadratic calibration function

12.8 Calculating the Concentration in the Sample

12.8.1 Calculation for Aqueous Samples

$$\text{Concentration, } \mu\text{g / L} = \frac{C_{ex}V_t}{V_o}$$

Where:

C_{ex} = Concentration in the extract

V_t = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then V_t = 2,000.)

V_o = Volume of the sample that was extracted (mL)

12.8.2 Calculation for Sediment, Soil, Sludge, and Waste Samples

Results for sediments, sludges, and soils are usually calculated on a dry-weight basis, and for waste, on a wet-weight basis.

$$\text{Concentration, } \mu\text{g / kg} = \frac{C_{ex}V_t}{W_sD}$$

Where:

C_{ex} = Concentration in the extract

V_t = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then V_t = 2,000.)

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry-weight basis or 1 for a wet-weight basis

12.9 MS/MSD Percent Recovery Calculation

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

S_{SR} = Spike sample result

S_R = Sample result

S_A = Spike added

12.10 Calculating the Relative Percent Difference (RPD) MS/MSD Pair

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD	=	Relative percent difference
MS_R	=	Matrix spike result
MSD_R	=	Matrix spike duplicate result

12.11 Relative Response Factor Calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x	=	Area of the characteristic ion for the compound being measured
A_{is}	=	Area of the characteristic ion for the specific internal standard
C_x	=	Concentration of the compound being measured (µg/L)
C_{is}	=	Concentration of the specific internal standard (µg/L)

12.12 Calculation of TICs

The calculation of TICs (tentatively identified compounds) is identical to the above calculation (12.11) with the following exceptions:

A_x	=	Area of the total ion chromatogram for the compound being measured
A_{is}	=	Area of the total ion chromatogram for the nearest internal standard without interference
RF	=	1

12.13 Calculating Percent DDT Breakdown

$$\%\ \text{DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDDarea}}$$

The areas for the 235 ion are used for this calculation.

12.14 Calculating the Peak Tailing Factor

$$TailingFactor = \frac{BC}{AB}$$

Where:

Peak width (AC) is measured at 10% peak height, and divided into two line segments at the peak centroid, so that:

AC = AB + BC, with

AB = left-hand segment

BC = right-hand segment

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

An initial MDL study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows

13.1.1 Prepare seven replicates at three to five times the estimated MDL concentration.

13.1.2 Extract and analyze the MDL standards as described in Section 10.

13.1.3 Calculate the mean concentration found (X) in µg/L, and the standard deviation of the mean concentration in µg/L, for each analyte. Then calculate the MDL (single-tailed, 99% confidence level, as described in Policy DV-QA-005P) for each analyte.

13.1.4 MDL studies are repeated annually, or verified quarterly, and MDL results are stored in the laboratory LIMS system. See Policy DV-QA-005P for further details concerning MDL studies.

13.1.5 The current MDL value is maintained in the TestAmerica Denver LIMS.

13.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard prepared at 1-2 times the calculated MDL concentration. An MDLV standard is analyzed immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

Acceptance Criteria:	The calculated MDL is verified if the MDLV standard is detected, nominally signal to noise ratio ≥ 3, under routine instrument conditions.
Corrective Actions:	If the first MDLV is not detected, the MDLV standard will be reprepared and analyzed at twice the original concentration. The lowest concentration that produces a detectable signal will then be reported as the MDL.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

13.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.3.2 Calculate the mean recovery and standard deviation for each analyte of interest.

13.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

13.4.1 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

13.4.2 Non-standard Analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration should include the analysis of an extracted standard at the reporting limit and a single point calibration.

14.0 Pollution Control

14.1 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, “Waste Management and Pollution Prevention”, of the Corporate Safety Manual, and HS-001, “Waste Management Program.”

15.2 The following waste streams are produced when this method is carried out

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Methylene Chloride- B

15.2.3 Flammable Solvent- Waste Stream C

15.2.4 Used vials- Waste Stream A

NOTE: Radioactive, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Revision 4, February 2007, Method 8270D.

17.0 Method Modifications:

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

Method 8270C stipulates qualitative identification based on relative retention time (RRT), which is calculated by dividing the retention time (RT) of the target analyte by the RT of the internal standard. The RRT of the suspected target analyte in the sample extract must be within ± 0.06 RRT units of the RRT for that analyte in the calibration standard. This SOP stipulates qualitative identification based on an absolute RT. Namely the RT of the suspected target analyte in the sample extract must be within ± 0.2 minute of the RT for that analyte in the calibration standard. Additionally, the RT for the internal standard in the sample extract must also be within ± 0.2 minute of the RT for the internal standard in the calibration standard. The criteria used in this SOP are more restrictive than those imposed by the referenced method. For the earliest eluting compounds, the RT for the internal standard is typically 8 minutes. The earliest eluting target analyte must be at a RRT of at least 0.8, which translates to a RT of 6.4 minutes. Assuming a worst-case scenario where the RT of the internal standard is 0.2 minute higher (i.e., 8.2 minutes) and the RT of the target analyte is 0.2 minute lower (i.e., 6.2 minutes), the calculated RRT is 0.76. The total deviation from the expected RRT is 0.04 RRT units, which is smaller than what is allowed by Method 8270C.

17.1.2 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.3 This procedure includes the option for weighted linear regression curves using $1/concentration^2$ weighting factors. Section 7.5.2 of Method 8000B discusses the use of weighted least square regression based on $1/standard\ deviation^2$ weighting factors, which would require multiple analyses of each standard to determine the standard deviation. IAETL has presented information to the EPA Office of Solid Waste demonstrating that the variance ($standard\ deviation^2$) is proportional to the standard concentration. EPA accepted this argument and issued a memorandum dated August 7, 1998 (Attachments dated July 1998), which authorizes the use of $1/concentration^2$ weighting factors.

18.0 Attachments

Table 1. TAL Primary Standard and Standard Reporting Limits

Table 2. TAL Appendix IX Standard Reporting Limits

Table 3. Suggested Instrument Conditions

Table 4. DFTPP Key Ions and Ion Abundance Criteria

Table 5. Characteristic Ions, Primary Standard (in approximate retention time order)

Table 6. Characteristic Ions, Appendix IX Standard (in approximate retention time order)

Table 7. 8270D LCS Compounds

Table 8. TCLP LCS Compounds

Table 9. 8270D Surrogate Compounds

Table 10. Calibration Levels for AFCEE Projects, µg/mL

Table 11. Calibration Levels, Primary Standard, µg/mL

Table 12. Calibration Levels, Appendix IX Standard, µg/mL

Table 13. Initial Demonstration Recovery and Precision Limits

Table 14. List 1 Reliably Performing Compounds

Table 15. List 2 Poorly Performing Compounds

Table 16 Minimum Response Factor Criteria for Initial and Continuing Calibration Verification

APPENDIX A. Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (8270BP)

- Table A-1. TAL Method 8270BP Standard Reporting Limits
- Table A-2. Method 8270BP Calibration Levels
- Table A-3. Method 8270BP LCS Spike Concentrations
- Table A-4. 8270BP Surrogate Compounds
- Table A-5. Suggested Instrument Conditions for 8270BP

APPENDIX B. Suggested Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

19.0 Revision History

- Revision 3, dated 4 January 2013
  - Changed storage of extracts from freezer to refrigerator.
- Revision 2, dated 30 November 2012
  - Deleted Section 5.1.5 – About the use of Separatory Funnels
  - Updated the Hazardous Materials table in Section 5.2 to reflect current solvent used.
  - Updated and clarified language in Attachments to reflect current practices.

- Revision 1.1, dated 01 December 2011
  - Added note to section 1.1.1 and Appendix A restricting use of Best Practice method
  - Revised sections 9.5, 9.6 and 9.7 to clarify use of flags and documentation in narrative for failed QC
  - Revised section 9.8.1 to clarify modifications might be made to accommodate the chemistry of the sample.
  - Added statement to 11.5.3 to flag data if target analyte reported for failed CCV.
  - Expanded the discussion in section 17.1.1 to clarify how the use of RT windows stipulated in this SOP meets or exceeds the requirements of Method 8270D.
  - Clarified reference from EPA for source of inverse weighted least squares regression for calibrations.

- Revision 1.0, dated 31 January 2011
  - Updated Table 3, Suggested Instrument Conditions
  - Added components and changed spike levels in Table 7, 8270D LCS Compounds
  - Added low level calibration standard to Table 11, Calibration Levels, Primary Standard
  - Minor grammatical, spelling and formatting changes were made throughout.

- Revision 0.1, dated 11 December 2009
  - Added requirements for degradation criteria for DDD and DDE in the tune to section 11.3.3.
  - Removed statement allowing an ICV standard from the same vendor and lot as long as it is prepared by a separate analyst from section 11.4.10.4.

Table 1.

TAL Primary Standard and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
Pyridine	110-86-1	20	660
N-Nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	4	330
1,4-Dichlorobenzene	106-46-7	4	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	4	330
2-Methylphenol	95-48-7	10	330
2,2'-Oxybis(1-chloropropane)[2]	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	4	330
2-Nitroaniline	88-74-4	10	1600
Dimethyl phthalate	131-11-3	4	330
Acenaphthylene	208-96-8	4	330
3-Nitroaniline	99-09-2	10	1600
Acenaphthene	83-32-9	4	330
2,4-Dinitrophenol	51-28-5	30	1600
4-Nitrophenol	100-02-7	10	1600
Dibenzofuran	132-64-9	4	330
2,4-Dinitrotoluene	121-14-2	10	330

Table 1.

TAL Primary Standard and Standard Reporting Limits (cont.)

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	4	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	4	330
4-Nitroaniline	100-01-6	10	1600
4,6-Dinitro-2-methylphenol	534-52-1	20	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	4	330
Anthracene	120-12-7	4	330
Carbazole	86-74-8	4	330
Di-n-butyl phthalate	84-74-2	4	330
Fluoranthene	206-44-0	4	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	4	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	4	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
4,4-Methylenebis(2-chloroaniline)	101-14-4	100	330
Chrysene	218-01-9	4	330
Di-n-octylphthalate	117-84-0	4	330
Benzo(b)fluoranthene	205-99-2	4	330
Benzo(k)fluoranthene	207-08-9	4	330
Benzo(a)pyrene	50-32-8	4	330
Indeno(1,2,3-cd)pyrene	193-39-5	4	330
Diethyl phthalate	84-66-2	4	660
Dibenz(a,h)anthracene	53-70-3	4	330
Benzo(g,h,i)perylene	191-24-2	4	330
Acetophenone	98-86-2	10	330
3/4-Methylphenol	108-39-4	10	330
1,4-Dioxane	54841-74-6	20	660

1. The TAL primary standard is the standard normally used at TAL. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.
2. 2,2'-Oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether.

Table 2.

TAL Appendix IX Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	10	660
N-Nitrosopiperidine	100-75-4	10	330
O,O,O-Triethyl-Phosphorothioate	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	1600
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	50	1600
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin	297-97-2	10	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp	3689-24-5	50	1000
Phorate	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate	2303-16-4	20	660
Dimethoate	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	10	660
Methyl Parathion	298-00-0	50	1600

Table 2.

TAL Appendix IX Standard Reporting Limits (cont.)

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
1-chloronaphthalene	90-13-1	10	330
Biphenyl	92-51-3	10	330
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
Isodrin	465-73-6	10	330
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	20	660
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	10	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660
Diphenylamine	122-39-4	10	330

1. The Appendix IX standard contains additional analytes required for the Appendix IX list. The TAL primary standard must also be analyzed to include all of the Appendix IX list.
2. May also be analyzed by method 8141, which can achieve lower reporting limits.
3. May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits.

Table 3.

Suggested Instrument Conditions[1]

Mass Range:	35 - 500 amu
Scan Time:	≤ 1 second/scan
Initial Column Temperature/Hold Time:	55 °C for 1.5 minutes
Column Temperature Program:	25 ˚C/min. to 250 °C then 5 °C/min. to 330 ˚C
Final Column Temperature/Hold Time:	330 ˚C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature:	250 °C
Transfer Line Temperature:	300 °C
Source Temperature:	According to manufacturer's specifications
Injector:	Grob-type, split / splitless
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 3.4 mL/min.

[1]The GC parameters should be optimized to provide appropriate resolution for benzo(b)fluoranthene and benzo(k)fluoranthene and dibenz(a,h)anthracene and indeno(1,2,3-cd)pyrene.

Table 4.

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	40 - 100% of mass 198
443	17 - 23% of mass 442

Table 5.

Characteristic Ions, Primary Standard (in approximate retention time order)

Analyte	Primary	Secondary	Tertiary
N-Nitrosodimethylamine	74	42	--
1,4-Dioxane	88	58	--
Pyridine	79	52	--
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d_5 (Surrogate Standard)	99	42	71
Aniline	93	66	--
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d_4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	77
2,2'-Oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d_5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d_8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	271
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153

1. 2,2'-Oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether.

Table 5.

Characteristic Ions, Primary Standard (in approximate retention time order) (cont.)

Analyte	Primary	Secondary	Tertiary
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d_{10} (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	105	51
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d_{10} (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d_{14} (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Famphur	218	93	125
Benzo(a)Anthracene	228	229	226
Chrysene-d_{12} (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
4,4-Methylenebis(2-Chloroanaline)	231	266	--
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d_{12} (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 6.

Characteristic Ions, Appendix IX Standard (in approximate retention time order)

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	77
3/4-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
O,O,O-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	--
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	54
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	50	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	--
2-Naphthylamine	143	115	--
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	--
Dimethoate	87	93	125
4-Aminobiphenyl	169	168	115
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160

Table 6.

Characteristic Ions, Appendix IX Standard (in approximate retention time order) (cont.)

Analyte	Primary	Secondary	Tertiary
Parathion	109	97	291
Isodrin	193	66	195
Famphur	218	125	93
Methapyrilene	97	58	--
Aramite 1	185	319	--
Aramite 2	185	319	--
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	--
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	--
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 7.

8270D LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
Azobenzene	80
Acetophenone	80
Acenaphthylene	80
Benzo[a]anthracene	80
Benzo[b]fluoranthene	80
Benzo[k]fluoranthene	80
Benzoic acid	80
Benzo[g,h,i]perylene	80
Benzo[a]pyrene	80
Benzyl alcohol	80
Bis(2-chloroethoxy)methane	80
Bis(2-ethylhexyl) phthalate	80
Butyl benzyl phthalate	80
Bis(2-chloroethyl)ether	80
Carbazole	80
Chrysene	80

Table 7.

8270D LCS Compounds (cont.)

LCS Compounds	Spiking Level, ng/µL in extract
Di-n-butyl phthalate	80
Di-n-octyl phthalate	80
Dibenz(a,h)anthracene	80
Dibenzofuran	80
Diethyl phthalate	80
Dimethyl phthalate	80
Diphenylamine	80
Ethyl methanesulfonate	80
Fluoranthene	80
Fluorene	80
Hexachlorobenzene	80
Hexachlorobutadiene	80
Hexachlorocyclopentadiene	80
Hexachloroethane	80
Indeno(1,2,3-cd)pyrene	80
Isosafrole	80
Isophorone	80
Methyl methanesulfonate	80
N-Nitrodimethylamine	80
N-Nitrosodi-n-butylamine	80
N-Nitrosodiethylamine	80
N-Nitrosodi-n-propylamine	80
N-Nitrosodiphenylamine	80
N-Nitrosomethylethylamine	80
N-Nitrosomorpholine	80
N-Nitrosopiperidine	80
N-Nitrosopyrrolidine	80
Pentachlorobenzene	80
Pentachloroethane	80
Pentachloronitrobenzene	80
Pentachlorophenol	80
Phenacetin	80

Table 7.

8270D LCS Compounds (cont.)

LCS Compounds	Spiking Level, ng/µL in extract
Phenanthrene	80
Phenol	80
Pyrene	80
Pyridine	80
Safrole, Total	80
1,4-Dichlorobenzene	80
Naphthalene	80
2-Methylnaphthalene	80
3-Methylcholanthrene	80
1-Naphthylamine	80
Nitrobenzene	80
2-Picoline	80
7,12-Dimethylbenz(a)anthracene	80
2-Fluorobiphenyl	80
2-Fluorophenol	80
2,4,6-Tribromophenol	80
Nitrobenzene-d5	80
Phenol-d5	80
Terphenyl-d14	80
1,4-Dichlorobenzene-d4	80
Acenaphthene-d10	80
Chrysene-d12	80
Naphthalene-d8	80
Phenanthrene-d10	80
Perylene-d12	80
1-Chloronaphthalene	80
2,3,4,6-Tetrachlorophenol	80
alpha,alpha-Dimethyl phenethylamine	80
Benzidine	80
Dibenz[a,j]acridine	80
p-Dimethylamino azobenzene	80
Pronamide	80
1,3,5-Trinitrobenzene	80

Table 7.

8270D LCS Compounds (cont.)

LCS Compounds	Spiking Level, ng/µL in extract
1,3-Dinitrobenzene	80
1,4-Dinitrobenzene	80
1,4-Naphthoquinone	80
2-Acetylaminofluorene	80
3,3'-Dimethylbenzidine	80
4-Nitroquinoline-1-oxide	80
N-Nitro-o-toluidine	80
Aramite, Total	80
Aramite Peak 1	80
Aramite Peak 2	80
1,1'-Biphenyl	80
Chlorobenzilate	80
Diallate	80
Dimethoate	80
Disulfoton	80
Hexachloropropene	80
Isodrin	80
Methapyrilene	80
Methyl parathion	80
O,O',O''-Triethylphosphorothioate	80
Ethyl Parathion	80
Phorate	80
p-Phenylenediamine	80
Sulfotepp	80
Thionazin	80
N-Nitrosodimethylamine	80
Dinoseb	80

Table 8.

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
1,4-Dichlorobenzene	50
2,4-Dinitrotoluene	50
Hexachlorobenzene	50
Hexachlorobutadiene	50
Hexachloroethane	50
2-Methylphenol	50
3/4-Methylphenol	100
Nitrobenzene	50
Pentachlorophenol	100
Pyridine	50
2,4,5-Trichlorophenol	50
2,4,6-Trichlorophenol	50

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA group.

Table 9.

8270D Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d_5	100
2-Fluorobiphenyl	100
Terphenyl-d_{14}	100
1,2-Dichlorobenzene-d_4[1]	100
Phenol-d_5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d_4[1]	150

1. Included in standard mix, but not routinely evaluated for method 8270D. Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 10.

Calibration Levels for AFCEE Projects, µg/mL

Analyte	Std Conc 1	Std Conc 2	Std Conc 3	Std Conc 4	Std Conc 5	Std Conc 6	Addnl Conc for 2nd Order ICALs
Pyridine	20	50	80	120	200	---	160
N-Nitrosodimethylamine	10	20	50	80	120	200	---
Aniline	10	20	50	80	120	200	---
Phenol	10	20	50	80	120	200	---
Bis(2-chloroethyl)ether	10	20	50	80	120	200	---
2-Chlorophenol	10	20	50	80	120	200	---
1,3-Dichlorobenzene	10	20	50	80	120	200	---
1,4-Dichlorobenzene	10	20	50	80	120	200	---
Benzyl alcohol	10	20	50	80	120	200	---
1,2-Dichlorobenzene	10	20	50	80	120	200	---
2-Methylphenol	10	20	50	80	120	200	---
2,2'-Oxybis(1-chloropropane)[1]	10	20	50	80	120	200	---
4-Methylphenol	10	20	50	80	120	200	---
N-Nitroso-di-n-propylamine	10	20	50	80	120	200	---
Hexachloroethane	10	20	50	80	120	200	---
Nitrobenzene	10	20	50	80	120	200	---
Isophorone	10	20	50	80	120	200	---
2-Nitrophenol	10	20	50	80	120	200	---
2,4-Dimethylphenol	10	20	50	80	120	200	---
Benzoic acid	20	50	80	120	200	---	160
Bis(2-chloroethoxy)methane	10	20	50	80	120	200	---
2,4-Dichlorophenol	10	20	50	80	120	200	---
1,2,4-Trichlorobenzene	10	20	50	80	120	200	---
Naphthalene	10	20	50	80	120	200	---
4-Chloroaniline	10	20	50	80	120	200	---
Hexachlorobutadiene	10	20	50	80	120	200	---
4-Chloro-3-methylphenol	10	20	50	80	120	200	---
2-Methylnaphthalene	10	20	50	80	120	200	---
Hexachlorocyclopentadiene	20	50	80	120	200	---	160
2,4,6-Trichlorophenol	10	20	50	80	120	200	---
2,4,5-Trichlorophenol	10	20	50	80	120	200	---
2-Chloronaphthalene	10	20	50	80	120	200	---
2-Nitroaniline	20	50	80	120	200	---	160
Dimethyl phthalate	10	20	50	80	120	200	---
Acenaphthylene	10	20	50	80	120	200	---
3-Nitroaniline	20	50	80	120	200	---	160
Acenaphthene	10	20	50	80	120	200	---
2,4-Dinitrophenol	20	50	80	120	200	---	160
4-Nitrophenol	20	50	80	120	200	---	160
Dibenzofuran	10	20	50	80	120	200	---
2,4-Dinitrotoluene	10	20	50	80	120	200	---
2,6-Dinitrotoluene	10	20	50	80	120	200	---
Diethylphthalate	10	20	50	80	120	200	---

Table 10.

Calibration Levels for AFCEE Projects, µg/mL (cont.)

Analyte	Std Conc 1	Std Conc 2	Std Conc 3	Std Conc 4	Std Conc 5	Std Conc 6	Addnl Conc for 2nd Order ICALs
4-Chlorophenyl phenyl ether	10	20	50	80	120	200	---
Fluorene	10	20	50	80	200	---	---
4-Nitroaniline	20	50	80	120	200	---	---
4,6-Dinitro-2-methylphenol	20	50	80	120	200	---	---
N-Nitrosodiphenylamine	10	20	50	80	200	---	---
Azobenzene[2]	10	20	50	80	200	---	---
4-Bromophenyl phenyl ether	10	20	50	80	200	---	---
Hexachlorobenzene	10	20	50	80	200	---	---
Pentachlorophenol	20	50	80	120	200	---	---
Phenanthrene	10	20	50	80	200	---	---
Anthracene	10	20	50	80	200	---	---
Carbazole	10	20	50	80	200	---	---
Di-n-butyl phthalate	10	20	50	80	200	---	---
Fluoranthene	10	20	50	80	200	---	---
Benzidine	20	50	80	120	200	---	---
Pyrene	10	20	50	80	200	---	---
Butyl benzyl phthalate	10	20	50	80	200	---	---
3,3'-Dichlorobenzidine	10	50	80	120	200	---	---
Benzo(a)anthracene	10	20	50	80	200	---	---
Bis(2-ethylhexyl)phthalate	10	20	50	80	200	---	---
Chrysene	10	20	50	80	200	---	---
Di-n-octylphthalate	10	20	50	80	200	---	---
Benzo(b)fluoranthene	10	20	50	80	200	---	---
Benzo(k)fluoranthene	10	20	50	80	200	---	---
Benzo(a)pyrene	10	20	50	80	200	---	---
Indeno(1,2,3-cd)pyrene	10	20	50	80	200	---	---
Dibenz(a,h)anthracene	10	20	50	80	200	---	---
Diethyl phthalate	10	20	50	80	200	---	---
Benzo(g,h,i)perylene	10	20	50	80	200	---	---

1. 2,2'-Oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether.
2. Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 11.

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Pyridine	--	10	20	50	80	120	160	200
N-Nitrosodimethylamine	4	10	20	50	80	120	160	200
Aniline	4	10	20	50	80	120	160	200
Phenol	4	10	20	50	80	120	160	200
Bis(2-chloroethyl)ether	4	10	20	50	80	120	160	200
2-Chlorophenol	4	10	20	50	80	120	160	200
1,3-Dichlorobenzene	4	10	20	50	80	120	160	200
1,4-Dichlorobenzene	4	10	20	50	80	120	160	200
Benzyl alcohol	4	10	20	50	80	120	160	200
1,2-Dichlorobenzene	4	10	20	50	80	120	160	200
2-Methylphenol	4	10	20	50	80	120	160	200
2,2'-Oxybis(1-chloropropane)[1]	4	10	20	50	80	120	160	200
4-Methylphenol	4	10	20	50	80	120	160	200
N-Nitroso-di-n-propylamine	4	10	20	50	80	120	160	200
Hexachloroethane	4	10	20	50	80	120	160	200
Nitrobenzene	4	10	20	50	80	120	160	200
Isophorone	4	10	20	50	80	120	160	200
2-Nitrophenol	4	10	20	50	80	120	160	200
2,4-Dimethylphenol	4	10	20	50	80	120	160	200
Benzoic acid	--	10	20	50	80	120	160	200
Bis(2-chloroethoxy)methane	4	10	20	50	80	120	160	200
2,4-Dichlorophenol	4	10	20	50	80	120	160	200
1,2,4-Trichlorobenzene	4	10	20	50	80	120	160	200
Naphthalene	4	10	20	50	80	120	160	200
4-Chloroaniline	4	10	20	50	80	120	160	200
Hexachlorobutadiene	4	10	20	50	80	120	160	200
4-Chloro-3-methylphenol	4	10	20	50	80	120	160	200
2-Methylnaphthalene	4	10	20	50	80	120	160	200
Hexachlorocyclopentadiene	--	10	20	50	80	120	160	200
2,4,6-Trichlorophenol	4	10	20	50	80	120	160	200
2,4,5-Trichlorophenol	4	10	20	50	80	120	160	200
2-Chloronaphthalene	4	10	20	50	80	120	160	200
2-Nitroaniline	4	10	20	50	80	120	160	200
Dimethyl phthalate	4	10	20	50	80	120	160	200
Acenaphthylene	4	10	20	50	80	120	160	200
3-Nitroaniline	4	10	20	50	80	120	160	200
Acenaphthene	4	10	20	50	80	120	160	200
2,4-Dinitrophenol	--	10	20	50	80	120	160	200
4-Nitrophenol	--	10	20	50	80	120	160	200
Dibenzofuran	4	10	20	50	80	120	160	200
2,4-Dinitrotoluene	4	10	20	50	80	120	160	200
2,6-Dinitrotoluene	4	10	20	50	80	120	160	200
Diethylphthalate	4	10	20	50	80	120	160	200
4-Chlorophenyl phenyl ether	4	10	20	50	80	120	160	200

Table 11.

Calibration Levels, Primary Standard, µg/mL (cont.)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Fluorene	4	10	20	50	80	120	160	200
4-Nitroaniline	--	10	20	50	80	120	160	200
4,6-Dinitro-2-methylphenol	--	10	20	50	80	120	160	200
N-Nitrosodiphenylamine	4	10	20	50	80	120	160	200
Azobenzene[2]	4	10	20	50	80	120	160	200
4-Bromophenyl phenyl ether	4	10	20	50	80	120	160	200
Hexachlorobenzene	4	10	20	50	80	120	160	200
Pentachlorophenol	--	10	20	50	80	120	160	200
Phenanthrene	4	10	20	50	80	120	160	200
Anthracene	4	10	20	50	80	120	160	200
Carbazole	4	10	20	50	80	120	160	200
Di-n-butyl phthalate	4	10	20	50	80	120	160	200
Fluoranthene	4	10	20	50	80	120	160	200
Benzidine	--	10	20	50	80	120	160	200
Pyrene	4	10	20	50	80	120	160	200
Butyl benzyl phthalate	4	10	20	50	80	120	160	200
3,3'-Dichlorobenzidine	--	10	20	50	80	120	160	200
Benzo(a)anthracene	4	10	20	50	80	120	160	200
Bis(2-ethylhexyl)phthalate	4	10	20	50	80	120	160	200
4,4-Methylenebis(2-chloroaniline)	4	10	20	50	80	120	160	200
Chrysene	4	10	20	50	80	120	160	200
Di-n-octylphthalate	4	10	20	50	80	120	160	200
Benzo(b)fluoranthene	4	10	20	50	80	120	160	200
Benzo(k)fluoranthene	4	10	20	50	80	120	160	200
Benzo(a)pyrene	4	10	20	50	80	120	160	200
Indeno(1,2,3-cd)pyrene	4	10	20	50	80	120	160	200
Dibenz(a,h)anthracene	4	10	20	50	80	120	160	200
Diethyl phthalate	4	10	20	50	80	120	160	200
Benzo(g,h,i)perylene	4	10	20	50	80	120	160	200

1. 2,2'-Oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether
2. Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 12.
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2-Picoline	10	20	50	80	120	160	200
N-Nitrosomethylethylamine	10	20	50	80	120	160	200
Methyl methanesulfonate	10	20	50	80	120	160	200
N-Nitrosodiethylamine	10	20	50	80	120	160	200
Ethyl methanesulfonate	10	20	50	80	120	160	200
Pentachloroethane	--	20	50	80	120	160	200
Acetophenone	10	20	50	80	120	160	200
N-Nitrosopyrrolidine	10	20	50	80	120	160	200
N-Nitrosomorpholine	10	20	50	80	120	160	200
o-Toluidine	10	20	50	80	120	160	200
3-Methylphenol	10	20	50	80	120	160	200
N-Nitrosopiperidine	10	20	50	80	120	160	200
O,O,O-Triethyl-Phosphorothioate	--	20	50	80	120	160	200
a,a-Dimethyl-phenethylamine	--	20	50	80	120	160	200
2,6-Dichlorophenol	10	20	50	80	120	160	200
Hexachloropropene	--	20	50	80	120	160	200
p-Phenylenediamine	--	20	50	80	120	160	200
n-Nitrosodi-n-butylamine	10	20	50	80	120	160	200
Safrole	--	20	50	80	120	160	200
1,2,4,5-Tetrachlorobenzene	10	20	50	80	120	160	200
Isosafrole 1 + 2	10	20	50	80	120	160	200
1,4-Dinitrobenzene	10	20	50	80	120	160	200
1,4-Naphthoquinone	--	20	50	80	120	160	200
1,3-Dinitrobenzene	10	20	50	80	120	160	200
Pentachlorobenzene	10	20	50	80	120	160	200
1-Naphthylamine	10	20	50	80	120	160	200
2-Naphthylamine	10	20	50	80	120	160	200
2,3,4,6-Tetrachlorophenol	--	20	50	80	120	160	200
5-Nitro-o-toluidine	10	20	50	80	120	160	200
Thionazin	10	20	50	80	120	160	200
1,3,5-Trinitrobenzene	--	20	50	80	120	160	200
Sulfotepp	--	20	50	80	120	160	200
Phorate	--	20	50	80	120	160	200
Phenacetin	10	20	50	80	120	160	200
Diallate 1 + 2	10	20	50	80	120	160	200
Dimethoate	10	20	50	80	120	160	200
4-Aminobiphenyl	--	20	50	80	120	160	200
Pentachloronitrobenzene	--	20	50	80	120	160	200
Pronamide	10	20	50	80	120	160	200
Disulfoton	--	20	50	80	120	160	200
2-secbutyl-4,6-dinitrophenol (Dinoseb)	10	20	50	80	120	160	200
Methyl parathion	--	20	50	80	120	160	200
4-Nitroquinoline-1-oxide	--	20	50	80	120	160	200
Parathion	--	20	50	80	120	160	200

Table 12.

Calibration Levels, Appendix IX Standard, µg/mL (cont.)

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Isodrin	10	20	50	80	120	160	200
Methapyrilene	--	20	50	80	120	160	200
Aramite 1 and 2	10	20	50	80	120	160	200
p-(Dimethylamino) azobenzene	10	20	50	80	120	160	200
p-Chlorobenzilate	10	20	50	80	120	160	200
3,3'-Dimethylbenzidine	10	20	50	80	120	160	200
2-Acetylaminofluorene	--	20	50	80	120	160	200
Dibenz (a,j)acridine	10	20	50	80	120	160	200
7,12-Dimethylbenz(a) anthracene	10	20	50	80	120	160	200
3-Methylcholanthrene	10	20	50	80	120	160	200

Table 13.

Initial Demonstration Recovery and Precision Limits

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(g,h,i)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
β-BHC[1]	60	31.5	41.5-130.6
δ-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7

Table 13.

Initial Demonstration Recovery and Precision Limits (cont.)

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

1. Organochlorine pesticides and PCBs project DQOs generally require better sensitivity than is provided by 8270D, so methods 8081 and 8082 are used instead. These compounds will not be included in the initial demonstration of capability for method 8270D.

Table 14.

List 1 Reliably Performing Compounds

Acenaphthene	Dibenzofuran	1H-Indene
Acenaphthylene	1,4-Dioxane	Indeno(1,2,3-cd)pyrene
Acetophenone	n-Dodecane	Isophorone
Alachlor	n-Docosane	1-Methylnaphthalene
Aniline	1,2-Dichlorobenzene	2-Methylnaphthalene
Anthracene	1,3-Dichlorobenzene	2-Methylphenol
Atrazine	1,4-Dichlorobenzene	4-Methylphenol
Benzo(a)anthracene	2,3-Dichlorobenzeneamine	Methylstyrene
Benzo(a)pyrene	3,3'-Dichlorobenzidine	Naphthalene
Benzo(b)fluoranthene	2,4-Dichlorophenol	2-Nitroaniline
Benzo(k)fluoranthene	Diethyl phthalate	3-Nitroaniline
Benzo(g,h,i)perylene	2,4-Dimethylphenol	4-Nitroaniline
Benzoic acid	Dimethyl phthalate	Nitrobenzene
Benzyl alcohol	Di-n-butyl phthalate	2-Nitrophenol
Bis(2-chloroethoxy)methane	4,6-Dinitro-2-methylphenol	4-Nitrophenol
Bis(2-chloroethyl)ether	2,4-Dinitrophenol	N-Nitrosodimethylamine
Bis(2-ethylhexyl)phthalate	2,4-Dinitrotoluene	N-Nitroso-di-n-propylamine
4-Bromophenyl phenyl ether	2,6-Dinitrotoluene	N-Nitrosodiphenylamine
Butyl benzyl phthalate	1,2-Diphenylhydrazine (as Azobenzene)	2,2'-Oxybis(1-chloropropane) aka "bis(2-chloroisopropyl) ether"
Caprolactam	Di-n-octyl phthalate	n-Octadecane
Carbazole	n-Eicosane	Pentachlorophenol
4-Chloroaniline	Famphur	Phenanthrene
4-Chloro-3-methylphenol	Fluoranthene	Phenol
2-Chloronaphthalene	Fluorene	Pyrene
2-Chlorophenol	Hexachlorobenzene	Pyridine
4-Chlorophenyl phenyl ether	Hexachlorocyclopentadiene	n-Tetradecane
Chrysene	Hexachlorobutadiene	1,2,4-Trichlorobenzene
n-Decane	Hexachloroethane	2,4,5-Trichlorophenol
Dibenz(a,h)anthracene	n-Hexadecane	2,4,6-Trichlorophenol

Table 15.

List 2 Poorly Performing Compounds

2-Acetylaminofluorene	Diphenylamine	N-Nitrosopyrrolidine
Acrylamide	Disulfoton	Parathion
4-Aminobiphenyl	2-Ethoxyethanol	Pentachlorobenzene
Aramite (#1)	Ethyl methanesulfonate	Pentachloroethane
Aramite (#2)	Hexachlorophene	Pentachloronitrobenzene
Benzenethiol	Hexachloropropene	Perylene
Benzidine	Isosafrole (#1)	Phenacetin
Benzyl chloride	Isosafrole (#2)	p-Phenylenediamine
Biphenyl	Isodrin	Phorate
Carbofuran phenol	Methapyrilene	Phthalic anhydride
Chlorobenzilate	Methomyl	2-Picoline
Diallate (#1)	3-Methylcholanthrene	Pronamide
Diallate (#2)	6-Methylchrysene	Quinoline
Dibenz(a,h)acridine	4,4'-Methylenebis(2-chloroaniline)	Safrole
Dibenz(a,j)acridine	Methyl methanesulfonate	2-secbutyl-4,6-dinitrophenol (Dinoseb)
Dibenzo(a,e)pyrene	Methyl Parathion	Sulfotepp
Tris(2,3-dibromopropyl) phosphate	1-Naphthylamine	1,2,4,5-Tetrachlorobenzene
2,6-Dichlorophenol	2-Naphthylamine	2,3,4,6-Tetrachlorophenol
Dimethoate	1,4-Naphthoquinone	Thionazin
p-(Dimethylamino)azobenzene	5-Nitro-o-toluidine	o-Toluidine
7,12-Dimethylbenz(a)anthracene	4-Nitroquinoline-1-oxide	2,4- and 2,6-Toluenediamine
3,3'-Dimethylbenzidine	N-Nitrosodiethylamine	Triethylamine
N,N-Dimethylformamide	n-Nitrosodi-n-butylamine	Triethylphosphate
a,a-Dimethyl-phenethylamine	N-Nitrosomethylethylamine	*O,O,O*-Triethylphosphorothioate
1,3-Dinitrobenzene	N-Nitrosomorpholine	1,3,5-Trinitrobenzene
1,4-Dinitrobenzene	N-Nitrosopiperidine	

Table 16
Minimum Response Factor Criteria for Initial and Continuing Calibration Verification

Analyte	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis(1-chloropropane)[1]	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethylphthalate	0.010
Acenaphthylene	0.900
2,6-Dinitrotoluene	0.200
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
Dibenzofuran	0.800
4-Nitrophenol	0.010
2,4-Dinitrotoluene	0.200
Diethylphthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
Fluorene	0.900
4-Chlorophenylphenylether	0.400
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
N-Nitrosodiphenylamine	0.010
4-Bromophenylphenylether	0.100

1. 2,2'-Oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether

Table 16
Minimum Response Factor Criteria for Initial and Continuing Calibration Verification (cont.)

Analyte	Minimum Response Factor (RF)
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butylphthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butylbenzylphthalate	0.010
Benzo(a)anthracene	0.800
3,3'-Dichlorobenzidine	0.010
Chrysene	0.700
Bis(2-ethylhexyl)phthalate	0.010
Di-n-octylphthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

APPENDIX A

Modifications Required for Analysis of Samples Following Method 8270 Best Practice (8270BP)

NOTE: The 8270 Best Practice method is NOT applicable for the analysis of South Carolina regulatory compliance samples.

REQUIREMENTS FOR METHOD 8270 BEST PRACTICE (8270BP)

- Method Best Practice is utilized to obtain lower reporting limits while still providing full scan data. The standard analyte list and reporting limits are listed in Table A-1.
- This method is only applicable to the analysis of low level samples. The appropriate range for aqueous samples is 1 to 100 ug/L, and 30 to 1650 ug/Kg for soils. Attempts to analyze samples with concentrations much higher than this for target compounds, or high concentrations of non-target compounds will likely result in a decline in the quality control parameters for the method. Once the instrument has been adversely impacted by high level samples, it may not be possible to bring it back into control in a reasonable time frame.
- The extraction is the same with one exception. The final volume of the extract is 2 mL.
- The tune period for this method is defined as 12 hours.
- Initial calibration curve requirements are as follows:
  - Same as for 8270 detailed in Section 11.4 of this SOP.
  - The calibrations levels are shown in Table A-2.
- Continuing calibration verification requirements are as follows:
  - Same as for 8270 detailed in Section 11.5 of this SOP, except that 7 calibration point levels are used.
- Matrix Spike and LCS requirements are as follows:
  - The spike levels are listed in Table A-3.
- Surrogates: The surrogate concentrations are listed in Table A-4.
- Instrument Conditions are shown in Table A-5.

Table A-1.

TAL Method 8270BP Standard Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Pyridine	110-86-1	20
N-Nitrosodimethylamine	62-75-9	5
Aniline	62-53-3	5
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	1
2-Chlorophenol	95-57-8	5
Benzyl alcohol	100-51-6	5
2-Methylphenol	95-48-7	5
2,2'-Oxybis(1-chloropropane)[1]	108-60-1	5
4-Methylphenol	106-44-5	5
N-Nitroso-di-n-propylamine	621-64-7	5
Hexachloroethane	67-72-1	5
Nitrobenzene	98-95-3	5
Isophorone	78-59-1	5
2-Nitrophenol	88-75-5	5
Benzoic acid	65-85-0	10
Bis(2-chloroethoxy)methane	111-91-1	5
2,4-Dichlorophenol	120-83-2	5
1,2,4-Trichlorobenzene	120-82-1	5
Naphthalene	91-20-3	5
4-Chloroaniline	106-47-8	5
Hexachlorobutadiene	87-68-3	5
4-Chloro-3-methylphenol	59-50-7	5
2-Methylnaphthalene	91-57-6	5
Hexachlorocyclopentadiene	77-47-4	5
2,4,6-Trichlorophenol	88-06-2	5
2,4,5-Trichlorophenol	95-95-4	5
2-Chloronaphthalene	91-58-7	5
2-Nitroaniline	88-74-4	5
Dimethyl phthalate	131-11-3	5
Acenaphthylene	208-96-8	5
3-Nitroaniline	99-09-2	5
Acenaphthene	83-32-9	5
2,4-Dinitrophenol	51-28-5	5
4-Nitrophenol	100-02-7	5
Dibenzofuran	132-64-9	5
2,4-Dinitrotoluene	121-14-2	5
2,6-Dinitrotoluene	606-20-2	5
4-Chlorophenyl phenyl ether	7005-72-3	5
Fluorene	86-73-7	5
4-Nitroaniline	100-01-6	5
4,6-Dinitro-2-methylphenol	534-52-1	10
N-Nitrosodiphenylamine	86-30-6	5
Azobenzene	103-33-3	5

1. 2,2'-Oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether

Table A-1.

TAL Method 8270BP Standard Reporting Limits (cont.)

Analytes	CAS Number	Aqueous, µg/L
4-Bromophenyl phenyl ether	101-55-3	5
Hexachlorobenzene	118-74-1	1
Pentachlorophenol	87-86-5	10
Phenanthrene	85-01-8	1
Anthracene	120-12-7	5
Carbazole	86-74-8	5
Di-n-butyl phthalate	84-74-2	5
Fluoranthene	206-44-0	1
Benzidine	92-87-5	1
Pyrene	129-00-0	5
Butyl benzyl phthalate	85-68-7	5
3,3'-Dichlorobenzidine	91-94-1	5
Benzo(a)anthracene	56-55-3	1
Bis(2-ethylhexyl)phthalate	117-81-7	5
Chrysene	218-01-9	1
Di-n-octylphthalate	117-84-0	5
Benzo(b)fluoranthene	205-99-2	5
Benzo(k)fluoranthene	207-08-9	5
Benzo(a)pyrene	50-32-8	5
Indeno(1,2,3-cd)pyrene	193-39-5	5
Diethyl phthalate	84-66-2	5
Dibenz(a,h)anthracene	53-70-3	5
Benzo(g,h,i)perylene	191-24-2	5
1,4-Dioxane	123-91-2	1

Table A-2.

Method 8270BP Calibration Levels

Calibration Level	Calibration Concentration, µg/mL
1	0.25
2	0.40
3	1.00
4	2.50
5	5.00
6	7.50
7	10.0
8	12.5
9	20.0
10	40.0
SSV	5.0

Table A-3.

Method 8270BP LCS Spike Concentrations

LCS Compounds	Spiking Level, ng/µL in extract
Phenol	10
Bis(2-chloroethyl)ether	10
2-Chlorophenol	10
1,3-Dichlorobenzene	10
1,4-Dichlorobenzene	10
1,2-Dichlorobenzene	10
2,2'-Oxybis(1-chloropropane)	10
N-Nitroso-di-n-propylamine	10
Hexachloroethane	10
Nitrobenzene	10
Isophorone	10
2-Nitrophenol	10
2,4-Dimethylphenol	10
Bis(2-chloroethoxy)methane	10
2,4-Dichlorophenol	10
1,2,4-Trichlorobenzene	10
Naphthalene	10
Hexachlorobutadiene	10
4-Chloro-3-methylphenol	10
Hexachlorocyclopentadiene	10
2,4,6-Trichlorophenol	10
2-Chloronaphthalene	10
Dimethyl phthalate	10
Acenaphthylene	10
Acenaphthene	10
2,4-Dinitrophenol	10
4-Nitrophenol	10
2,4-Dinitrotoluene	10
2,6-Dinitrotoluene	10
Diethylphthalate	10
4-Chlorophenyl phenyl ether	10
Fluorene	10
4,6-Dinitro-2-methylphenol	10
N-Nitrosodiphenylamine	10
4-Bromophenyl phenyl ether	10
Hexachlorobenzene	10
Pentachlorophenol	10
Phenanthrene	10
Anthracene	10
Di-n-butyl phthalate	10
Fluoranthene	10
Benzidine	10
Pyrene	10
Butyl benzyl phthalate	10
3,3'-Dichlorobenzidine	10
Benzo(a)anthracene	10

Table A-3.

Method 8270BP LCS Spike Concentrations (cont.)

LCS Compounds	Spiking Level, ng/µL in extract
Bis(2-ethylhexyl)phthalate	10
Chrysene	10
Di-n-octylphthalate	10
Benzo(b)fluoranthene	10
Benzo(k)fluoranthene	10
Benzo(a)pyrene	10
Indeno(1,2,3-cd)pyrene	10
Dibenz(a,h)anthracene	10
Benzo(g,h,i)perylene	10
N-Nitrosodimethylamine	10
1,4-Dioxane	10

Table A-4.

8270BP Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d_5	5
2-Fluorobiphenyl	5
Terphenyl-d_{14}	5
1,2-Dichlorobenzene-d_4	5
Phenol-d_5	7.5
2-Fluorophenol	7.5
2,4,6-Tribromophenol	7.5
2-Chlorophenol-d_4	7.5

Table A-5.

Suggested Instrument Conditions for 8270BP

Mass Range:	35 - 500 amu
Scan Time:	≤1 second/scan
Initial Column Temperature/Hold Time:	50 °C for 1 minutes
Column Temperature Program:	50 - 320 °C at 35°C/min.
Final Column Temperature/Hold Time:	325 °C/4 min hold
Injector Temperature:	275 °C
Transfer Line Temperature:	290 °C
Source Temperature:	230 °C
Injector:	Single Taper Direct Connect Liner /splitless
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 1.0mL/min.
Column:	DB-5 Capillary 20m x 0.18mm x 0.36 um film thickness

APPENDIX B

Suggested Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

MASS SPECTROMETER Instrument Maintenance Schedule				
Daily (when used)	**Weekly**	**As Needed**	**Quarterly**	**Annually**
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure	Check mass calibration (PFTBA or FC-43).	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.	Check vacuum, relays, gas pressures, and flows.	Replace the exhaust filters on the mechanical rough pump every 1 to 2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.		Change the oil in the mechanical rough pump.
Check inlets, septa.		Clean source, including all ceramics and lenses. Source cleaning is indicated by a variety of symptoms, including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.		Relubricate the turbomolecular pump-bearing wick.
Check baseline level.		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates the need for replacement.		

APPENDIX B

Suggested Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph (cont.)

GAS CHROMATOGRAPH Instrument Maintenance Schedule (*For GC/MS only.*)	
Daily (when used)	***As Needed***
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or guard column or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance indicates it is required (e.g., peak tailing, poor resolution, high backgrounds, etc.).
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Check inlets, septa. Clean injector port.	Replace septa.
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Repair or replace flow controller if constant gas flow cannot be maintained.
	Reactivate flow controller filter dryers when the presence of moisture is suspected.
	Autosampler: Replace syringe, fill wash bottle, dispose of waste bottle contents.

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Title: Mercury in Water by Cold Vapor Atomic Absorption (CVAA) [SW 7470A]

Approvals (Signature/Date):

Doug Gomer 7/10/12 Doug Gomer Date Metals Supervisor	Adam W Alban 11 July 12 Adam Alban Date Health & Safety Manager / Coordinator
7/6/12 John P. Morris Date Quality Assurance Manager	Robert C Hanisch 7/11/12 Robert C. Hanisch Date Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A.

1.2 Method 7470 is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, wipes, TCLP, EP and other leachates/extracts.

1.3 All matrices require sample preparation prior to analysis.

1.4 The reporting limit is 0.0002 mg/L (0.2 μg/L), except for TCLP leachates that have a 0.002 mg/L (2 ug/L) reporting limit.

2.0 Summary of Method

This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3.0 Definitions

3.1 Dissolved Metals: Those elements that pass through a 0.45-μm membrane. (Sample is acidified after filtration).

3.2 Total Metals: The concentration determined on an unfiltered sample following digestion.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Potassium permanganate, which is used to breakdown organic mercury compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.3 Copper also has been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.4 Chlorides can cause a positive interference. Seawaters, brines, and industrial effluents high in chlorides will require dilution. During the oxidation step, chlorides

are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

4.5 Interference from certain volatile organic materials that absorb at the wavelength used for the method may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.6 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.7 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.3 Potassium permanganate is a strong oxidizing agent. It is incompatible

and must be stored separately from hydroxylamine hydrochloride and stannous chloride, the reducing agents used in this procedure, and from acids.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 ppm in Reagent)	Oxidizer Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Digestion Block, with adjustable heating, capable of maintaining a sample temperature of 90-95°C.

6.1.2 Mercury Auto-analyzers:

- CETAC Mercury Analyzer with Autosampler and Auto-Diluter

6.2 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

6.3 Supplies

6.1.3 Disposable 50ml digestion tubes with caps. Accuracy verified to +-3% gravimetrically prior to use.

6.1.4 Disposable glass test tubes, 16 mm x 100 mm

6.1.5 Argon, 99.999% purity

6.1.6 Calibrated automatic pipettes or Class A glass volumetric pipettes (see SOP No. DV-QA-0008 for details on calibrating mechanical pipettes).

6.1.7 Class A volumetric flasks.

6.1.8 Thermometer, non-mercury column, accurate to ±1°C at 95 °C (see SOP No. DV-QA-0001 for calibration details).

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 **Reagent water:** Must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2 **Nitric acid (HNO_3):** concentrated, trace metal grade or better.

7.3 **Hydrochloric acid (HCl):** concentrated, trace metal grade or better.

7.4 Sulfuric acid (H_2SO_4): concentrated, trace metal grade or better.

7.5 Calibration Blank, Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Method Blank (MB), 1% HNO_3:

Add 0.5 L of concentrated HNO_3 to a 50-liter carboy partially filled with reagent water. Dilute to 50 L with reagent water.

7.6 Stannous Chloride Solution, Hg grade, 10% (w/v) per manufacturer's (CETAC) instructions

7.6.1 Place approximately 100 mL of deionized water in a 2-L volumetric flask

7.6.2 Slowly add 200 mL of concentrated HCl to the flask and swirl to mix.

7.6.3 Add 200 grams of $SnCl_2$ to the flask.

7.6.4 Place a large stir bar in the flask and put the flask on a stir plate.

7.6.5 Stir the contents of the flask until the reagent is completely dissolved.

7.6.6 Remove the stir bar and bring to volume with deionized water.

7.7 Sodium chloride-hydroxylamine hydrochloride solution (Hg grade):

Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride (Hg grade) to every 100 mL of reagent water.

NOTE: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.8 Potassium permanganate ($KMnO_4$), 5% solution (w/v):

Dissolve 5 g of potassium permanganate (reagent grade, "suitable for mercury determination") for every 100 mL of reagent water.

7.9 Potassium persulfate ($K_2S_2O_8$), 5% solution (w/v):

Dissolve 5 g of potassium persulfate, reagent grade, for every 100 mL of reagent water.

7.10 Purchased Mercury Stock Solutions

7.10.1 Primary Mercury Calibration Standard Solution, 1,000 mg/L

7.10.2 Second-source Mercury Standard (different vendor than primary calibration standard), 100 mg/L.

7.11 Calibration Working Standard Solution, 10 mg/L.

7.11.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL Class A volumetric flask.

7.11.2 Pipet 1.00 mL of the 1000 mg/L primary mercury calibration standard solution (see Section 7.10.1) into the flask.

7.11.3 Dilute to the mark on the flask with 1% HNO_3.

7.11.4 Stopper the flask and shake to mix.

7.11.5 Transfer the solution to a 125 mL Nalgene bottle.

7.11.6 Document the preparation of the solution in the Standards Log database.

7.11.7 Prepare this solution fresh monthly or more often if necessary.

7.12 Daily Calibration Working Solution, 100 µg/L

7.12.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL Class A volumetric flask.

7.12.2 Pipet 1.00 mL of the 10 mg/L Calibration Working Standard solution (see Section 7.11) into the flask.

7.12.3 Dilute to the mark on the flask with 1% HNO_3 (final volume of 100.0 mL).

7.12.4 Stopper the flask and shake to mix.

7.12.5 Transfer the solution to a 125 mL Nalgene bottle.

7.12.6 Document the preparation of the solution in the Standards Log database.

7.13 Daily Initial Calibration (ICAL)Standards.

7.13.1 To each of six volumetric flasks, add approximately 80 mL of 1% HNO_3.

7.13.2 For each calibration level, add the amount of Daily Calibration Working Solution to the flask as indicated in the following table and bring the solution to a final volume of 100.0 mL. The final concentration for each calibration level is listed in the following table:

Daily ICAL Standards

Calibration Level	Volume of Daily Calibration Working Solution (100 µg/L) (mL)	Final Hg Concentration (µg/L)
1	0.20	0.2
2	0.50	0.5
3	1.0	1.0
4	2.0	2.0
5	5.0	5.0
6	10.0	10.0

7.13.3 Stopper each flask and mix thoroughly.

7.13.4 Document the preparation of the solution in the Standards Log database.

7.13.5 Prepare the calibration solutions each day prior to calibration.

7.14 Continuing Calibration Verification Standard, 5.0 µg/L.

7.14.1 The CCV is prepared exactly as the 5.0 µg/L calibration standard, and from the same source. Refer to Section 7.13.

7.14.2 Prepare sufficient volume of the standard for analysis of a CCV after every 10 samples.

7.15 Second-Source Initial Calibration Verification (ICV) Intermediate Standard, 400 µg/L.

Add 0.4 mL of the 100 mg/L ICV stock standard (see Section 17.10.2) to a 100 mL volumetric flask partially filled with 1%HNO_3 and dilute to the mark. Record this information in the Standards Log database.

7.16 Second-Source Initial Calibration Verification (ICV) Working Standard, 4.00 µg /L.

Add 1.0 mL of the 400 µg/L ICV intermediate standard (see Section 7.15) to a 100 mL volumetric flask partially filled with 1%HNO_3 and dilute to the mark. Record this information in the Standards Log database.

7.17 Laboratory Control Sample (LCS), 5 µg/L

The LCS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Standard to 30 mL of reagent blank in a digestion tube.

7.18 Matrix Spike and Matrix Spike Duplicate (MS/MSD), 5 µg/L

7.18.1 The MS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Solution to a digestion tube containing a second 30-mL aliquot of the selected sample.

7.18.2 The MSD is prepared in the same manner as the MS using a third aliquot of the selected sample.

7.19 Reporting Limit (RL) Check Standard, 0.2 µg/L

The 0.2 µg/L calibration standard is analyzed as a sample to verify the reporting limit. Denoted as RL or RLSTD in the run sequence.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client

requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	HDPE	50 mL	HNO_3, pH < 2	28 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.2.1 Preparation Batch

A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD) pair. As discussed in the following sections, special program or project requirements can include

additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.2.2 Method Blank (MB)

The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. At least one method blank must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than the project-specific data quality objectives. In the absence of project-specific data quality objectives, the blank must be less than ½ the reporting limit or less than 10% of the mercury concentration found in the associated samples, whichever is higher.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If mercury was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.2.3 Laboratory Control Sample (LCS)

The LCS is a blank to which a known concentration of the target analyte has been added. At least one aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure.

Acceptance Criteria: Maximum control limits for LCS recoveries for Method 7470A are 80-120%. In-house control limits based on three standard deviations of the mean of past results are used as long as they are at least as tight as the limits in the methods (see TestAmerica Denver Policy DV-QA-003P for further details on establishing control limits).

Corrective Action: If LCS recoveries are outside established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and mercury is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.2.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is a second aliquot of a selected field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a third aliquot of the same sample (spiked exactly as the MS) prepared and analyzed along with the sample and matrix spike. One MS/MSD pair must be processed for each preparation batch. Some programs may require the use of sample duplicates in place of or in addition to MS/MSDs. In addition, some programs will allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Attachment 1).

Acceptance Criteria: Control limits are statistically determined based on three standard deviations of the mean of the laboratory's historical data. The recoveries for the MS and MSD must fall within 75-125%. The relative percent difference between the MS and MSD cannot exceed 20%.

Corrective Action: If analyte recoveries or the RPD between duplicates fall outside the acceptance range, then LCS recovery must be in control for the data to be reported. If there is no evidence of analytical problems and all other QC criteria are met, then qualified results may be reported and the situation must be described in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC, then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

9.2.5 Serial Dilution

Some programs (e.g., DoD programs) require that a fivefold (1+4) dilution must be included in each analytical batch for each sample matrix.

Acceptance Criteria: The results must be within 10% of the expected value, assuming that the sample concentration is at least 25x the MDL concentration.

Corrective Action: If the control limit is not met, all associated

sample results must be qualified.

9.2.6 Post-Digestion Spikes

Some programs require the inclusion of a post-digestion spike in each analytical batch. The post-digestion spike is prepared by adding 0.25 mL of the 100 μg/L Daily Calibration Working Solution to 6.6 mL of filtered sample digestate. Post-digestion spikes are performed as an additional check for matrix interference.

Acceptance Criteria: The percent recovery limits for the post-digestion spike are 85 to 115%.

Corrective Action: If the acceptance criteria are not met, all associated sample results must be qualified.

9.2.7 Method of Standard Addition (MSA)

The method of standard additions is an option for the analysis of samples shown to have significant matrix effects, e.g., unacceptably low MS/MSD recoveries or under certain conditions for TCLP analysis (see Attachment 5)

9.3 Instrument QC

9.3.1 Initial Calibration (ICAL)

9.3.1.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP No. CA-Q-S-005, *Calibration Curves (General).*

9.3.1.2 Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

9.3.1.3 Calibrate using six standards and a blank. The concentration levels are listed in Attachment 1.

NOTE: It is generally not acceptable to reject calibration points for this method.

9.3.1.4 The calibration curve must have a correlation coefficient of ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

9.3.1.5 Record the number of counts for the 10 ppb standard in the instrument maintenance log.

9.3.2 Initial and Continuing Calibration Blanks

9.3.2.1 An initial calibration blank is tested immediately after the daily

ICAL standards.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Client specific requirements take precedence. For example, DoD requires control of blanks to a concentration less than or equal to the LOD.

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

9.3.2.2 Continuing calibration blanks are run after every 10 samples and at the end of the run.

Acceptance Criteria: The absolute value of the blank result must be less than ½ the reporting limit. Some programs require that blanks be less than 2x the MDL or less than the LOD (refer to special project requirements).

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

9.3.3 Initial Calibration Verification (ICV), 4 µg/L

The accuracy of the calibration standards is verified by testing a second source standard (ICV).

Acceptance Criteria: The ICV result must be within 10% of the true value.

Corrective Action: If the ICV acceptance limit is exceeded, the analysis should be terminated, the accuracy of the calibration standards checked, and the instrument recalibrated.

9.3.4 Reporting Limit Check Standard (RL), 0.2 µg/L

The accuracy of results at the reporting limit is verified by testing a standard in every analytical run that is prepared at the reporting limit concentration.

Acceptance Criteria: The results for this standard must be within 50% of the expected value (20% for USACE and DoD projects).

Corrective Action: If the RL check acceptance limit is exceeded, the analysis should be terminated, the instrument operation checked, and the instrument recalibrated.

9.3.5 Continuing Calibration Verification (CCV), 5.0 µg/L

Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the run. The CCV must be a mid-range standard at a concentration other than that of the ICV.

Acceptance Criteria: The CCV result must fall within 20% of the true value.

Correction Action: Sample results may be reported only when bracketed by valid CCV pairs. If a mid-run CCV fails, the CCV may be re-analyzed once without modification to the instrument's operating conditions. If the re-analyzed CCV is found to be in control, the CCV analysis must be repeated with successful results or the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV failure was not directly instrument related, the associated samples must be re-prepared and reanalyzed.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

10.3.1 All calibration and calibration verification standards (ICV, ICB, CCV,

CCB), as well as the field samples, are processed through the digestion procedure.

10.3.2 Transfer 30.0 mL of well mixed sample and 30.0 mL of each calibration and calibration verification standard to a clean sample digestion tube. The calibration standards are prepared in duplicate to ensure sufficient volume to complete the analytical sequence. Additional aliquots of CCV and CCB solution may have to be prepared for larger sample runs to ensure that CCV and CCB samples bracket every 10 samples in the analytical sequence.

10.3.3 Prepare an MB, LCS, MS, and MSD for each batch.

10.3.3.1 The MB consists of 30.0 mL of 1% HNO_3.

10.3.3.2 The LCS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Solution to 30 mL of 1% HNO_3 in a digestion tube.

10.3.3.3 The MS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Solution to a digestion tube containing a second 30-mL aliquot of the selected sample.

10.3.3.4 The MSD is prepared in the same manner as the MS using a third aliquot of the selected sample.

10.3.4 Add 1.5 mL of concentrated H_2SO_4 and 0.75 mL of concentrated HNO_3 to the samples in the digestion tubes, mixing after each addition.

10.3.5 Add 4.5 mL of 5% potassium permanganate solution to each sample. For samples high in organic materials or chlorides, dilute the sample until the purple color persists for at least 15 minutes.

10.3.6 Add 2.4 mL of potassium persulfate solution, cap the vial, and heat for two hours at 90 - 95°C. Record the start and stop times and the temperature on the bench sheet. Verify that a purple color persists or a black precipitate is present after the two hours of heating. If this is not true, repeat the digestion using a smaller aliquot of sample.

10.3.7 Allow the samples and standards to cool at room temperature.

10.4 Calibration

10.4.1 All calibration standards are digested together with samples, as described in Section 10.3, prior to analysis.

10.4.2 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.5 Sample Analysis

NOTE: Because of differences between various makes and models of CVAA instrumentation, detailed push-button operating instructions are not provided here. Refer to the specific instrument-operating manual for detailed autosampler setup and operation protocols.

NOTE: The injection of samples and the addition of stannous chloride are done automatically by the instrument. Refer to the specific instrument manual for details.

10.5.1 When ready to begin analysis, add 1.8 mL of sodium chloride-hydroxylamine hydrochloride solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains.

10.5.2 Add additional 1% HNO_3 to the samples, QC samples and calibration standards to bring the final volume of each sample to 45 mL.

10.5.3 Aliquot each sample and calibration standard into a disposable test tube for analysis.

10.5.4 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

NOTE: The instrument auto-dilutes samples. Any samples that require greater than a 10x dilutions MUST be diluted manually.

10.5.5 If the sample results are negative and the absolute value is greater than the reporting limit, the sample must be diluted and reanalyzed.

10.5.6 The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

10.5.7 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB. Re-sloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

10.5.8 The following analytical sequence must be used for Method 7470A. Refer to Quality Control Section 9.0 and Attachment 2 for quality control criteria to apply to Method 7470A.

Instrument Calibration
ICV
ICB
RL
Maximum of 10 samples
CCV
CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete the run.
CCV
CCB

NOTE: Samples included in the count between CCVs include the method blank, LCS, MS, MSD, and field samples.

10.5.9 For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

- Recovery of the analyte in the matrix spike is not at least 50%;
- The concentration of the analyte does not exceed the regulatory level; and
- The concentration of the analyte is within 20% of the regulatory level.
- The reporting and matrix spike levels for TCLP analyses are detailed in Attachment 1. Attachment 5 provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

10.5.10 To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.

10.5.11 See Attachment 6 for guidelines for minimizing contamination of samples and standards. See Attachments 5 and 7 for guidance on troubleshooting and preventive maintenance.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate SOP CA-Q-S-005 "Calibration Curves" and under the public folder, Arizona Calibration Training.

11.2 Accuracy

$$\textbf{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\textbf{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.3 Precision (RPD)

$$\textbf{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.4 Concentration = Hg concentration $(\mu g/L) = C \times D$

Where:

C = Concentration (μg/L) from instrument readout
D = Instrument dilution factor

11.5 Appropriate factors must be applied to sample values if dilutions are performed.

11.6 Sample results should be reported with up to three significant figures in accordance with the TestAmerica significant figure policy (DV-QA-004P).

11.7 Documentation and Record Management

The following documentation comprises a completed CVAA raw data package:

- Sample preparation bench sheet(s), to include the batch number, list of samples, preparation analyst and date, instrument analysis analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes). This information is stored in the LIMS.
- Raw data (direct instrument printout).
- Data review checklist - See Attachment 4.
- Standards Documentation to include source, lot, preparation date, and expiration date.
- Nonconformance summary (if applicable).

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Demonstration of Capabilities

12.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

12.2.2 IDOCs and on-going proficiency demonstrations are conducted as follows. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample is typically the LCS spike level. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Aqueous Acidic (Metals) - Corrosive - Waste Stream J

14.2.2 Expired reagents and standards – Contact the Waste Coordinator.

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, March 2005.

15.3 U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 7470A	Chapter 1 of SW846 specifies the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
2	EPA 7470A	This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."
3	EPA 7470A	Methods 7470A and 7471A state that working mercury standards "should be prepared fresh daily." The laboratory frequently prepares up to three batches of mercury samples, including digested calibration standards, each day. The third batch is typically prepared and digested late in the day, and then is analyzed the morning of the next day. The laboratory has developed the following information demonstrating that analysis within 24 hours, but on the second calendar day from preparation produces reliable results and is acceptable to the EPA: • Successful proficiency testing PT results for samples that were prepared and analyzed within 24 hours, but on successive days (e.g., ERA WP-66); • Successful analysis of true NIST mercury standards within every analytical batch; and • A written comment from the EPA MICE Hotline stating that, with the supporting lab data, their opinion was that the laboratory's practice is "within the letter of the method as written."
4	EPA 7470A	Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above ½ the reporting limit.

17.0 Attachments

Figure 1: Aqueous Sample Preparation Flow Chart
Figure 2: CVAA Mercury Analysis Flow Chart
Attachment 1: Mercury Reporting Limits, Calibration Levels, QC Standard and Spiking Levels
Attachment 2: Summary of Quality Control Requirements
Attachment 3: Example Raw Data Checklist
Attachment 4: MSA Guidance
Attachment 5: Troubleshooting Guide
Attachment 6: Contamination Control Guidelines
Attachment 7: Preventative Maintenance

18.0 Revision History

- Revision 1.2 dated July 13, 2012
  - Updated Sections 7.6 and 7.7 to state Hg reagents are used
  - Updated Sections 9.3.2.1 and 9.3.2.2 to control calibration blanks to ½ RL
  - Added Section 9.3.1.5 to record the counts for the 10 ppb high standard
  - Updated Sections 10.5.2 to bring samples to a final volume of 45 mL with 1% HNO_3
  - Formatting and grammatical changes

- Revision 1.1 dated February 03, 2012
  - Annual technical review
  - Added introductory statement to section 7.0 regarding reagent purity
  - Updated Section 9.1.2 and Attachment 2 for Method Blank acceptance criteria
  - Added dilution note to Section 10.3.4
  - Updated section 12.0 to reflect current laboratory practice
  - Removed Leeman instrument and replaced Nitrogen with Argon for Attachment 7

- Revision 1.0 dated 23 August 2011
  - Updated Section 7.15 ICV Intermediate Standard to 400ug/l
  - Updated Section 7.16 ICV Working Standard level to 4ug/l
  - Updated Section 9.2.3 ICV true value to 4ug/l
  - Updated Section 10.3.8 ICV and ICB run order

- Revision 0.5 dated 25 April 2011
  - Removed all references to the FIMS Analyzer
  - Sections 6.1 and 6.3 were updated to reflect the use of digestion blocks from water baths.
  - The reagent amounts were updated to reflect using a 30ml aliquot from 10ml.
  - Section 10.3.2 was updated to show a final volume of 40ml.

- Revision 0.4 dated 07 February 2011
  - Revised section 10 to reflect use of calibrated digestion tubes and calibration standard volumes
  - Revised supplies list
  - Revised section 6.2 to include reference to Master List of Documents, Software and Hardware
  - Added section 11.1 to reference corporate SOP CA-Q-S-005 “Calibration Curves”

- Revision 0.3 dated 01 September 2010
  - Section 7.0: Removed note about standards log with the change in LIMS systems
  - Section 12.2 added section about MDLV verifications
  - Updated Section 11.6 for new LIMS
  - Removed Attachments 3a and 3b
  - Annual Technical Review

- Revision 0.2 dated 07 August 2009
  - Sections 7.17 and 7.18 were updated to use 1% HNO3 from reagent blank.
  - Sections 10.1.3.1 and 10.1.3.2 were updated to use 1% HNO3 from reagent blank.
  - Changed SOP name DV-QA-003P from QC Policy to Quality Assurance Program.

- Revision 0.1, dated 16 February 2008
    - Section 9.1.2: Changed control limit to 10% to match soil SOP
    - Section 9.2.2: Changed the stated control limits for special projects from ½ the RL to 2x the MDL
    - Deleted section 12.2 for IDL requirements
    - Section 12.3: Noted that LCSs will be used for verification

Figure 1.

Aqueous Sample Preparation Flow Chart

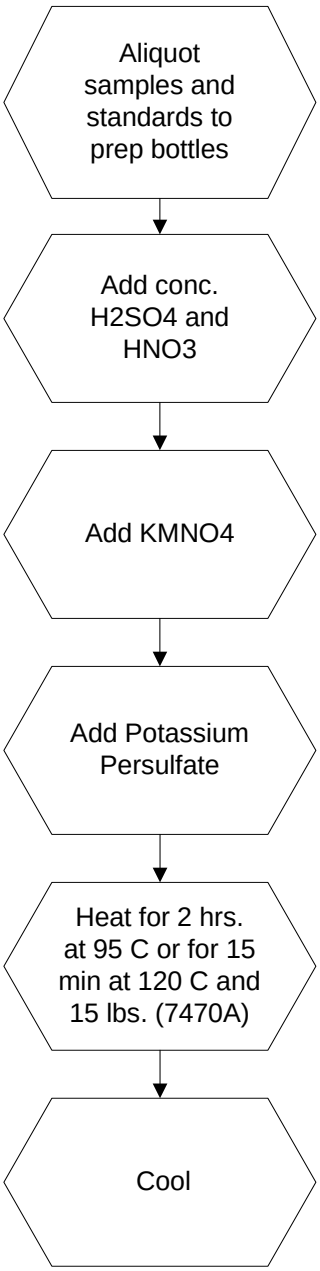
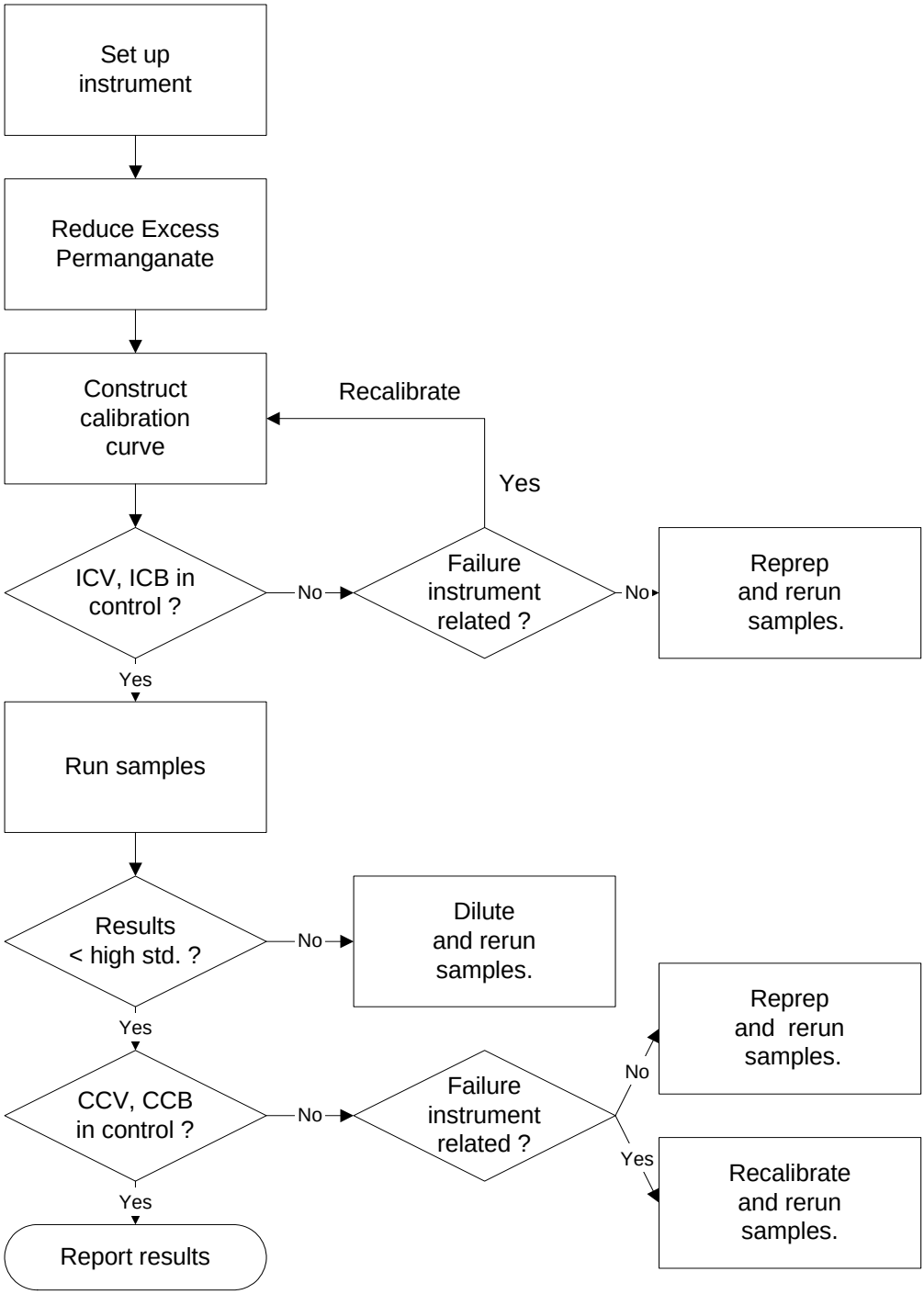


Figure 2.

CVAA Mercury Analysis Flow Chart



Attachment 1.

Mercury Reporting Limits, Calibration Levels, QC Standard and Spiking Levels (µg/L)

Standard Aqueous RL	0.2
TCLP RL	0.2
Std 0	0
Std 1	0.2
Std 2	0.5
Std 3	1.0
Std 4	2.0
Std 5	5.0
Std 6	10.0
ICV	4.0
LCS/CCV	5.0
Aqueous MS	5.0
TCLP MS	5.0

Attachment 2.

Summary of Quality Control Requirements

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
ICV	Beginning of every analytical run.	90 - 110% recovery	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.3.3).
ICB	Beginning of every analytical run, immediately following the ICAL.	Absolute value must be < ½ RL, 2x the MDL for DoD	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.3.2).
CCV	Every 10 samples and at the end of the run.	80 - 120% recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.3.5).
CCB	Immediately following each CCV.	Absolute value must be < ½ RL, 2x the MDL for DoD	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep batch (see Section 9.3.2).
Method Blank	One per sample preparation batch of up to 20 samples.	Project specific or ½ RL Sample results greater than 10x the blank concentration are acceptable.	Re-digest and reanalyze samples. Note exceptions under criteria section. See Section 9.2.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	In-house 3 standard deviation control limits, not to exceed 80-120% recovery.	Terminate analysis; Correct the problem; Re-digest and reanalyze all samples associated with the LCS (see Section 9.2.3).
Matrix Spike	One per 10 samples preparation batch of up to 20 samples.	In-house 3 standard deviation control limits, not to exceed 75-125% recovery	In the absence of client-specific requirements, flag the data (see Section 9.2.4).
Matrix Spike Duplicate	See Matrix Spike	In-house 3 standard deviation control limits, not to exceed 20% RPD	See Corrective Action for Matrix Spike.

Attachment 3.

Example Raw Data Checklist

TESTAMERICA-DENVER

Applicable QC Batches:_____
_____
_____

Industrial Mercury Analysis Raw Data Checklist

Revision 2 -March 5, 2008

Analyst's Checklist

	yes	no	n/a
1. Were the special instructions for prep and/or analysis followed?	☐	☐	☐
2. Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3. Is the blank less than the reporting limit or properly anomalized?	☐	☐	☐
4. Is the LCSs within limits or properly anomalized?	☐	☐	☐
5. Is the ICV and all CCVs within limits?	☐	☐	☐
6. Are all CCBs within ± one reporting limit from zero?	☐	☐	☐
7. Were the CCVs and CCBs run with up to 10 samples between each set?	☐	☐	☐
8. Are the reporting limits correct and reflect any dilutions?	☐	☐	☐
9. Are the number of significant figures correctly reported?	☐	☐	☐
10. Are the benchsheets complete (including calibration and standard verification #'s)?	☐	☐	☐
11. Are all comments, footnotes, and anomalies properly documented?	☐	☐	☐
12. Are holding time violation forms completed and attached?	☐	☐	☐
13. Has all sample data been entered into LIMS?	☐	☐	☐
14. Has all QC data been entered into LIMS?	☐	☐	☐
15. Has the data entered into LIMS been checked for errors?	☐	☐	☐
16. For TCLP results, if the sample data within 20% of Regulatory Level (0.2 mg/L)?	☐	☐	☐

Analyst's Name:_____ Date:_____

Data Review's Checklist

	yes	no	n/a
1. Have the calculations been checked?	☐	☐	☐
2. Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3. Is all the QC data within the control limits and/or properly anomalized?	☐	☐	☐
4. Are all the significant figures and reporting limits correct?	☐	☐	☐
5. Have any comments, footnotes, and anomalies been properly documented?	☐	☐	☐
6. Have any data errors been documented and entered into LIMS?	☐	☐	☐
7. Is prep date correct in LIMS?	☐	☐	☐
8. Has the data package been copied and filed?	☐	☐	☐
9. If TCLP result within 20% of Reg. Level (0.2 mg/l) and MS < 50%, was MSA performed?	☐	☐	☐

Reviewed by:_____ Date:_____

Comments:_____
_____
_____

Anomalies:_____
_____
_____

Attachment 4.

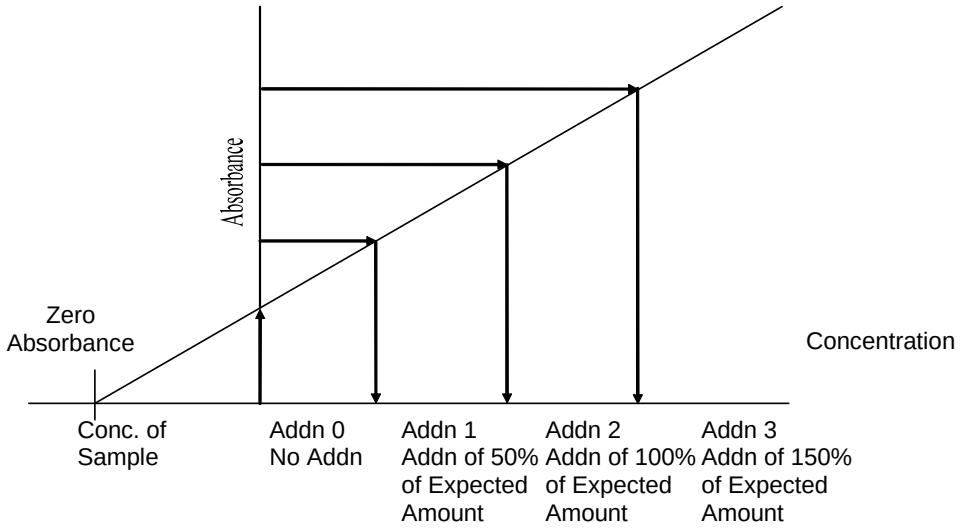
MSA Guidance

Method of Standard Addition (MSA)

Four equal volume aliquots of sample are measured and known amounts of standards are added to three of the aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration, and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of an analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. The absorbance (or response) is plotted on the vertical axis versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. The correlation coefficient (r) and the x-intercept (where y=0) of the curve are calculated. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Attachment 5.

Troubleshooting Guide

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak
Erratic Readings	Source lamp not aligned properly Lamp not pre-warmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

Attachment 6.

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered gloves should not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- If an unusually high sample is analyzed, segregate the glassware and soak with sulfuric acid prior to routine cleaning.

Attachment 7.

Preventative Maintenance

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs, record the date, time, and instrument number; describe the problem; and explain the corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational:

Cold Vapor Atomic Absorption (CETAC Analyzers)

Daily	Monthly	Annually
Change rinse solution.	Check Hg lamp intensity.	Change Hg lamp.
Optimize light path.		Check liquid/gas separator.
Check argon flow.		
Check tubing. Replace as needed.		
Check drain.		
Check condition of dryer		

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ICP Analysis for Trace Elements by SW-846 Method 6010C

Approvals (Signature/Date):

Doug Gomer 7/10/12
Doug Gomer Date
Metals Supervisor

Adam Alban 11 July 12
Adam Alban Date
Health & Safety Manager / Coordinator

John Morris 7/6/12
John Morris Date
Quality Assurance Manager

Robert C. Hanisch 7/11/12
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICPAES). This procedure references Method 6010C for hazardous waste (RCRA) testing.

1.2 The elements that can be determined by this procedure are listed in Attachment 1, together with the routine reporting limits. Additional elements may be analyzed under Method 6010C provided that the method performance criteria presented in Section 12.0 are met.

1.3 The laboratory digests water samples according to SOP DV-IP-0010. The methods require digestion of waters, with the following exceptions, i.e.:

1.3.1 The sample is visibly transparent with a turbidity measurement of 1 NTU or less.

1.3.2 The sample consists of one liquid phase and is free of particulate or suspended matter following acidification.

1.4 Silver concentrations must be below 1.0 mg/L in aqueous sample digestates and 100 mg/kg in solid matrix sample digestates. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data. Samples with silver concentrations exceeding these levels must be re-prepared and reanalyzed using a smaller sample amount.

1.5 The digestion procedure for soil samples is described in SOP DV-IP-0015.

1.6 The digestion procedure for oil samples is described in SOP DV-IP-0017.

1.7 State-specific requirements may take precedence over this SOP for drinking water sample analyses. Review special instructions for each project before starting work.

2.0 Summary of Method

2.1 The laboratory uses simultaneous ICPAES instruments, with both axial and radial viewing configurations. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs.

2.2 Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photo-multiplier tubes or a charge injection device (CID). The photo-currents from the photo-multiplier tubes or a charge injection device (CID) are processed and controlled by a computer system.

2.3 A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The

position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

2.4 Refer to the appropriate SOPs for details on sample preparation methods: DV-IP-0010 for aqueous samples, DV-IP-0015 for soil samples, and DV-IP-0017 for oil samples.

3.0 Definitions

3.1 **Dual View ICP** – an ICP equipped with both radial and axial viewing capabilities.

3.2 **Dissolved Metals** - Those elements which pass through a 0.45-μm membrane. (The sample is acidified after filtration).

3.3 **Potentially Dissolved Metals** - Potentially dissolved metals is the concentration of metals in solution after acidifying the sample with nitric acid to pH <2, holding at room temperature for 8 to 96 hours, and then filtering through a 0.45-μm membrane filter. This definition is based on the Colorado surface water regulations.

3.4 **Suspended Metals** - Those elements which are retained by a 0.45-μm membrane.

3.5 **Total Metals** - The concentration determined on an unfiltered sample following vigorous digestion.

3.6 **Total Recoverable Metals** - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

3.7 **Reporting Limit (RL) -** The lowest concentration to which results are reported without qualification. Details concerning RLs are presented in Policy QA-009.

4.0 Interferences

4.1 Spectral, physical, and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by the following:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.2 A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.3 Spectral Interferences

Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte signal. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections must be applied to the analyte to compensate for the effects of these unwanted emissions.

4.4 Physical Interferences

An internal standard (IS), yttrium or other suitable element, is added to all solutions to correct and monitor physical interferences. Use of a peristaltic pump and the mass flow controller also help to overcome physical interferences. Physical interferences are generally considered to be effects associated with sample transport, nebulization, and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If internal standard recoveries are not acceptable (see Section 9.11), then dilution of the sample may be necessary to overcome the interferences. Where the use of an internal standard might actually degrade the accuracy of the analytical result, sample results may be reported without IS correction.

4.5 Chemical Interferences

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not significant with the ICP technique, but if observed, can be minimized by buffering the sample, matrix matching, or standard addition procedures.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex

gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma. The RF Generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 A Thermo Fischer ICP 6500E Trace Analyzer is currently used. Instruments with demonstrated equivalent performance can also be used

6.1.2 Radio Frequency Generator.

6.1.3 Argon gas supply, welding grade or equivalent.

6.1.4 Coolflow or appropriate water-cooling device.

6.1.5 Peristaltic Pump.

6.1.6 Autosampler.

6.2 Supplies

6.2.1 Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.2.2 Class A volumetric flasks.

6.2.3 Autosampler tubes.

6.2.4 Glass beads, <1 mm diameter, acid washed.

6.3 Computer Software and Hardware

6.3.1 Please refer to the master list of documents and software located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Shelf-Life

7.1.1 Stock standards, standards as received from the vendor, expire on the date assigned by the vendor. If no date is assigned by the vendor, a one-year expiration will be assigned by the laboratory.

7.1.2 The expiration date of intermediate concentration standards or working standards cannot be later than the date assigned to any of the stock standards used to prepare the intermediate solution.

7.1.3 If visible deterioration is noted for any standard, it must be re-verified against a second-source. Any standard that does not verify must be replaced immediately.

7.2 Standards

7.2.1 Standards used for calibration and quality control purposes must be NIST traceable, where available. Multi-component custom blend standards must be verified against a second-source standard before they are put into use (the only exception is standards purchased directly from NIST), as described in SOP DV-QA-0015.

7.2.2 Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon, polyethylene, or polypropylene bottles. Silver standards must be protected from light. The preparation frequency is governed by the parent standard with the earliest expiration date unless specified otherwise in this SOP. Detailed instructions regarding the preparation of standards and reagents are given in this section. Alternate procedures are allowed as necessary to accommodate volume requirements as long as final concentrations are maintained and an accurate description of the standard or reagent used is entered into the Standards Log database.

7.2.3 Calibration and QC standards are prepared in water with hydrochloric and nitric acids in order to approximate the acidic matrix of the various digests analyzed. This is an important point. Even with the use of yttrium as an internal standard, deviations from these concentrations can cause physical effects, as discussed in Section 4.4 of this procedure.

7.3 Reagent Blank / Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

7.3.1 Fill a 20-liter carboy with about 18 liters of reagent water. Slowly add the appropriate amount of concentrated HNO_3 and concentrated HCl. Mix carefully.

7.4 Stock ICSA and ICSAB Standards

The following standards are purchased from commercial sources:

Stock ICSA & ICSAB Standard	Elements	Concentration (mg/L)
ICSA Std	Fe Al, Ca, Mg	2,000 5,000
ICSAB Std	Ba, Be, Co, Cr, Cu, Mn, V Ag, Cd, Ni, Pb, Zn	50 100
ICSAB 1	Li, Mo, Sb, Sr As, B, P Se	100 200 500

Stock ICSA & ICSAB Standard	Elements	Concentration (mg/L)
	K, Na	5000
ICSAB 1B	Tl	1,000
ICSAB 2	Ti Sn	100 1,000
10,000 Si	Si	10,000
Th	Th	1,000
Zr	Zr	1,000
S	S	1,000
Bi	Bi	1,000

7.5 ICSA Working Standard

A combined working ICSA standard is made in a 250-mL volumetric flask using the following volumes of the Stock ICSA and ICSAB Standards:

Stock Standard	Volume of Stock Added (mL)
ICSA Std	25

Adjust to volume (250 mL) using the reagent blank solution. This produces the final ICSA standard concentrations shown in Attachment 5.

7.6 ICSAB Working Standard

A combined working ICSAB standard is made in a 250-mL volumetric flask using the following volumes of the Stock ICSAB Standards:

Combined Working ICSAB Standard

Stock Standard	Volume of Stock Added (mL)
ICSA Std	25
ICSAB Std	2.5
ICSAB 1	2.5
ICSAB 1B	2.5
ICSAB 2	2.5
10,000 mg/L Si	0.25
1,000 mg/L S	0.25
1,000 mg/L Th	0.5
1,000 mg/L Zr	0.25
1,000 mg/l Bi	0.25

Adjust to volume (250 mL) using the reagent blank solution. This produces the final ICSAB standard concentrations shown in Attachment 5.

7.7 High Calibration Check Standard

The high concentration check standard is the same as the Working ICAL Standards.

7.8 Laboratory Control Sample (LCS) Stock Standards

The LCS stock standards are purchased from commercial sources. The stocks are custom-made standards purchased at ready-to-use concentrations as follows:

LCS STOCK STANDARD	ELEMENTS	CONCENTRATION (MG/L)
ICP Prep Spike #1	Ca, K, Mg, Na P Al, As, Ba, Se, Th, Tl, Bi Fe, Sr Co, Mn, Ni, Pb, V, Zn Cu Cr Ag, Be, Cd	5,000 1,000 200 100 50 25 20 5
ICP Prep Spike #2	Sb, Zr B, Mo, Ti Sn Si (SiO_2)	50 100 200 1000 (2140)
Sulfur	S	200

Soil Batches – LCS spikes for soil batches are prepared by adding 1.0 mL of the LCS to a digestion tube containing 5 mL of reagent water. The AFCEE and DOD programs require the addition of 1 g of glass beads to the digestion tube.

Water Batches – LCS spikes for water batches are prepared by adding 0.5 mL of LCS Stock Standard to a digestion tube containing 50 mL of reagent water.

7.9 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

The same LCS stock standards described in the previous section, 7.8, are also used to prepare matrix spikes. The same media and spike concentrations are used as well.

7.10 Post Digestion Spike (PDS) Standards (Analyte Addition Spike Standards)

The custom standards tabulated below are purchased from a commercial source. Add 0.08 mL of each to 8 mL (100X) of digestate or dilution of digestate.

PDS Stock	Elements	Conc. (mg/L)
PDS 1	Ag, Be, Cd, Co, Cr, Cu, Mn, Ni, Sr, V	5.0 10

PDS Stock	Elements	Conc. (mg/L)
	Ba, Pb, Li As, Se, Tl, Zn, Th U Al, Fe P Ca, Mg, Na, K	20 50 100 200 2,000
PDS 2	Mo, Ti, Zr B, Sb, Sn Si	5.0 10 500

7.11 Initial Calibration (ICAL) Standards for the Dual View ICP

7.11.1 Stock Calibration Standards

The following stock solutions are purchased from commercial sources.

Stock Standard	Elements	Conc. (mg/L)
STLDEN-STD-2	Mo, Ti, Zr Sn Si	100 200 1,000
STLDEN-STD-3B	Ag, Al, B, Ba, Cd, Co, Cr, Cu, Be, Mn, Ni, Sr, V, Zn Li, P Ca, Na Mg K Fe	100 200 1,000 4,000 10,000 500
Al, Ca, Fe, Na, S, Th Stocks	Al, Ca, Fe, Na, S, Th	10,000
As, Bi, Pb, Sb, Se, Tl, U, Stocks	As, Bi, Pb, Sb, Se, Tl, U	1,000

7.11.2 Working Initial Calibration Standard (ICAL1) for Dual View ICP

Add 5.0 mL each of STLDEN-STD-2 and STLDEN-STD-3B to a 500-mL volumetric flask partially filled with reagent blank solution. Add 1 mL of the As, Pb, Sb, Se, Tl, stock. Dilute to the mark with reagent blank.

7.11.3 Working Initial Calibration Standard (ICAL2a) for Dual View ICP

Add 10 mL of each of the Al, Fe, and 50 mL Na 10,000 mg/L stock solutions; 1 mL each of the Th and 20 mL of the U 1,000 mg/L stock solution; 2ml of 1000mg/l Bi and1 mL of 10,000 mg/L Sulfur to a 1,000-mL volumetric flask partially filled with reagent blank and dilute to the mark with reagent blank.

7.12 Initial Calibration Verification (ICV) for Dual View ICP

7.12.1 ICV Stock Standards

The following stock solutions are purchased from commercial sources:

Stock Standard	Elements	Conc. (mg/L)
ICVL SOL A	Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Li, Mn, Ni, Pb, Sr, V, Zn Se, Tl Ca, Na Mg K	25 25 50 200 1,000 2,000
ICVL SOL B	Ag, Mo, Sb, Ti, Zr Sn Si P	25 50 200 200
ICVH Stock	Fe Al, Ca, Na U Th Zn	8,000 4,000 500 300 250
Sulfur	S	1000
Bismuth	Bi	1000

7.12.2 Working High Initial Calibration Verification (ICVH)

Add 2.0 mL of the ICVH Stock, 0.1ml of 1000mg/l Bi and 0.8 mL of the Sulfur 1000 mg/L to a 200 mL volumetric flask partially filled with reagent blank solution and dilute to the mark.

7.12.3 Working Low Initial Calibration Verification (ICVL)

Add 2.0 mL of each of the ICVL SOL A and ICVL SOL B stock solutions to a 200-mL volumetric flask partially filled with reagent blank solution and dilute to the mark.

7.13 Reporting Limit Standard (RLSTD)

7.13.1 RL Stock Standard

The following stock solutions are purchased from commercial sources:

Standard	Elements	Conc. (mg/L)
STLDEN-RL-1A	As, Sb, Se, Tl Pb	10 3.0
STLDEN-RL-2	Si Sn Mo, Ti, Zr	500 20 10
STLDEN-RL-3	Ag, Cr, Cu, Ni, Th, V, Zn, Li Al, B Ba, Cd, Co, Sr Be Ca, Mg Fe K, Na, P Mn U	10 100 5.0 1.0 200 30 1,000 3.0 60
Sulfur Bismuth	S Bi	100 100

7.13.2 Daily Working Reporting Limit Standard (RLSTD2 or RLSTD3)

Add 100 µL of each of STLDEN-RL-1A (optional for RLSTD2), STLDEN-RL-2, STLDEN-RL-3, Bismuth 100 mg/l and Sulfur 100 mg/L to a 100-mL volumetric flask partially filled with reagent blank and dilute to the mark. Working RL standards must be prepared fresh each day.

7.14 Working High Continuing Calibration Verification (CCVH1) for Dual View ICP

Perform a 2X dilution of the working ICAL2 solution (section 7.11.3) with reagent blank solution.

7.15 Working Low Continuing Calibration Verification (CCVL1) for Dual View ICP

Perform a 2X dilution of the working ICAL1 solution (section 7.11.2) with reagent blank solution.

7.16 Low Level ICV/Low Level CCV for Dual View ICP

The low level ICV/CCV verification stock standards are custom-made commercial standards as follows:

LLICV/LLCCV Stock Standard	Elements	Conc. (mg/L)
LLICV/LLCCV-1	K	300
	Na	100
	Ca, Mg	20

LLICV/LLCCV Stock Standard	Elements	Conc. (mg/L)
	Al, Bi, Fe	10
	U	6
	Ni	4
	Zn	2
	As, Cu, Se,Tl,Th	1.5
	Ba, Cr, Co, Li, Mn, Ag, Sr, V	1
	Pb	0.9
	Cd	0.5
	Be	0.1
LLICV/LLCCV-2	P	300
	Si	50
	B	10
	Sn	10
	Mo	2
	Zr	1.5
	Sb	1
	Ti	1

7.16.1 Low Level ICV \ Low Level CCV, Working Standards

RL Standard	Vol. of Stock Added (mL)
ICP-LLCCV1	5
ICP-LLCCV-2	5

Adjust to volume (500 mL) using the reagent blank solution.

7.17 Reagents

7.17.1 Concentrated nitric acid (HNO_3), trace metals grade or better.

7.17.2 Concentrated hydrochloric acid (HCl), trace metals grade or better.

7.17.3 Reagent water must be produced by a Millipore DI system or equivalent, with a minimum resistivity of 1.0 Mohm/cm at 25˚C.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50 mLs	HNO_3, pH < 2; Cool ≤ 6°C	180 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool ≤ 6°C [2]	180 Days	N/A

[1] Inclusive of digestion and analysis.

The exception is the analysis of dissolved silica by Method 200.7, which must be analyzed within 28 days from the date of collection.

Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

[2] Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration. Samples which will be used to aliquot for both analyses must be refrigerated.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the

laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See Policy DV-QA-003P for further details.

9.3 Method Blank

The blank is de-ionized water taken through the procedure as if it were a sample. For soil samples analyzed under the AFCEE and DoD QAPPs, the method blank consists of <1 mm glass beads that have been processed in the same manner as the samples.A method blank is required with every batch of 20 or less samples.

Acceptance Criteria: The method blank must not contain any analyte of interest above ½ the reporting limit or above one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL).

Corrective Action: If the method blank exceeds allowable levels, all associated samples must be redigested and reanalyzed. A possible exception is the situation in which the analyte is not detected in any of the associated samples, but this can only be done with client approval and it must be addressed in the final report case narrative.

9.4 Laboratory Control Sample (LCS)

The LCS is prepared as described in Section 7.8. One LCS is required with each analytical batch.

Acceptance Criteria: The recovery of the LCS must be within historical control limits. Historical control limits are based on three standard deviations of past results, and must be 80-120% or tighter. In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the report narrative. The process of establishing control limits is described in more detail in the Policy DV-QA-003P. The control limits are stored in the lab's LIMS system.

Corrective Action: If the LCS recovery falls outside of the established limits, all associated samples must be redigested and reanalyzed

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

9.5.1 MS/MSDs are prepared as described in Section 7.9. One MS/MSD pair is required with each analytical batch. Note that some programs (e.g., North Carolina and South Carolina) require the MS/MSDs to be run at a 10% frequency. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing on only the specific sample spiked.

9.5.2 Samples identified as field blanks cannot be used for MS/MSD analysis. Note that if client instructions on the chain of custody form tell the lab to use a field blank for the MS/MSD, this should be double-checked with the laboratory PM.

Acceptance Criteria: The recoveries for the MS and MSD must be within the historical control limits or the project-required control limits, whichever are appropriate. Historical control limits are based on three standard deviations of past results, and should be within the established project-specific method control limits, if they exist. The process of establishing control limits is described in more detail in Policy DV-QA-003P. The control limits are stored in the laboratory's LIMS system. Acceptance limits derived from historical data should be no wider than +/-25%.

Corrective Action: If MS/MSD recoveries fall outside of the established limits and the LCS is in control, the data will be flagged as outside of control limits. Document the results, which are then used by the lab PM to prepare the case narrative to warn the client that the sample result is suspect.

Acceptance Criteria: The relative percent difference (RPD) between the MS and MSD is evaluated to measure precision and must be less than or equal to the historical RPD control limit. Historical control limits are based on three standard deviations of past results, and must be no greater than 20%.

Corrective Action: If the RPD fails to meet precision limit and the recoveries pass, the control limits should be checked as this would be a very rare occurrence if the limits are set properly. If the LCS is in control, it indicates long-term precision, and precision failures within the batch may be due to sample

non-homogeneity. MS/MSD results which fall established control limits must be addressed in the narrative. Document the result, which is then used by the lab PM to prepare the case narrative.

9.6 Serial Dilution Test

A dilution test is performed for each batch of samples. The purpose of this test is to ensure that neither positive or negative interferences are biasing the analytical results. The serial dilution test should be performed on the same sample used to perform the MS/MSD.

Acceptance Criteria: If the analyte concentration is sufficiently high (minimally, a factor of 10 times the lower limit of quantitation after dilution), an analysis of a 1:5 dilution (e.g., 1 mL of sample diluted to 5 mL with reagent blank solution) must agree within ± 10% of the original determination.

Corrective Action: If the two results do not agree within ± 10%, then a chemical or physical interference is suspected. A qualifier flag is assigned to the data and an NCM prepared, which is then used by the lab PM to prepare the case narrative to warn the client the sample result is suspect.

9.7 Post Digestion Spike (PDS)

Whenever the MS/MSD recoveries are unacceptable, a PDS spike must be performed. The PDS spike is prepared as described in Section 7.10. Some programs, e.g., AFCEE, require a PDS analysis whenever the serial dilution test fails. Other programs, e.g., DoDQSM, require a PDS to be included in every batch. Check project requirements. For these programs, the same sample that was used for the serial dilution test should be used for the PDS.

Acceptance Criteria: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 80-120% for Method 6010C. The spike addition should produce a minimum level of 10 times to a maximum of 100 times the lower limit of quantitation.

Corrective Action: If the spike is not recovered within the specified limits, a matrix effect is confirmed. The series of tests (MS/MSD, serial dilution, and PDS) should be described in NCMs so that they can be included in the report case narrative.

9.8 Method of Standard Additions (MSA)

This technique involves constructing a calibration curve in the sample matrix itself to compensate for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift.

Attachment 11 provides more guidance on performing MSA analyses.

9.9 Interference Check Analysis (ICSA / ICSAB)

The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Sections 7.4, 7.5, and 7.6 for the preparation of the ICSA and ICSAB solutions. Attachment 4 lists the final concentrations. All analytes are spiked into the ICSAB solution. The ICSA and ICSAB solutions are analyzed at the beginning of the run.

Acceptance Criteria: The ICSAB results for the all analytes must fall within 80-120% of the true value. If any ICSAB analyte result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the samples rerun.

The absolute value of ICSA results for the non-interfering elements must be $\leq$ 2 x RL. The DoD and AFCEE programs have their own criteria based on the version used.

Corrective action: If the ICSA results for the non-interfering elements do not meet these limits, the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
- If the affected element was not required, then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration which would result in an absolute value > 2 x RL, then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal > 2x RL, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.
- If the data do not meet the above conditions, then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually, the calculations must be clearly documented on the raw data.

9.10 Monitoring Internal Standard Results

Yttrium is automatically added as an internal standard (IS) to every solution tested through use of a third pump channel and mixing coil. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

Acceptance Criteria: If the internal standard counts fall within ±30% of the counts observed in the ICAL blank, then the data are acceptable.

Corrective Action: If the internal standard counts in the field samples are outside of the control limits, the following apply:

- The field samples must be diluted and reanalyzed;
- The IS concentrations must be raised; or
- A different internal standard must be used.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

Solid and aqueous samples must be digested prior to analysis by the appropriate method (see SOPs DV-*IP-0010, DV-IP-0015, and DV-IP-0017*).

10.4 Calibration

10.4.1 Instrument Start Up

10.4.1.1 Set up the instrument with the operating parameters recommended by the manufacturer. Complete any required preventative maintenance and record in the ICPAES Preventative Maintenance Log. Preventive maintenance recommendations a list in the TestAmerica Denver Quality Assurance Manual.

10.4.1.2 Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required)

10.4.2 Initial Calibration (ICAL)

10.4.2.1 The calibration curve is established on each day of operation using a blank and one standard. The preparation of the ICAL standards is described in Section 7. The final concentrations of the ICAL standards are presented in Attachment 6.

10.4.2.2 The validity of the calibration curve is confirmed by analysis of the ICV, CCV, ICB, RL Check standard and Low Level ICV/CCV) which are run immediately after the ICAL. Some programs require a high-level verification check as well.

10.4.3 Initial Calibration Verification (ICV)

Calibration accuracy is verified using a second-source standard (ICV) that is at or below a concentration near the mid-point of the working range. The ICV is analyzed immediately after the ICAL. The preparation of this standard is described in Section 7. The concentrations of the ICV standard are presented in Attachment 6.

Acceptance Criteria: For Method 6010, the ICV result must fall within 10% of the true value for that solution. The standard deviation must be <5% (the laboratory is using at least two exposures for all ICP analyses).

Corrective Action: If the ICV fails to meet acceptance limits, the standard may be reanalyzed without modification to the instrument operating conditions. Two consecutive, acceptable analyses are required before the analytical run may continue. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.4 Mid Level Continuing Calibration Verification (CCV)

The preparation of the CCV solutions are described in Section 7. The final concentrations of the CCVs are presented in Attachment 6. Note that the CCV is made at a different concentration than the ICV to meet NELAC requirements. CCVs are analyzed after the ICV, after every ten samples, and at the end of the analytical run.

Acceptance Criteria: The CCV must be within 10% of the expected value to meet Method 6010 requirements. The relative standard deviation must be <5%.

Corrective Action: If the CCV fails to meet any of these criteria, the standard may be reanalyzed without modification

to the instrument operating conditions. *Two consecutive, acceptable analyses are required before the analytical run may continue.* Otherwise, the instrument must be recalibrated and the samples reanalyzed since the last successful CCV must be reanalyzed.

10.4.5 Low Level Initial Calibration (LLICV) and Continuing Calibration Verification (LLCCV)

The preparation of the LLCCV solution is described in Section 7. The low-level CCV needs to be analyzed at the beginning and end of every run sequence. If low level samples are expected then the low-level CCV should also be run every ten samples.

Acceptance Criteria: The LLCCV must be within +/-30% of the expected value to meet Method 6010C requirements.

Corrective Action: If the CCV fails to meet any of these criteria, the standard may be reanalyzed without modification to the instrument operating conditions.

Two consecutive, acceptable analyses are required before the analytical run may continue. If the calibration cannot be verified within theses specified limits, the analysis of samples containing the affected analytes at similar concentrations cannot continue until the cause is determined and the LLCCV standard successfully analyzed. Otherwise, the instrument must be recalibrated and the samples reanalyzed since the last successful CCV must be reanalyzed. TestAmerica will not hold samples with concentrations greater than 10x the reporting limit to the 30% acceptance criteria.

10.4.6 Initial Calibration Blank (ICB)

System cleanliness is verified by analyzing an ICB after the first CCV. The preparation of the ICB is described in Section 7.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as sodium must be less than the RL. Client specific requirements take precedence. For example, DoD requires control of blanks to a concentration less than or equal to the LOD.

Corrective Action: If the ICB fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem

corrected, the instrument recalibrated, and the calibration re-verified.

10.4.7 Calibration Check Standard (RLSTD)

Calibration accuracy at the RL is verified by analyzing a standard prepared at a concentration at or below the laboratory's standard reporting limit. The preparation of this standard is described in Section 7. Alternate RLSTD concentrations may be used as necessary to meet client requirements as long as an accurate description of the standard used is entered into the Standards Log database.

Acceptance Criteria: For routine work and for programs that allow the RL to be as low as 2 x MDL (e.g., AFCEE), the acceptance limits are ± 50% of the expected value. For some programs (e.g., DoDQSM), the RLSTD needs to be 5 x MDL, and the acceptance limit is then ± 20%.

Corrective Action: If the RL Check standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.8 Lower Limit of Quantitation Check (LLQC)

The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits and on an as needed basis to demonstrate the desired detection capability. The difference between the LLQC and the LLICV/CCV is that this standard is carried through the entire preparation and analytical procedure.

Acceptance Criteria: LLQC is verified when all analytes are detected within ± 30% of their true value.

Corrective Action: If the LLQC fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.9 High-Level Calibration Check Standard

The method 6010 defines the linear working range used for daily analysis based on the LDR studies performed every six months, in which case this standard is not required. However, some programs require verification of the high end of the linear range at different frequencies. For example, the

AFCEE QAPP, version 4.0, requires evaluation of a high check standard every three months.

Acceptance Criteria: The result for this standard must be within 10% of the expected value.

Corrective Action: If the High-Level Calibration Check standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified. Alternately, results that do not exceed the level of the highest calibration standard may be accepted and reported.

10.4.10 Continuing Calibration Blank (CCB)

CCBs, prepared as in Section 7.3, are analyzed after each CCV.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as sodium must be less than the RL. Client specific requirements take precedence. For example, DoD requires control of blanks to a concentration less than or equal to the LOD.

Corrective Action: If the CCB is greater than these limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, instrument maintenance should be considered, the calibration re-verified, and all samples analyzed since the last successful CCB must be reanalyzed.

10.5 Sample Analysis

10.5.1 Replicate Readings

The laboratory averages the results from two exposures for Axial and Dual View ICP for each standard, field sample, and QC sample due to sample volume limitations of the autosampler tube.

10.5.2 Rinse Time Between Samples

Prior to calibration and between each sample/standard, the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 12.7 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

10.5.3 The following analytical sequence is used:

Instrument Calibration
High Standard Verification
ICV
LLICV
CCV
ICB
RL Verification Standard
LLQC (as needed)
ICSA
ICSAB
LRA
CCV
CCB
LLCCV
10 samples
CCV
CCB
LLCCV
10 samples
CCV
CCB
LLCCV
Repeat sequence with 10 samples between CCV/CCB pairs
CCV
CCB
LLCCV

10.5.4 Full method-required QC must be available for each wavelength used in determining reported analyte results. Guidelines are provided in the appendices for minimizing contamination of samples and standards (Attachment 11) and troubleshooting (Attachment 10).

10.5.5 Dilutions for High Levels of Elements of Interest

For 6010, results must fall within the linear range. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. Dilutions must be prepared using the reagent blank solution to maintain the correct acid strength.

10.5.6 Dilutions for High Levels of Interfering Elements

Dilutions are also required for an element that is included in an IEC calculation if it exceeds the linear range. If a dilution is not performed, the IEC may be inaccurately applied. Therefore, even if an over-range analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted to a level at or below the working range. An NCM will be written in these instances.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate SOP CA-Q-S-005 "Calibration Curves" and under the public folder, Arizona Calibration Training.

11.2 ICV percent recoveries are calculated according to the following equation:

$$\%\mathrm{R} = \left(\frac{\text{ICV Found Value}}{\text{ICV True Value}} \right) \times 100\%$$

11.3 CCV percent recoveries are calculated according to the following equation:

$$\%\mathrm{R} = \left(\frac{\text{CCV Found Value}}{\text{CCV True Value}} \right) \times 100\%$$

11.4 Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \left(\frac{SSR - SR}{SA} \right) \times 100\%$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

11.5 The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equation:

$$RPD = \left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2} \right)} \right] \times 100$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

11.6 The final concentration for a digested aqueous sample is calculated as follows:

$$\text{Final Concentration (mg/L)} = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

11.7 The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$\text{Final Concentration (mg/kg), dry weight} = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

NOTE: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

11.8 The LCS percent recovery is calculated according to the following equation:

$$\%R = \left(\frac{\text{LCS Found Value}}{\text{LCS True Value}} \right) \times 100\%$$

11.9 The IEC's are calculated according to the following equation:

$$IEC = \left(\frac{\text{observed concentration}}{\text{observed concentration of the interfering element}} \right)$$

11.10 The dilution test percent difference for each component is calculated as follows:

$$\%\,Difference = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

11.11 Appropriate factors must be applied to sample values if dilutions are performed.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy DV-QA-005P. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD, AFCEE, and DOE projects, an MDL verification is performed quarterly.

12.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard prepared at 1-2 times the calculated MDL concentration. An MDLV standard is analyzed immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

Acceptance Criteria: The calculated MDL is verified if the MDLV standard is detected and the result is significantly different than the blank.

Corrective Actions: If the first MDLV is not detected, the MDLV standard will be re-prepared and analyzed at twice the original concentration. The lowest concentration that produces a detectable signal will then be reported as the MDL.

12.3 Instrument Detection Limit Study

12.3.1 Instrument detection limit (IDL) studies are conducted quarterly for each instrument and each wavelength used for analysis.

12.3.2 Run seven blanks on three non-consecutive days.

12.3.3 Calculate the standard deviation for each day. The final IDL concentration is the average of the three daily standard deviation values.

12.3.4 See Policy DV-QA-014P for a discussion of IDL studies and evaluation of IDL results.

12.4 Linear Dynamic Range (LDR)

12.4.1 The LDR must be determined initially (i.e., at initial setup) and then every three months for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample.

12.4.2 The LDR must be determined from a linear calibration prepared in the normal manner using the normal operating procedures described in Sections 10 and 11.

12.4.3 The LDR is determined by analyzing successively higher standard concentrations of the analyte. A minimum of three standards is required for the initial and on-going studies, and one of the levels must be close to the upper end of the range. The highest concentration must be within 10% of the stated concentration.

12.4.4 The highest standard that meets this criterion defines the maximum concentration that can be reported for sample analysis without dilutions.

12.4.5 If the instrument is adjusted in any way that may affect the LDRs, new dynamic ranges must be determined. The LDR data must be documented and kept on file.

12.5 Background Correction Points

12.5.1 To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength of interest and record the apparent emission intensity from all other method analytes. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations.

12.5.2 Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

12.6 Interelement Corrections (IECs)

12.6.1 ICP interelement correction (IEC) factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be re-determined.

12.6.2 When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC, then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs.

12.6.3 Refer to the facility-specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which produces a false analytical result with an absolute value greater than the RLs shown in Attachment 1. Note that the USACE program requires a control limit of 2|MDL|, which is feasible when verified MDLs are used.

12.6.4 To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

12.6.5 Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace and CID detector instruments as reflected by the ICSA response.

12.7 Rinse Time Determination

12.7.1 Rinse times must be determined annually.

12.7.2 To determine the appropriate rinse time for a particular ICP system, a standard containing the highest concentration level that would be reported for samples is aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system.

12.7.3 For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level).

12.7.4 Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

12.8 Demonstration of Capabilities

12.8.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

12.8.2 Four aliquots of the ICV are analyzed using the same instrumental conditions and procedures used to analyze samples. The analyst must employ ICV's from four distinct analytical sequences. Using these four ICV's demonstrates the analyst's ability to optimize and calibrate the instrument and to prepare analytical solutions. Calculate the mean recovery and standard deviation of the mean recovery for each analyte of interest.

12.8.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.8.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.8.5 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

12.9 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the

required experience. Further details concerning the training program are described in SOP DV-QA-0024.

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Safety Manual, and HS-001, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Acid solutions from ICP drain - Waste Stream J

14.2.2 Metals waste potentially contaminated with Cat 1 radioactive materials – Waste Stream RJ

Note: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 6010C, Revision 3, Update IV, February 2007.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 6010C	This procedure uses mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.
2	EPA 6010C	The alternate run sequence presented in Section 10.5.3 is consistent with method requirements. Additional QC (i.e., ICSA) analyses were added to accommodate the CLP protocol requirements.

Item	Method	Modification
3	EPA 6010C	Method 6010 states that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific "concentration range around the calibration blank." Because of the lack of definition for "concentration range around the calibration blank," the laboratory has adopted the procedure in EPA CLP ILMO4.0 for determining IECs,
4	EPA 6010C	Section 9.9 of Method 6010C states: "If less than acceptable accuracy and precision data are generated, additional quality control tests are recommended prior to reporting concentration data for the elements in this method." The dilution test helps determine if a chemical or physical interference exists. Because the laboratory sometimes does not have prior knowledge if the MS/MSD will be within criteria, the analyst may select to perform a dilution test on one sample in each preparation batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. In this procedure, matrix interference is determined by evaluating data for the LCS, MS/MSD, and serial dilutions. The laboratory must request documented, clear guidance when a unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.0 Attachments

Attachment 1 Metals Analyzed by ICP and Reporting Limits
Attachment 2 Matrix Spike and Aqueous Laboratory Control Sample Levels
Attachment 3 Low Level ICV and CCV Spiking Levels
Attachment 4 Interference Check Sample Concentrations
Attachment 5 TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels
Attachment 6 6500 Initial Calibration &Continuing Calibration Verification Standards
Attachment 7 Summary of Quality Control Requirements
Attachment 8 ICP Data Review Checklist
Attachment 9 MSA Guidance
Attachment 10 Troubleshooting Guide
Attachment 11 Contamination Controls

18.0 Revision History

Revision 0.3, dated 13 July 2012
- Annual Review
- Clarified soil preservation for ICP only analysis, Section 8
- Updated section 9.1, 10.1, 10.2, and 12.1 to reflect current practice
- Updated sections 10.4.6 and 10.4.10 to control calibration blanks to ½ the RL

Revision 0.2, dated 30 June 2011
- Added reference to DV-IP-0017 "Microwave Digestion" throughout document
- Added section 6.3 "Computer Software and Hardware"

- Removed Uranium from the ICSA/ICSAB tables in sections 7.4, 7.5, and 7.6
- Updated sections 7.14 and 7.15 to reflect current practices
- Updated the Acceptance Criteria in sections 9.4, 9.6, and 9.10
- Referenced the TestAmerica Denver Quality Assurance Manual in section 10.4.1
- Updated section 11 to reference corporate SOP CA-Q-S-005, “Calibration Curves” and Arizona Calibration Training spreadsheet
- Added IEC calculation to section 11

Revision 0.1, dated 18 June 2010

- Basic Annual Review

Revision 0, dated 19 June 2009

Attachment 1

Metals Analyzed by ICP and Reporting Limits

ELEMENT	Symbol	CAS #	6010 Analyte	Reporting Limit (µg/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	100	10
Antimony[trace]	Sb	7440-36-0	X	10	1
Arsenic[trace]	As	7440-38-2	X	15	1
Barium	Ba	7440-39-3	X	10	1
Beryllium	Be	7440-41-7	X	1	0.1
Bismuth	Bi	7440-69-9		100	10
Boron	B	7440-42-8	X	100	10
Cadmium[trace]	Cd	7440-43-9	X	5	0.5
Calcium	Ca	7440-70-2	X	200	20
Chromium	Cr	7440-47-3	X	10	1
Cobalt	Co	7440-48-4	X	10	1
Copper	Cu	7440-50-8	X	15	2
Iron	Fe	7439-89-6	X	100	10
Lead[trace]	Pb	7439-92-1	X	9	0.8
Lithium	Li	7439-93-2	X	10	5
Magnesium	Mg	7439-95-4	X	200	20
Manganese	Mn	7439-96-5	X	10	1
Molybdenum	Mo	7439-98-7	X	20	2
Nickel	Ni	7440-02-0	X	40	4
Phosphorus	P	7723-14-0	X	3,000	300
Potassium	K	7440-09-7	X	3,000	300
Selenium[trace]	Se	7782-49-2	X	15	1.3
Silicon	Si	7631-86-9		500	50
Silver[trace]	Ag	7440-22-4	X	10	1
Sodium	Na	7440-23-5	X	1	100
Strontium	Sr	7440-24-6	X	10	1
Sulfur	S	7704-34-9	X	200	2
Thallium[trace]	Tl	7440-28-0	X	15	1.2
Thorium	Th	7440-29-1		15	15
Tin	Sn	7440-31-5	X	100	10
Titanium	Ti	7440-32-6	X	10	1
Uranium	U	7440-61-1		60	20
Vanadium	V	7440-62-2	X	10	2
Zinc	Zn	7440-66-6	X	20	2
Zirconium	Zr	7440-67-7		15	1

Attachment 2

Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (µg/L)	Matrix Spike Level (µg/L)
Aluminum	2,000	2,000
Antimony	500	500
Arsenic	2,000	2,000
Barium	2,000	2,000
Beryllium	50	50
Bismuth	2,000	2,000
Boron	1,000	1,000
Cadmium	50	50
Calcium	50,000	50,000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1,000	1,000
Lead	500	500
Lithium	1,000	1,000
Magnesium	50,000	50,000
Manganese	500	500
Molybdenum	1,000	1,000
Nickel	500	500
Phosphorous	10,000	10,000
Potassium	50,000	50,000
Selenium	2,000	2,000
Silicon	10,000	10,000
Si (as SiO_2)	21,400	21,400
Silver	50	50
Sodium	50,000	50,000
Strontium	1,000	1,000
Sulfur	2,000	2,000
Thallium	2,000	2,000
Thorium	2,000	2,000
Tin	2,000	2,000
Titanium	1,000	1,000
Uranium	2,000	2,000
Vanadium	500	500
Zinc	500	500
Zirconium	500	500

Attachment 3

Low Level ICV/CCV

ELEMENT	LCS Level (µg/L)
Aluminum	100
Antimony	10
Arsenic	15
Barium	10
Beryllium	1
Bismuth	100
Boron	100
Cadmium	5
Calcium	200
Chromium	10
Cobalt	10
Copper	15
Iron	100
Lead	9
Lithium	10
Magnesium	200
Manganese	10
Molybdenum	20
Nickel	40
Phosphorous	3,000
Potassium	3,000
Selenium	15
Silicon	500
Si (as SiO_2)	1070
Silver	10
Sodium	1,000
Strontium	10
Thallium	15
Thorium	15
Tin	10
Titanium	10
Uranium	60
Vanadium	10
Zinc	20
Zirconium	15

Attachment 4

Interference Check Sample Concentrations

Element	ICSA (µg/L)	ICSAB (µg/L)
Aluminum	500,000	500,000
Antimony	-	1,000
Arsenic	-	2,000
Barium	-	500
Beryllium	-	500
Bismuth	-	1,000
Boron	-	2,000
Cadmium	-	1,000
Calcium	500,000	500,000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200,000	200,000
Lead	-	1,000
Lithium	-	1,000
Magnesium	500,000	500,000
Manganese	-	500
Molybdenum	-	1,000
Nickel	-	1,000
Phosphorous	-	2,000
Potassium	-	50,000
Selenium	-	5,000
Silicon	-	10,000
Silica	-	21,400
Silver	-	1,000
Sodium	-	50,000
Strontium	-	1,000
Sulfur	-	1,000
Thallium	-	10,000
Titanium	-	1,000
Vanadium	-	500

Attachment 4

Interference Check Sample Concentrations cont'd

Element	ICSA (µg/L)	ICSAB (µg/L)
Zinc	-	1,000
Tin	-	10,000
Thorium	-	10,000
Uranium	2,000	2,000
Zirconium	-	1,000

Attachment 5

TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (µg/L)	Regulatory Limit (µg/L)	Spike Level (µg/L)
Arsenic	500	5000	4000
Barium	10000	100000	12000
Cadmium	100	1000	1100
Chromium	500	5000	5200
Lead	500	5000	5500
Selenium	250	1000	3000
Silver	500	5000	1050
Copper	100	N/A	2250
Zinc	200	N/A	2500

Attachment 6

6000 Dual View Calibration, ICV & CCV Standards

Element	Calibration Level	ICV (µg/L)	CCV (µg/L)
Aluminum Lo	1,000	250	500
Aluminum Hi	100,000	40,000	50,000
Antimony	2,000	250	1,000
Arsenic	2,000	250	1,000
Barium	1,000	250	500
Beryllium	1,000	250	500
Bismuth	2,000	500	1000
Cadmium	1,000	250	500
Calcium	10,000	2,000	5,000
Chromium	1,000	250	500
Cobalt	1,000	250	500
Copper	1,000	250	500
Iron Lo	5,000	250	2,500
Iron Hi	100,000	80,000	50,000
Lead	2,000	250	1000
Magnesium	40,000	10,000	20,000
Manganese	1,000	250	500
Molybdenum	1,000	250	500
Nickel	1,000	250	500
Phosphorous	2,000	2,000	1,000
Potassium	100,000	20,000	50,000
Selenium	2,000	500	1,000
Silver	1,000	250	500
Sodium Lo	10,000	2000	5,000
Sodium Hi	500,000	40,000	250,000
Strontium	1,000	250	500
Sulfur	10,000	4,000	5,000
Thallium	2,000	500	1,000
Thorium	10,000	3,000	5,000
Tin	2,000	500	1,000
Vanadium	1,000	250	500
Uranium	20,000	5,000	10,000
Zinc	1,000	250	500
Zirconium	1,000	250	500

Attachment 7
Summary Of Quality Control Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between multiple exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
CCV	After the ICV, after every 10 samples and at the end of the run.	90-110% recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
RL Standard	At the beginning of the run	Results must within 50%	Terminate analysis; Correct the problem; Recalibrate.
LLICV/CCV	At the beginning of the run and after every 10 samples	Recovery must be within 30%	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable LLCCV.
ICB	Beginning of every analytical run, immediately following the initial CCV.	The result must be within +/- ½ RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCB	Immediately following each CCV (except for the CCV following the ICV).	The result must be within +/- ½ RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.10	See Section 9.10
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.10
Dilution Test	One per prep batch.	For samples > 10x LQM (after dilution)' dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.

See Section 10.5.3 for run sequence to be followed.

Attachment 7

Summary of Quality Control Requirements (Continued)

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to ½ the RL. Sample results greater than 10x the blank concentration are acceptable. Samples for which the contaminant is < ½ RL may not require redigestion or reanalysis (see Section 9.3)	Re-run once in a clean tube. If > ½ RL, re-digest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.4)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike (MS)	One per sample preparation batch of up to 20 samples.	75 – 125% recovery or tighter in-house control limits.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate (MSD)	One per sample preparation batch of up to 20 samples. 10% frequency for some programs (see 9.5)	75 – 125 % recovery; RPD ≤ 20% or tighter in-house control limits.	See Corrective Action for Matrix Spike.

Attachment 8

ICP Data Review Checklist

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ICP Data Review Checklist

Run/Project Information:		
Run Date:	**Analyst:**	**Instrument:**
Prep Batches Run:		
Methods Used: 6010B / 200.7		

Review Items	Yes	No	N/A	2nd Level
A. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits?				
2. Method blank done per prep batch and < RL or CRDL (CLP)?				
3. MS run at required frequency and within limits?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
5. Serial dilution done per prep batch (or per SDG for CLP)?				
6. Post digest spike analyzed if required (CLP & AFCEE only)?				
B. Calibration/Instrument Run QC				
1. ICV/CCV analyzed at appropriate frequency and within control limits ? (**6010B**: CLP = 90 - 110%; **200.7:** ICV = 95 - 105%, CCV = 95 - 105% for 40 CFR 136.3, 90 - 110% for RCRA / SDWA) If not in control, was the CCV reanalyzed twice to show return to control as per NELAP?				
2. ICB/CCB analyzed at appropriate frequency and < RL or < CRDL (CLP) or < 2X MDL (AFCEE 4.0)?				
3. High Standard (HIGH) reanalyzed before samples and recovered within QC limits? (6010B/200.7 - 95-105%)				
4. RL STD run and recovered within QC limits ? (± 50% for non-CLP, ± 20% for DoD / AFCEE 4.0 / USACE)				
5. ICSA/ICSAB run at required frequency and within SOP limits? (ICSA < 2X MDL AFCEE 4.0)				
C. Sample Results				
1. For 6010B, were samples with concentrations > the linear range for any parameter diluted and reanalyzed? For 200.7, were samples with concentrations within 90% of the linear range diluted and reanalyzed?				
2. Are all reported results bracketed by in control QC?				
D. Other				
1. Are all nonconformances documented appropriately?				
2. Calculations checked for errors?				
3. Transcriptions checked for errors? (Example: Are dilution factors that are entered into the sequence log correct?)				
4. All client/project specific requirements met?				
5. Date/time of analysis verified as correct?				

Analyst: _____ **Date:** _____

Comments: _____

2nd Level Reviewer: _____ **Date:** _____

Comments: _____

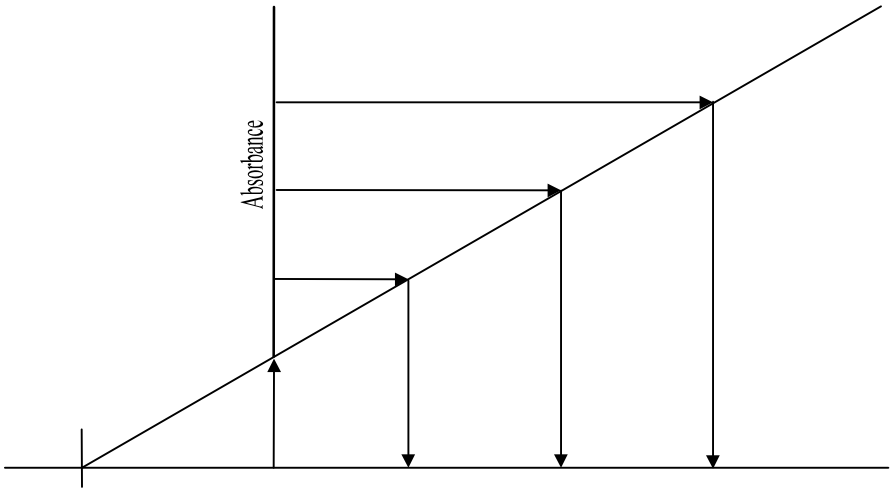
Attachment 9

MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the absolute value of the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear (r=0.995 or greater) over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Attachment 10

Troubleshooting Guide

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Replace RF generator
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

Attachment 11

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered gloves should not be used in the metals laboratory because the powder contains silica and zinc as well as other metallic analytes.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipette tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

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Title: Mercury in Solids by Cold Vapor Atomic Absorption

[SW 7471B]

Approvals (Signature/Date):

Doug Gomer — 7/10/12
Doug Gomer, Date
Metals Supervisor

Adam Alban — 11 July 12
Adam Alban, Date
Health & Safety Manager / Coordinator

7/6/12
John P. Morris, Date
Quality Assurance Manager

Robert C. Hanisch — 7/11/12
Robert C. Hanisch, Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471B.

1.2 Method 7471B is applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, and sludge-type materials. All matrices require sample preparation prior to analysis. This is not an appropriate procedure for the digestion of tissues or other organic matrices, which require the use of EPA 245.6 instead.

1.3 If sample preparation utilizing the Incremental Sampling Method is required, see SOP DV-OP-0013 for the procedure required prior to acid digestion for metals incorporating this procedure.

1.4 The routine reporting limit for mercury in solid matrices is 17 μg/kg.

2.0 Summary of Method

A representative portion of the sample is digested in aqua regia in the first digestion cycle and potassium permanganate in the second cycle. Mercury is reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorption of light at 253.7nm is calibrated as a function of mercury concentration.

3.0 Definitions

3.1 **Total Mercury:** Inorganic forms of mercury are effectively dissolved by the acids used in the digestion. The potassium permanganate reagent breaks down organo-mercury compounds to inorganic forms that are detected by this procedure.

3.2 **Aqua Regia:** A 3:1 mixture of hydrochloric and nitric acids. This mixture is effective at dissolving metals in the solid form.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Potassium permanganate “suitable for mercury determination” is specified because of the potential for mercury contamination in the reagent. In addition, potassium permanganate crystals will absorb mercury vapors from the air. Reagent bottles must be kept tightly closed to avoid contamination.

4.3 Potassium permanganate, in addition to breaking down organic compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 ppm of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.4 Copper has also been reported to interfere; however, copper concentrations as high as 10 ppm had no effect on the recovery of mercury from spiked samples.

4.5 Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

NOTE: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.6 Interference from certain volatile organic materials that absorb at the wavelength used for the method may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.7 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be lessened by reducing the volume of original sample used. Alternatively, EPA Method 245.6 can be used.

4.8 The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately. It is recommended that a disposable face shield be worn when making up aqua regia

5.3.2 Potassium permanganate is a strong oxidizing agent. It is incompatible and must be stored separately from hydroxylamine hydrochloride and stannous chloride, the reducing agents used in this procedure, and from acids.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury Nitrate Solutions	Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydroxylamine hydrochloride	Corrosive Poison	No OSHA PEL listed for this compound	Direct contact with skin or eyes causes irritation. May cause skin sensitization, an allergic reaction. Inhalation or ingestion may cause methemoglobinemia and resulting cyanosis (bluish discoloration of skin due to deficient oxygenation of the blood), and labored breathing.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Digestion Block, with adjustable heating, capable of maintaining a sample temperature of 90-95°C.

6.1.2 Mercury Autoanalyzers:

6.1.2.1 CETAC Mercury Analyzer with Autosampler and Auto-Diluter

6.2 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

6.3 Supplies

6.3.1 Disposable digestion tubes with caps, accuracy verified to +-3% gravimetrically prior to use

6.3.2 Disposable glass tubes, 16mm x 100mm

6.3.3 Argon, 99.999% purity

6.3.4 Calibrated automatic pipettes or Class A glass volumetric pipettes (see SOP No. DV-QA-0008 for details on calibrating mechanical pipettes)

6.3.5 Class A volumetric flasks.

6.3.6 Thermometer, non-mercury column, accurate to ±1°C at 95 °C (see SOP No. DV-QA-0001 for calibration details).

6.3.7 Glass beads, <1 mm diameter, acid washed.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 **Reagent water:** Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2 **Nitric acid (HNO_3):** concentrated, trace metal grade or better.

7.3 **Hydrochloric acid (HCl):** concentrated, trace metal grade or better.

7.4 **Aqua Regia:** Add 600ml concentrated HCL and 200ml concentrated HNO3 to a 1 liter container. Aqua regia will be prepared immediately before use

7.5 **Calibration Blank, Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Method Blank (MB), 1% HNO_3:**

7.5.1 Add 0.5 L of concentrated HNO_3 to a 50-L carboy partially filled with reagent water.

7.5.2 Dilute to 50 L with reagent water.

7.6 **Stannous Chloride Solution ($SnCl_2$), Hg grade, 10% (w/v) per manufacturer's instructions. (CETAC Only)**

7.6.1 Place approximately 100 mL of deionized water into a 2-L volumetric flask

7.6.2 Slowly add 200 mL of concentrated HCl to the flask and swirl to mix.

7.6.3 Add 200 grams of $SnCl_2$ to the flask.

7.6.4 Place a large stir bar in the flask and put the flask on a stir plate.

7.6.5 Stir the contents of the flask until the reagent is completely dissolved.

7.6.6 Remove the stir bar and make to volume with deionized water.

7.7 Sodium chloride-hydroxylamine hydrochloride solution (Hg grade):

Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride (Hg grade) to every 100 mL of reagent water.

NOTE: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.8 Potassium permanganate, 5% solution (w/v):

Dissolve 5 g of potassium permanganate (reagent grade, "suitable for mercury determination") for every 100 mL of reagent water.

7.9 Purchased Mercury Stock Solutions

Primary Mercury Calibration Standard Solution, 1,000 mg/L

7.10 Calibration Working Standard Solution, 10 mg/L

7.10.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL Class A volumetric flask.

7.10.2 Pipet 1.00 mL of the 1000 mg/L primary mercury calibration standard solution into the flask.

7.10.3 Dilute to the mark on the flask with 1% HNO_3.

7.10.4 Stopper the flask and shake to mix.

7.10.5 Transfer the solution to a 125-mL Nalgene bottle.

7.10.6 Document the preparation of the solution in the Standards Log database.

7.10.7 Prepare this solution fresh monthly or more often if necessary.

7.11 Daily Calibration Working Solution (100 μg/L)

7.11.1 Add approximately 90 mL of 1% HNO_3 to a 100-mL volumetric flask.

7.11.2 Add 1.00 mL of the 10 mg/L Calibration Working Standard (see section 7.10).

7.11.3 Bring the solution to a final volume of 100.0 mL.

7.11.4 Stopper and mix thoroughly.

7.11.5 Document the preparation in the Standards Log database.

7.11.6 Prepare this solution each day prior to calibration.

7.12 Initial Calibration (ICAL) Standards

The initial calibration standards are prepared directly in the digestion tubes as follow:

ICAL	Daily Cal Working Std (mL)	1% HNO_3 (mL)	Final Conc (µg/L)
Blank	0.0	5.0	0.0
Std 1	0.1	4.9	0.20
Std 2	0.25	4.75	0.50
Std 3	0.5	4.5	1.0
Std 4	1.0	4.0	2.0
Std 5	2.5	2.5	5.0
Std 6	5.0	0.0	10.

7.13 Second-Source Initial Calibration Verification (ICV) Daily Intermediate Standard, approximately 400 µg/L.

Add 400uL of the 100 mg/L ICV Standard to a 100-mL volumetric flask partially filled with 1% HNO_3 and dilute to the mark. Record this information in the Standards Log database.

7.14 Second-Source Initial Calibration Verification (ICV) Daily Working Standard, approximately 4.00 µg/L.

Add 0.5 mL of the 400 µg/L ICV Daily Intermediate Standard (see section 7.13) to a soil digestion tube and add 4.5 mL of 1% HNO_3. Record this information in the Standards Log database.

7.15 Continuing Calibration Verification (CCV) Standards, 5 µg/L

7.15.1 The CCVs are prepared exactly as the 5.0 µg/L ICAL standard shown above (see section 7.12).

7.15.2 Prepare sufficient volume of the standard for analysis of a CCV after every 10 samples.

7.16 Laboratory Control Standard (LCS), 417 µg/kg

7.16.1 The LCS is prepared in an empty digestion tube for which 0.6 g of glass beads are used.

7.16.2 Add 2.5 mL of the 100 µg/L Daily Calibration Working Standard (see section 7.11) to a digestion tube. No additional reagent water is added at this time, per method, but is accounted for when digestate is brought to the final volume of 50 mL.

7.16.3 This is equivalent to a 5.0 µg/L ICAL standard, which is the concentration that appears on the raw data printout from the instruments.

7.17 Matrix Spike and Matrix Spike Duplicate (MS/MSD), 417 µg/kg

MS/MSD pairs are spiked in the same manner as the LCS (see section 7.16) and prepared in the same manner as the samples, using 0.6 g of sample.

7.18 Reporting Limit (RL) Check Standard, 17 µg/kg

7.18.1 Add 0.1 mL of 100 µg/L Daily Calibration Working Standard (see section 7.11) and 4.9 mL of reagent water to a digestion tube.

7.18.2 This is equivalent to a 0.2 µg/L ICAL standard, which is the concentration that appears on the raw data printout from the instruments.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soil	Glass	3 grams	Cool $\leq 6^{o}C$	28 Days	N/A

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Preparation Batch

A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate pair (MS/MSD). As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.3 Method Blank (MB)

The MB consists of an empty vessel or <1-mm glass beads (for DoD and AFCEE projects) containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. At least one method blank (MB) must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than the project-specific data quality objectives. In the absence of project-specific data quality objectives, the blank must be less than the ½ the reporting limit or less than 10% of the mercury concentration found in the associated samples, whichever is higher.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If mercury was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.4 Laboratory Control Sample (LCS), 417 µg/kg

The preparation of the LCS is described in Section 7.16. The LCS is spiked at the project-specific action level, or when lacking project-specific action levels, between the low and midlevel standards. At least one aqueous LCS must be processed

with each preparation batch. The LCS must be carried through the entire analytical procedure.

Acceptance Criteria: Maximum control limits for LCS recoveries are 80-120%. In-house control limits based on three standard deviations of the mean of historical results are used as long as they are at least as tight as 80-120% (see Policy QA-003 for further details on establishing control limits).

Corrective Action: If LCS recoveries are outside established control limits, the system is out of control and corrective action must occur. If recoveries are above control limits and mercury is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD), 417µg/kg

One MS/MSD pair must be processed for each preparation batch. Some programs may require the use of sample duplicates in place of or in addition to MS/MSDs. In addition, some programs will allow spikes to be reported only for project-related samples. Samples identified as field blanks cannot be used for MS/MSD analysis. MS/MSD is spiked at the same levels as the corresponding LCS.

Acceptance Criteria: Control limits are statistically determined based on three standard deviations of the mean of the laboratory's historical data. The MS/MSD recovery must fall within 80-120%; the relative percent difference (RPD) between the MS and MSD cannot exceed 20%.

Corrective Action: If analyte recovery or RPD fails acceptance criteria, the LCS recovery must be in control for the data to be reported. If there is no evidence of analytical problems and all other QC criteria are met, then qualified results may be reported and the situation must be described in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC, then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

9.6 Serial Dilution

Some programs (e.g., DoD and AFCEE programs) require that a fivefold (1+4) dilution must be included in each analytical batch for each sample matrix.

Acceptance Criteria: The results must be within 10% of the expected value, assuming that the sample concentration is at least 25x the MDL concentration.

Corrective Action: If the result fails the acceptance criteria, all associated sample results must be qualified.

9.7 Post-Digestion Spikes

Some programs require the inclusion of a post-digestion spike in each analytical batch. The post-digestion spike is prepared by adding 0.5 mL of the 100 µg/L Daily Calibration Working Solution to 10 mL of filtered sample digestate. Post-digestion spikes are performed as an additional check for matrix interference.

Acceptance Criteria: The percent recovery limits for the post-digestion spike are 85 to 115%.

Corrective Action: If the result fails the acceptance criteria, all associated sample results must be qualified.

9.8 Method of Standard Addition (MSA)

The method of standard additions is an option for the analysis of samples shown to have significant matrix effects, e.g., unacceptably low MS/MSD recoveries or under certain conditions for TCLP analysis (see Attachment 4 for details).

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

10.3.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB), as well as the field samples, are processed through the digestion

procedure. Prepare digestion tubes containing volumes of standards required for each tube as listed in section 7.

10.3.2 Weigh a 0.5 – 0.6 g aliquot of a well homogenized sample and place in a sample digestion tube. (See No. DV-QA-0023 for additional information on subsampling.)

10.4 Digestion of 3 g sample aliquot obtained utilizing previously prepared Incremental Sampling Method soil aliquot

10.4.1 Five 0.6 g samples and QC are digested in five individual digestion tubes and combined into one after adding the sodium chloride-hydroxylamine hydrochloride.

10.4.2 Prepare an MB, LCS, MS, and MSD for each batch. The MB is either an empty digestion tube or is prepared by placing 0.6 g of glass beads in a digestion tube, depending on client requirements. The LCS is prepared by adding 2.5 mL of the 100 μg/L Daily Calibration Working Solution to a digestion tube. The MS is prepared by adding 2.5 mL of the 100 μg/L Daily Calibration Working Solution to a digestion tube containing a second aliquot of the chosen sample. The MSD is prepared in the same manner as the MS using a third aliquot of the chosen sample.

NOTE: The spike must be added after the sample aliquot but before the addition of reagents.

10.4.3 Add 5.0 mL of aqua regia to each tube.

10.4.4 Add 5.0 mL of reagent water to all un-spiked field samples and the method blanks. Add 2.5 mL of reagent water to the LCS, MS and MSD.

10.4.5 Heat for 2 minutes at 95+/-3 ° C. Record the start and stop times and the temperature on the bench sheet.

10.4.6 Allow the samples and standards to cool at room temperature.

10.4.7 Add 19 mL of reagent water.

10.4.8 Add 15 mL of 5% potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may need to be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If after the addition of up to 25 mL additional permanganate, the color does not persist, sample dilution prior to reanalysis may be required.

NOTE: It is important that equal volumes of the potassium permanganate solution are added to all solutions in the batch. Unequal volumes used with the automated method will result in dilution errors. Unequal volumes used with the manual method will result in differing purging efficiencies.

10.4.9 Cap the samples and standards and heat for 30 minutes on the digestion block at 95 +/-3°C. Record the start and stop times and the temperature on the bench sheet. The analyst will verify that a purple color persists or a black precipitate is present after the thirty minutes of heating. If this is not true, the digestion must be repeated using more potassium permanganate or less sample.

10.4.10 Allow the samples and standards to cool at room temperature.

10.4.11 Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate. Add additional 1%HNO_3 to bring the final volume to 50 mL.

Note: The sample volume must be verified before mixing. Due to the limited headspace left in the digestion tube it is likely that the sample will react with the sodium chloride-hydroxylamine hydrochloride solution possibly causing some loss.

10.4.12 For aliquots taken by the Incremental Sampling Method combine the 5 individual sample cups for each sample and QC into one marked 250ml container.

10.5 Calibration

10.5.1 All calibration standards are digested together with samples, as described in Section 10.1, prior to analysis. Preparation of calibration materials is described in Section 7.

10.5.2 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.5.3 Detailed information regarding calibration models and calculations can be found in Corporate SOP No. CA-Q-S-005, Calibration Curves (General).

10.5.4 Initial Calibration (ICAL)

10.5.4.1 Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.5.4.2 Calibrate using six standards and a blank.

NOTE: It is generally not acceptable to reject calibration points for this method.

10.5.4.3 The calibration curve must have a correlation coefficient of ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

10.5.4.4 Record the counts for the 10 ppb standard in the instrument maintenance log.

10.5.5 Initial and Continuing Calibration Blanks

10.5.5.1 An initial calibration blank (ICB) is tested immediately after the daily ICAL standards.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as sodium must be less than the RL. Client specific requirements take precedence. For example, DoD requires control of blanks to a concentration less than or equal to the LOD.

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

10.5.5.2 Continuing calibration blanks (CCBs) are run after every 10 samples and at the end of the run.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as sodium must be less than the RL. Client specific requirements take precedence. For example, DoD requires control of blanks to a concentration less than or equal to the LOD.

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

10.5.6 Initial Calibration Verification (ICV), approximately 4.0 µg/L

The accuracy of the calibration standards is verified by testing a second source standard (ICV).

Acceptance Criteria: The ICV recovery must be within 90-110%.

Corrective Action: If the ICV acceptance limit is exceeded, the analysis should be terminated, the accuracy of the calibration standards checked, and the instrument recalibrated.

10.5.7 Reporting Limit Check Standard (RL), 0.2 µg/L

The accuracy of results at the reporting limit is verified by testing a standard in every analytical run that is prepared at the reporting limit concentration.

Acceptance Criteria: The results for this standard must be within 50% of the expected value (20% for some programs).

Corrective Action: If the RL check acceptance limit is exceeded, the analysis should be terminated, the instrument operation checked, the instrument recalibrated, and associated samples reanalyzed.

10.5.8 Continuing Calibration Verification (CCV), 5.0 µg/L

Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the run.

Acceptance Criteria: The CCV recovery must be within 80-120%.

Correction Action: Sample results may be reported only when bracketed by valid CCV pairs. If a CCV fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and the affected samples reanalyzed. If the cause of the CCV failure was not directly related to the instrument, the associated samples must be reanalyzed.

10.6 Sample Analysis

NOTE: Because of differences between various makes and models of CVAA instrumentation, detailed push-button operating instructions are not provided here. Refer to the specific instrument operating manual for detailed autosampler setup and operation protocols.

NOTE: The injection of samples and the addition of stannous chloride are done automatically by the instrument. Refer to the specific instrument manual for details.

10.6.1 Set up the instrument and autosampler according to the manufacturer's instructions.

10.6.2 Allow the samples to cool to room temperature prior to analysis or a decrease in the response signal can occur.

10.6.3 Aliquot each sample and calibration standard into a disposable test tube for analysis.

10.6.4 Analyze the standards and samples according to the manufacturer's instructions.

10.6.5 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for mercury concentrations that exceed the highest calibration standard.

NOTE: The instrument auto-dilutes samples. Any samples that require greater than a 10x dilutions MUST be diluted manually.

10.6.6 If the sample results are negative and the absolute value is greater than the reporting limit, the sample must be reanalyzed.

10.6.7 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB. Re-sloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

10.6.8 The analytical sequence listed below must be followed. Refer to Quality Control Section 9.0 and Table I (Appendix A) for quality control limits.

Instrument Calibration
ICV
ICB
RL
Maximum of 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

NOTE: Samples included in the count between CCVs include the method blank, LCS, MS, MSD, and field samples.

10.6.9 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\textbf{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\textbf{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{} \times 100$$

spiked concentration

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Concentration:

$$\text{mg/kg or mg/L} = \frac{C \times V \times D}{W}$$

Where:

C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4 Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Sample preparation bench sheet(s), to include the batch number, list of samples, preparation analyst and date, instrument analysis analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes). This information is stored in the LIMS.
- Raw data (direct instrument printout).
- Data review checklist - See Attachment 3
- Standards Documentation to include source, lot, preparation date, and expiration date. This information is stored in the LIMS.
- Nonconformance summary (if applicable).

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy DV-QA-005P. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD, AFCEE, and DOE projects, an MDL verification is performed quarterly.

12.2 Demonstration of Capabilities

12.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

12.2.2 IDOCs and on-going proficiency demonstrations are conducted as follows. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample is typically the LCS spike level. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Safety Manual, and HS-001, "Waste Management Program."

14.1 The following waste streams are produced when this method is carried out:

14.1.1 Aqueous Acidic (Metals) - Corrosive - (J)

14.1.2 Expired reagents and standards – Contact the Waste Coordinator.

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Update IV, February 2007, Method 7471B (Mercury).

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, March 2005.

15.3 U.S.EPA Statement of Work for Inorganic Analysis, ILMO3.0

16.0 Method Modifications:

Item	Method	Modification
1	7471B	An additional QC analysis, RL verification, is added
2	7471B	Methods 7470A and 7471B state that working standards "should be prepared fresh daily." The laboratory frequently prepares up to three batches of mercury samples, including digested calibration standards, each day. The third batch is typically prepared and digested late in the day, and then is analyzed the morning of the next day. The laboratory had developed the following information demonstrating that analysis within 24 hours, but on the second calendar day from preparation produces reliable results and is acceptable to the EPA: • Successful proficiency testing (PT) results for samples that were prepared and analyzed within 24 hours, but on successive days • Successful analysis of true NIST mercury standards within every analytical batch; and • A written comment from the EPA MICE Hotline stating that, with the supporting lab data, their opinion was that the laboratory's practice is "within the letter of the method as written."
3	Chapter 1, SW846	Chapter 1 of SW-846 specifies the use of reagent water with a purity equivalent of ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
4	7471B	7471B uses reagent water for the method blank. TestAmerica Denver is currently using glass beads.
5	7471B	Section 11.1 requires 50 mL of reagent water to be added to the sample with 15 mL of Potassium permanganate. TestAmerica Denver utilizes digestion tubes which do no allow for 50 mL of reagent water. 19 mL of reagent water is currently being added.

17.0 Attachments

Attachment 1: Mercury Preparation & Analysis Flowchart
Attachment 2: Summary of Quality Control Requirements
Attachment 3: Example Data Review Checklist
Attachment 4: MSA Guidance
Attachment 5: Instrument Maintenance

18.0 Revision History

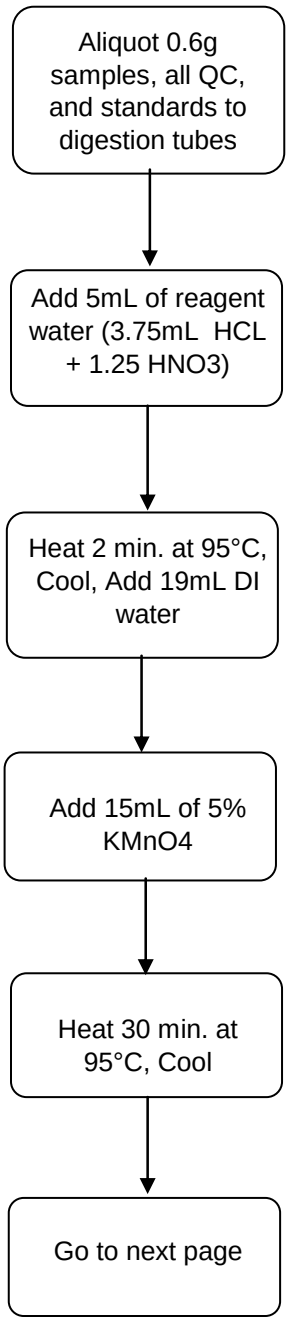
- Revision 1.2 dated 13 July 2012
  - Updated Sections 7.6 and 7.7 for using Hg grade reagents
  - Updated Sections 10.4.11 to bring samples to 50 mL with 1% HNO3

  - Added Section 10.5.4.4 to record the counts of the 10 ppb high standard
  - Updated Section 10.5.5 to control the calibration blanks to ½ the RL
  - Updated Sections 9.1, 10.1, 10.2, 12.1 to reflect current practice
  - Formatting and grammatical changes throughout

- Revision 1.1 dated 03 February 2012
  - Annual technical review
  - Changed references of Multi-Incremental Sampling to Incremental Sampling Method throughout document
  - Section 1.3 Added statement for Incremental Sampling Method
  - Added introductory statement to section 7.0 regarding reagent purity
  - Updated section 9.5 and Attachment 2 for Method Blank criteria
  - Section 10.2.12 Added Incremental Sampling Method combination procedure
  - Section 10.2.13 Added Incremental Sampling Method final volume
  - Added dilution note to Section 10.3.5
  - Updated section 12.0 to reflect current laboratory practice
- Revision 1.0, dated 31 August 2011
  - Added a note to section 10.1.3 for the addition of the LCS/MS spike before reagents.
  - Updated Section 5.1.1 to include using a face shield when making up aqua regia
  - Added Section 7.4 for how to make aqua regia
  - Removed previous Section 7.6 (FIMS information)
  - Updated Section 7.13 ICV daily intermediate standard level to 400ug/l
  - Updated Section 7.14 ICV daily working standard level to 4ug/l
  - Updated Section 10.2.3 ICV level to 4ug/l
  - Changed run order for ICV and ICB in section 10.3.8
  - Changed section 10.1.4 to say 5ml of aqua regia
- Revision 0.8, dated 25 April 2011
  - Removed all references to the FIMS Hg Analyzer
  - Sections 6.1 and 6.3 were updated to reflect the use of digestion blocks from water baths
- Revision 0.7, data 07 February 2011
  - Updated supplies list for implementation of calibrated digestion tube
  - Updated Section 10 to incorporate use of calibrated tube for digestion.
  - Updated Section 6 to include reference to the Master List of Documents, Software and Hardware.
- Revision 0.6, dated 01 September 2010
  - Annual Technical Review
  - Removed comment about Standards Log in Section 7
  - Updated Section 11.4 for new LIMS
  - Removed example prep sheet (Attachment 3)
- Revision 0.5, dated 07 August 2009
  - Removed Reagent Blank from Section 7.4
  - Changed table heading in section 7.12 to say 1% HNO_3 from reagent blank
  - Changed Section 7.13 and 7.14 to use 1% HNO_3 from reagent blank

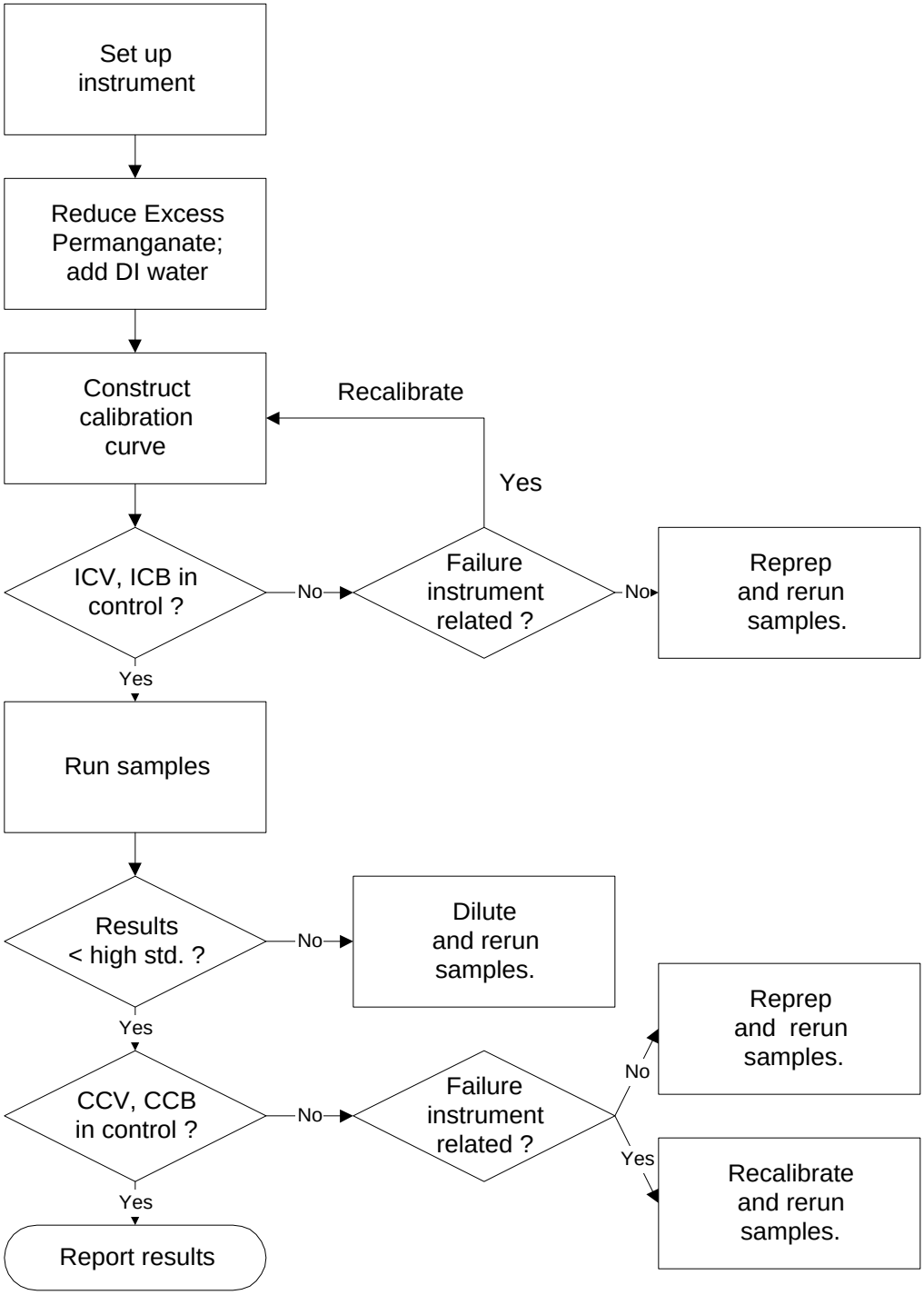
Attachment 1.

Mercury Preparation & Analysis Flowchart



Attachment 1.

Mercury Preparation & Analysis Flowchart (continued)



Attachment 2.

Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICB	Immediately following ICAL	Absolute value < ½ RL (2x MDL for DoD)	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
ICV	Following ICB	90- 110% recovery	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
RL Check Standard	Following the ICV	50-150% recovery (80-120% for DoD)	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
CCV	Every 10 samples and at the end of the run	80 - 120 % recovery.	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
CCB	Immediately following each CCV	Absolute value < ½ RL (2x MDL for DoD)	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
Method Blank	One per sample preparation batch of up to 20 samples.	Project specific or ≤1/2 RL Sample results greater than 10% the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion	Redigest and reanalyze samples. Note exceptions under criteria section.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Recovery must be within statistical control limits, not to exceed 80 - 120%	Terminate analysis; correct the problem; redigest and reanalyze all samples associated with the failed LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Recovery must be within statistical control limits, not to exceed 75-125%	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate	See Matrix Spike	Recovery within statistical control limits, not to exceed 75-125 % recovery or in-house control limits; RPD ≤ 20%	See Corrective Action for Matrix Spike.

Attachment 3.

Example Data Review Checklist

TESTAMERICA-DENVER

Applicable QC Batches:_____
_____
_____

Industrial Mercury Analysis Raw Data Checklist

Revision 3 -December 19, 2007

Analyst's Checklist

	yes	no	n/a
1. Were the special instructions for prep and/or analysis followed?	☐	☐	☐
2. Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3. Is the blank less than the reporting limit or properly anomalized?	☐	☐	☐
4. Is the LCSs within limits or properly anomalized?	☐	☐	☐
5. Is the ICV and all CCVs within limits?	☐	☐	☐
6. Are all CCBs within ± one reporting limit from zero?	☐	☐	☐
7. Were the CCVs and CCBs run with up to 10 samples between each set?	☐	☐	☐
8. Are the reporting limits correct and reflect any dilutions?	☐	☐	☐
9. Are the number of significant figures correctly reported?	☐	☐	☐
0. Are the benchsheets complete (including calibration and standard verification #'s)?	☐	☐	☐
1. Are all comments, footnotes, and anomalies properly documented?	☐	☐	☐
2. Are holding time violation forms completed and attached?	☐	☐	☐
3. Has all sample data been entered into LIMS?	☐	☐	☐
4. Has all QC data been entered into LIMS?	☐	☐	☐
5. Has the data entered into LIMS been checked for errors?	☐	☐	☐
6. For TCLP results, is the sample data within 20% of Regulatory Level (0.2 mg/L)?	☐	☐	☐

Analyst's Name:_____ Date:_____

Data Review's Checklist

	yes	no	n/a
1. Have the calculations been checked?	☐	☐	☐
2. Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3. Is all the QC data within the control limits and/or properly anomalized?	☐	☐	☐
4. Are all the significant figures and reporting limits correct?	☐	☐	☐
5. Have any comments, footnotes, and anomalies been properly documented?	☐	☐	☐
6. Have any data errors been documented and entered into LIMS?	☐	☐	☐
7. Is prep date correct in LIMS?	☐	☐	☐
8. Has the data package been copied and filed?	☐	☐	☐
9. If TCLP result within 20% of Reg. Level (0.2 mg/l) and MS < 50%, was MSA performed?	☐	☐	☐

Reviewed by:_____ Date:_____

Comments:_____
_____
_____

Anomalies:_____
_____
_____

Attachment 4.

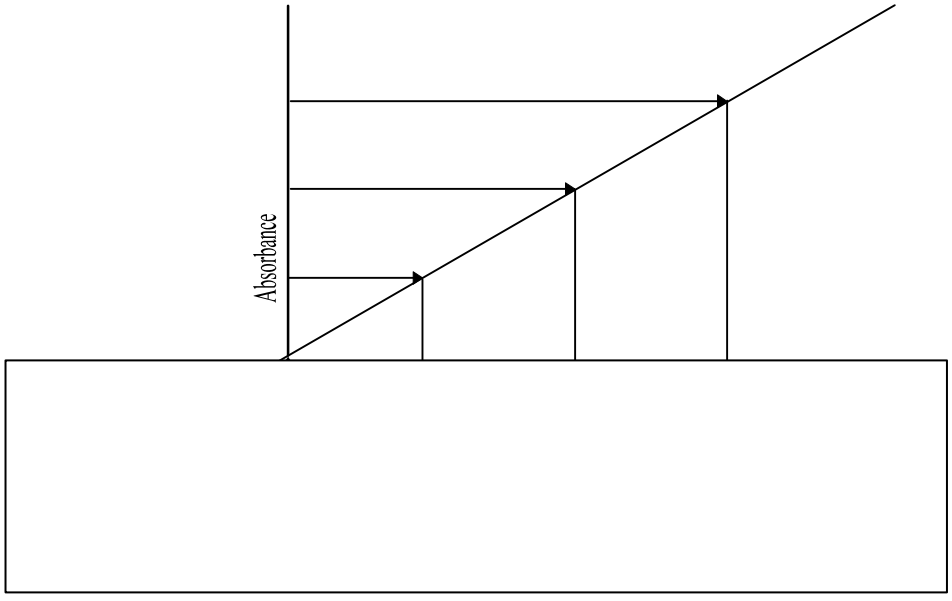
MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Attachment 5.

Instrument Maintenance

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs, record the date, time and instrument number, then identify the problem and corrective action in the maintenance log. When the instrument is returned to service, record the return to service, the date, and any tests performed to verify proper operation.

The following preventative maintenance procedures are <u>required</u> to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption

Daily	Monthly	Annually
Change rinse solution.	Check Hg lamp intensity.	Change Hg lamp.
Optimize light path.		Check liquid/gas separator.
Check argon flow.		
Check tubing. Replace as needed.		
Check drain.		
Check condition of dryer		

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series

Approvals (Signature/Date):

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Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

This Standard Operating Procedure (SOP) is applicable to the solvent extraction of organic compounds from water samples, TCLP leachates, SPLP leachates, and Wyoming Leachates using a separatory funnel. This SOP based on SW-846 Method 3510C, EPA 608, EPA 610, EPA 614, AK102, NWTPH-Dx, and Oklahoma DRO method.

The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate pH and spiking mixtures are used.

This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, "Concentration of Organic Extracts", for details concerning the concentration and cleanup of extracts.

2.0 Summary of Method

A measured volume of sample, usually 1 liter, is placed in a separatory funnel. The pH is adjusted as required for the efficient extraction of specific compounds. The organic compounds are extracted with three portions of methylene chloride. The water phase is discarded. The organic phase is dried using sodium sulfate.

3.0 Definitions

3.1 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. Please reference WI-DV-0032 for details on Method Comments.

3.4 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.5 **Aliquot**: A part that is a definite fraction of a whole; as in "take an aliquot of a sample for testing or analysis." In the context of this SOP, "aliquot" is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.3 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented in an NCM.

4.4 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. Especially take note of the possibility of phthalate contamination from gloves. Gloves should be changed out frequently and whenever they come in contact with solvent. Glassware should be handled in a fashion that keeps gloves away from the interior and mouth of the glassware.

4.5 The decomposition of some analytes has been demonstrated under basic extraction conditions. Organochlorine pesticides may dechlorinate, phthalate esters may exchange, and phenol may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510C. Method 3510C is preferred over Method 3520C for the analysis of these classes of compounds. However, the recovery of phenols is optimized by using Method 3520C and performing the initial extraction at the acid pH.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 The use of separatory funnels to extract samples using methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity. Either a face shield must be worn over safety glasses or goggles must be worn when it is performed.

5.1.2 Glass centrifuge tubes can break in the centrifuge if proper care is not taken. This can lead to a hazardous material spill and endanger employees. Do not exceed the manufacturer's recommended maximum RPM for glass containers. Normally speeds greater than 2700 rpm are not advisable.

5.1.3 The procedure calls for the use of an electric rotator. The rotator is equipped with a safety latch that does not allow the rotator to rotate even if the power switch is turned on. The separatory funnels are secured to the rotator using straps. During the procedure it will be necessary to loosen the straps in order to un-stopper the separatory funnels. Whenever the straps are loose, the safety latch must be fastened to prevent the rotator from rotating.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Serious or Significant Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 mg/m3	Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, and runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and can cause burns that may result in permanent impairment of vision, even blindness with greater exposures.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Sulfuric Acid	Corrosive Carcinogen	1 mg/m3	Inhalation may cause irritation of the respiratory tract with burning pain the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema. Causes chemical burns to the respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Causes skin burns. Causes severe eye burns. May cause irreversible eye injury, blindness, permanent corneal opacification.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit			

6.0 Equipment and Supplies

NOTE: All glassware used in this procedure is cleaned following SOP DV-OP-0004. In addition, the glassware is rinsed with methylene chloride immediately prior to use.

6.1 Supplies

- Separatory funnel, 2-liter with polytetrafluoroethylene (PTFE) stopcock and stopper.
- Separatory funnel, 500-mL with polytetrafluoroethylene (PTFE) stopcock and stopper.
- Separatory funnel rack and mechanical rotator.
- Balance, ≥ 1400 g capacity, accurate to ± 1 g, calibration checked daily per SOP DV-QA-0014.
- pH indicator paper, wide range.
- Class A Graduated Cylinder, sizes ranging from 50 mL to 1 L.
- Media bottles, 300 mL with Teflon-lined caps or capped with aluminum foil.
- Media bottles, 100 mL with Teflon-lined caps or capped with aluminum foil.
- Disposable pipettes, various volumes.
- Stemless glass funnel.
- Glass wool, baked at 400 °C for four hours.
- Mechanical pipette, 1 mL, positive displacement, with disposable tips, calibrated per SOP DV-QA-0008.
- Aluminum foil.
- Paper towels.

6.2 Computer Software and Hardware

- Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagent Water

7.1.1 TestAmerica Denver has two ELGA water purification systems. The water coming from the ELGA system should be 18-18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026.

7.2 Methylene Chloride

Each lot of solvent is tested following SOP CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.3 Acids and Bases

7.3.1 Sulfuric Acid (H2SO4), 1:1
TALS Reagent ID "1:1 H2SO4"

Place an ice water bath on a stir plate. Place a container with a magnetic stir bar in the bath. While stirring, slowly add 1 part concentrated reagent grade sulfuric acid (36N) to 1 part water from the ELGA purification system. Assign a 1 year expiration date from the date made or the vender expiration date, whichever is shorter.

7.3.2 Sodium Hydroxide (NaOH), 10N
TALS Reagent ID "10N_NaOH"

Purchased at ready-to-use concentration from commercial vendors. Assign a 1 year expiration date from the date opened or the vender expiration date, whichever is shorter.

7.3.3 Hydrochloric Acid (HCl), 1N
TALS Reagent ID "1N_HCl"

Dilute 100 mL of stock reagent grade, concentrated HCl to 1000 mL with reagent water.

7.4 Baked Sodium Sulfate, 12-60 mesh

Heat sodium sulfate in a 400 °C oven for at least four hours. Store in tightly closed container.

7.5 Baked Sodium Chloride

Bake in 400 °C oven for at least 4 hours.

Standards

7.6 Please reference SOP DV-OP-00020 and WI-DV-009 for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix and Method	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	Amber Glass	1000 mL	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
Water for Method AK 102	Amber Glass	1000 mL	Cool 4 ± 2°C and pH ≤ 2 with HCl	14 Days if properly preserved. 7 Days if un-preserved.	Method AK 102
Water for Method Oklahoma DRO	Amber Glass	1000 mL	Cool 4 ± 2°C and pH ≤ 2 with HCl	7 Days	Oklahoma Dept. of Environmental Quality
Water for Method NWTPH-DX	Amber Glass	1000 mL	Cool 4 ± 2°C and pH ≤ 2 with HCl	7 Days	NWTPH-Dx
Water for Method 8082 or 8082A	Amber Glass	1000 mL	Cool 4 ± 2°C	None [2]	SW-846 Chapter 4, Revision 4, Feb 2007
Water for Method 8270 by Large Volume Injection	Amber Glass	250 mL	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
TCLP Leachates	Glass	200 mL for 8270 100 mL for 8081	Cool 4 ± 2°C	7 Days from the completion of the leach	SW-846 1311
SPLP Leachates	Glass	1000 mL	Cool 4 ± 2°C	7 Days from the completion of the leach	SW-846 1312
Wyoming Leachates	Glass	1000mL	Cool 4 ± 2°C	7 Days from the completion of the leach	

[1] Exclusive of analysis.

[2] Some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require a 1 week hold time for method 8082 and 8082A. The states of California, South Carolina, Pennsylvania, and Connecticut require a 1 week hold time.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, refer to the Method Comments and QAS to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Assurance Program.*

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, *Requirements for Federal Programs*.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS and in the Quality Assurance Summaries (QAS) available in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank is processed and analyzed just as if it were a field sample.

The method blank for batches of aqueous samples for 8270 with Large Volume Injection (prep method 3510C_LVI) consists of 250mL of reagent water free of any of the analyte(s) of interest.

The method blank for batches of aqueous samples for all other methods consists of 1 L of reagent water free of any of the analyte(s) of interest.

The method blank for batches of TCLP leachates for method 8081 consists of 100 mL of leach fluid.

The method blank for batches of TCLP leachates for method 8270 consists of 200 mL of leach fluid.

The method blank for batches of SPLP or Wyoming leachates consists of 1 L of leach fluid.

Acceptance Criteria: The result for the method blank must be less than the reporting limit for the analyte(s) of interest or less than 10% of the analyte concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: If target analytes in the blank exceed the acceptance limits, an acceptable method blank must be re-prepared and reanalyzed. If the analyte was not detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.5 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

At least one LCS must be processed with each preparation batch. The LCS is carried through the entire analytical procedure just as if it were a sample.

The method blank for batches of aqueous samples for 8270 with Large Volume Injection (prep method 3510C_LVI) consists of 250mL of reagent water to which the analyte(s) of interest are added at known concentrations.

For aqueous sample batches for all other methods, the LCS consists of 1 L of reagent water to which the analyte(s) of interest are added at known concentration.

For method 8081 TCLP leachates, the LCS consists of 100 mL of leach fluid to which the analyte(s) of interest are added at known concentration.

For method 8270 TCLP leachates, the LCS consists of 200 mL of leach fluid to which the analyte(s) of interest are added at known concentration.

For SPLP leachates and Wyoming leachates, the LCS consists of 1 L of leach fluid to which the analyte(s) of interest are added at known concentration.

Method 608, 614, 610 requires a LCS at a 10% frequency. In other words one LCS is required for a batch of 10 or less samples. A LCSD is required for a batch of 11 or more samples.

Method AK102 requires LCS and a LCSD for every batch for every spike compound.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared unless Method Comments indicate otherwise.

Method 608, 610, and 614 requires one matrix spike for every 10 samples. If the batch has more than 10 samples, then two matrix spikes must be performed. The two matrix spikes

are to be performed on two different samples. If there is insufficient sample volume for matrix spikes, then a LCSD must be performed.

Method NWTPH-Dx requires a matrix spike and a matrix spike duplicate for every 10 samples. If insufficient sample volume is available for MS/MSD, a NCM must be written and a LCS and LCSD must be performed for every 10 samples.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at ± 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Critical Procedural Considerations

10.2.1 As stated throughout this SOP, analysts must review the Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).

10.2.2 Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other separatory funnel than the designated one should be cleaned or disposed of before coming into contact with the sample.

10.3 Assemble and clean the glassware immediately before use.

10.3.1 Place a stopcock in each separatory funnel. For 1-liter extractions use a 2000mL sepfunnel. For 250mL, 200mL and 100mL extractions, use a 500mL sepfunnel. Place a stopper for each separatory funnel on a clean sheet of aluminum foil that is marked with individual positions for each stopper. This is done to prevent cross-contamination.

10.3.2 For each separatory funnel, plug a glass funnel with baked glass wool and add baked sodium sulfate. Place the funnel on a media bottle and place the media bottle below the separatory funnel.

10.3.3 Rinse each separatory funnel once with methylene chloride. Be sure that all surfaces come into contact with the solvent. Drain the methylene chloride into the media bottle through the sodium sulfate.

10.3.4 Rinse the sodium sulfate with additional methylene chloride if the first rinse did not completely saturate the sodium sulfate.

10.3.5 Allow the methylene chloride to drain completely into the media bottle. Swirl the media bottle to ensure all surfaces come into contact with the solvent. Add additional methylene chloride to the rinse if necessary.

10.3.6 Discard the methylene chloride.

10.3.7 Label each media bottle with the sample ID or batch QC ID.

10.4 Prepare LCS and Method Blank Samples

NOTE: For SW-846 methods if there is not a MS/MSD pair in the batch then perform a LCS/LCSD. Methods 608, 610, and 614 require a LCS and LCSD in batches of 11 samples or more or if there are no Matrix Spikes in batches of 10 or less.

10.4.1 For aqueous sample batches logged for Large Volume Injection, (3510_LVI), pour 250mL of reagent water into the separatory funnels marked for the LCSs and the MB.

10.4.2 For all other aqueous sample batches, pour 1 liter of reagent water into the separatory funnels marked for the LCSs and the MB.

NOTE: If any sample in the batch is preserved with sodium thiosulfate, then the LCS's and MBs should also contain sodium thiosulfate. Add 1mL of the 0.08 g/mL solution to the water in the separatory funnel.

10.4.3 For 8270 TCLP leachates, use a 250mL or 500mL Class A graduated cylinder to measure out 200 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest mL. Place the

leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.4.4 For 8081 TCLP leachates, use a 100mL or 250mL Class A graduated cylinder to measure out 100 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.4.5 For SPLP leachates, use a 1000mL Class A graduated cylinder to measure out 1000 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest 10 mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.4.6 For Wyoming leachates, measure out 1000 mL of the appropriate leach fluid for each MB and LCS and LCSD. This can be done gravimetrically or volumetrically. If done volumetrically, record the volume to the nearest 10mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct blank fluid was used.

10.5 Measure the initial sample pH of the samples.

10.5.1 Measure the initial sample pH with wide-range pH paper and record the pH on the extraction bench sheet.

10.5.2 If the sample is logged for AK102_103, Okla_DRO, or NWTPH_Dx the samples should have been field preserved. See Section 8. If the samples are not preserved, an NCM should be written.

10.6 Aliquot the samples

10.6.1 For 8270 TCLP leachates, use a 250mL or 500mL Class A graduated cylinder to measure out 200 mL of the leachate. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.6.2 For 8081 TCLP leachates, use a 100mL or 250mL Class A graduated cylinder to measure out 100 mL of the leachate. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.6.3 For SPLP leachates, use a 1 Liter Class A graduated cylinder to measure out 1000 mL of the leachate. Record the volume to the nearest 10 mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.6.4 For Wyoming leachates, measure out 1000 mL of leachate. This can be done gravimetrically or volumetrically. If done volumetrically, record the volume to the nearest 10mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct blank fluid was used.

10.6.5 For water samples, it should be noted that TestAmerica Denver routinely aliquots gravimetrically. This is done to prevent cross-contamination due to volumetric glassware and to provide a more accurate initial volume measurement. However, some clients and regulatory programs require the

laboratory to aliquot samples volumetrically. The Method Comments and QASs must be read before samples are aliquotted to check for this requirement. If samples are to be aliquotted volumetrically, use Class A volumetric glassware only and proceed to Section 10.6.7

10.6.6 Weigh the bottle (250mL amber bottles for 3510C_LVI or 1000mL amber bottles for all other aqueous samples) and record the gross weight to the nearest gram. If there is any indication that the sample's density is not 1g=1mL, then measure the density of the sample using a calibrated pipette and an analytical balance. The weight of the sample extraction will be corrected for the density later. See Section 11 for the calculation. For example, normally a 1 liter bottle weighs 500g when empty and when filled completely can only hold 1060mL, therefore a full bottle weighing more than 1560g is an indication that either the sample density is greater than 1g or the sample bottle contains a lot of sediment. Document any sample with a density greater than 1g in an NCM.

10.6.7 Inspect the samples for large amounts of sediment that may interfere with the extraction of the sample by causing excessive emulsions or clogging the stop-cock.

10.6.7.1 If the sample contains so much sediment that the entire sample volume cannot be extracted, decant the sample into the separatory funnel (or a 1 L graduated cylinder if volumetric aliquotting is required), careful not to transfer the sediment. Write a NCM to document the sediment and that it prevented the entire sample volume from being extracted and the sample container from being solvent rinsed.

10.6.7.2 If the sample does not contain a significant amount of sediment, then the entire sample volume will be used in the extraction. Do not pour the sample into the separatory funnel (or into the graduated cylinder if volumetric aliquotting is required) until after the surrogates and any necessary spikes have been added to the samples.

10.6.8 Place the sample containers in front of the separatory funnel labeled for that sample. A second analyst should then check the labels to make sure the correct sample is being extracted. This check is documented in the Organic Extraction Checklist (WI-DV-0009)

10.7 Add Surrogates to All Field Samples and QC Samples

10.7.1 The standards should be allowed to come to room temperature before spiking the samples. Record the ID of the standard used on the benchsheet.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-009.

10.7.2 Only one batch should be surrogated at a time to ensure the correct standards are used.

10.7.3 Add the appropriate volume of the appropriate working surrogate standard to the sample container for each sample and MS/MSD. Add the surrogate standard to the MB and the LCS's in the separatory funnels. Record the ID of the standard used on the bench sheet. Reference work instruction WI-DV-009 to determine the appropriate standard and the appropriate volume.

Note: If the sample contains an amount of sediment that has been deemed to interfere with the extraction process then the surrogate standard is added to the sample in the separatory funnel or in the graduated cylinder. This is considered a deviation and must be documented in a NCM.

10.8 Add Spikes to all LCS's and MS/MSDs

10.8.1 Add the appropriate volume of the appropriate working spike standard to the MS/MSD sample containers and the separatory funnels for the LCS and/or LCSD samples. Record the ID of the standard used on the bench sheet. Reference work instruction WI-DV-009 to determine the appropriate standard and the appropriate volume.

10.9 Add approximately 3g (1/2 teaspoon) of NaCl to all samples and all QC samples. This is done to give the reagent water used in the MBs and LCSs some ionic strength to more closely mimic the matrix of actual water samples and to aide in the extraction of the more polar target compounds. Record the lot number of the sodium chloride on the bench sheet.

NOTE: South Carolina samples must be batched separately. QC samples for these batches use reagent water directly from the Elga system. ***DO NOT ADD NaCl to any South Carolina samples or QC samples.***

10.10 If volumetric aliquotting is required, transfer the entire sample into a Class A graduated cylinder and record the volume on the benchsheet. If the sample bottle contains more than 1000 mL, a 100mL Class A graduated cylinder can be used to complete the measurement. The entire sample volume must be used. Record the volume to the nearest 10 mL. Then pour the sample into the labeled separatory funnel. Place the used graduated cylinder in front of the appropriate separatory funnel so it can be solvent rinsed later.

NOTE: A 1000 mL Class A graduated cylinder is not accurate enough to measure to the nearest 1 mL. Therefore all samples that are aliquoted using a 1000 mL Class A graduated cylinder will have the initial volume recorded to the nearest 10 mL. This accuracy is sufficient.

10.11 If volumetric aliquotting is not required, pour the sample directly into the separatory funnel. Place the empty sample container in front of the appropriate separatory funnel so it can be solvent rinsed.

10.12 Adjust pH of Field Samples and QC Samples

Adjust the sample pH as indicated in the chart below using a minimum amount of 1:1 sulfuric acid (or 1 M hydrochloric acid for Methods AK102, Okla_DRO and NWTPH_Dx) or 10 N sodium hydroxide, as necessary. Record the adjusted pH and the lot number of the acid or base on the bench sheet.

NOTE: For 250mL water samples logged for 8270C with a 3510C_LVI prep method, normally 1mL of acid is required.

NOTE: TCLP Leachates may have pH of < 5. In those cases, the pH should be adjusted per the table below.

Method	Initial Extraction pH	Secondary Extraction pH
8270C 8270_AFCEE 8270_DoD 8270D	1 – 2	If samples are TCLP leachates extract at 14. If samples are water extract at 11 - 12
8270C_SIM 8270_SIM_AFCEE 8270_SIM_DoD	As Received	None
608 8082 8082A 8081A 8081B	5 - 9	None
614 8141A 8141B	5-8	None
8015B_DRO 8015C_DRO 8015D_DRO 8015B_Terp	As Received	None
610 8310	As Received	None
AK102_103 Okla_DRO NWTPH_Dx	If samples are preserved between pH 1 – 2, then acidify the MB and LCS. Otherwise extract as received and document insufficient preservation in an NCM.	None

10.13 For 1 Liter samples, add 60 mL of methylene chloride to each empty sample container, unless the entire sample volume was not used. For 250mL or smaller samples, add 15mL of methylene chloride to each empty sample container, unless the entire sample volume was not used. Cap the container and shake gently to rinse all internal surfaces of the bottle. Pour the methylene chloride from the sample container into the appropriate separatory funnel. If a graduated cylinder was used to aliquot volumetrically, rinse the cylinder and add that rinse to the separatory funnel as well. Record the lot number of the methylene chloride on the bench sheet. If the sample contained significant sediment and the entire sample contents could not be extracted, do not rinse the empty sample container, but instead add the solvent directly to the separatory funnel. If the solvent rinse of the sample container cannot be performed, prepare a NCM.

10.14 For water samples that were aliquoted gravimetrically, reweigh the bottle and calculate the initial sample volume by subtracting the empty bottles weight from the full bottles weight, assuming a density of 1g=1mL. If there is any indication that the samples density is not 1g=1mL then measure the density of the sample and correct the calculated initial volume accordingly using the formula in Section 11. Document abnormal sample density in an

NCM. For example, normally a 1 liter bottle when filled completely can only hold 1060mL, therefore an initial volume greater than 1060mL is an indication that the density is not 1g. Document any sample with a density greater than 1g in an NCM.

10.15 If the initial volume is less than 80% of the nominal volume, the sample reporting limits and method detection limits will be elevated substantially. Document this in a NCM.

10.16 Stopper and rotate the separatory funnel for 3 minutes with periodic venting to release excess pressure. Document the extraction date and time on the benchsheet.

WARNING: Methylene chloride creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken a few seconds. Vent into hood away from people and other samples. A face shield or goggles must be worn during venting.

10.17 Allow the organic layer to separate from the water phase for at least 5 minutes or until complete visible separation has been achieved. This can take up to 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, use mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, pouring the solvent layer and emulsion back through the top of the separatory funnel (pour-back), or centrifugation. The emulsion could also be filtered through the glass funnel by adding additional sodium sulfate to remove all water in the emulsion. This technique should only be used after other techniques have failed to make complete phase separation and only after the last shake.

NOTE: As much as 15 to 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.

10.18 Drain the lower methylene chloride layer into the sodium sulfate filled glass funnel. Allow the methylene chloride to drain completely into the media bottle. Rinse the sodium sulfate with a small amount of methylene chloride to ensure that all compounds of interest are collected in the media bottle. Record the lot number of the sodium sulfate on the bench sheet. If the sodium sulfate becomes saturated with water, add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

10.19 Repeat the extraction two more times for a total of 3 extractions. Collect all three methylene chloride extracts in the same media bottle.

10.20 For the base/neutral and acid extractable method 8270, adjust the pH of the samples according to chart in Section 10.12. For 250mL water samples, usually 2mL of base is required. Then extract the sample 3 more times.

10.21 Cap the media bottle with a Teflon-lined cap or aluminum foil and submit for concentration and possible clean-up steps.

10.22 Dispose of the solvent-saturated water remaining in the separatory funnel in the appropriate waste container. See Section 14.

10.23 Initial weights and volumes of samples are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklists (see WI-DV-009).

11.0 Data Analysis and Calculations

$$InitialVolume(mL) = \frac{FullBottle(g) - EmptyBottle(g)}{Density(g/mL)}$$

12.0 Method Performance

12.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL). See Policy DV-QA-005P, "Determination of Method Detection Limits", for more information on the method detection limit studies.

12.2 An initial demonstration of capability (IDOC) must be performed by each analyst. On-going proficiency must be demonstrated by each analyst on an annual basis. See DV-QA-0024, "Employee Training", for more information on the IDOCs.

12.3 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

13.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methylene chloride – Waste Stream B

14.2.2 Solid waste/sodium sulfate – Waste Stream D

14.2.3 Basic aqueous sample waste saturated with methylene chloride – Waste Stream X.

14.2.4 Acidic aqueous sample waste saturated with methylene chloride – Waste Stream Y.

14.2.5 Neutral aqueous sample waste saturated with methylene chloride – Waste Stream X or Waste Stream Y.

14.2.6 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

15.2 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608, Organochlorine Pesticides and PCBs.

15.3 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 610, Polynuclear Aromatic Hydrocarbons.

15.4 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 614, Organophosphorous Pesticides.

15.5 Alaska Method AK102, “For the Determination of Diesel Range Organics”, Version 04/08/02.

15.6 Alaska Method AK103, “For the Determination of Residual Range Organics”, Version 04/08/02.

15.7 NWTPH-Dx “Semi-Volatile Petroleum Products Method for Soil and Water.

15.8 Oklahoma Department of Environmental Quality Methods 8000/8100 (Modified) Diesel Range Organics (DRO) Revision 4.1 Date 10/22/97

16.0 Modifications:

16.1 Modifications from SW-846 Method 3510C

16.1.1 Section 7.1 of the method calls for initial sample volume to be determined volumetrically either by measuring out exactly 1 liter or marking the meniscus on the sample container and later determining the volume of water required to fill the bottle back up to the mark. This SOP allows the initial sample volume

to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware.

16.1.2 Section 7.5 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.

16.1.3 Section 7.6 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic layer to separate from the water phase for a minimum of 5 minutes, up to 10 minutes if the separation is not complete.

16.1.4 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 3g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.1.5 The source method calls for samples to be extracted for method 8141 at the pH they are received. This procedure calls for the extraction to be performed at a pH between 5 and 8. This is done per guidelines found in Section 2 and Section 8 of SW-846 8141B.

16.2 Modifications from 40 CFR Method 608, and 610

16.2.1 Section 10.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

16.2.2 Section 10.2 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.

16.2.3 Section 10.2 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic layer to separate from the water phase for a minimum of 5 minutes, up to 10 minutes if the separation is not complete.

16.2.4 Section 10.3 of the method calls for rinsing the sample collection bottle with the 60 mL methylene chloride aliquot for the second and third extraction as well as the first extraction. This SOP calls for rinsing the sample collection bottle with only the first 60-mL methylene chloride aliquot.

16.2.5 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 3g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.3 Modifications from 40 CFR Method 614

16.3.1 Section 10.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

16.3.2 Section 10.2 of the method calls for the extraction to be performed with at 15% v/v methylene chloride in hexane solvent. This procedure uses methylene chloride for the extraction. SOP DV-OP-0007 calls for the methylene chloride extract to be concentrated and exchanged to hexane.

16.3.3 Section 10.2 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.

16.3.4 Section 10.2 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic layer to separate from the water phase for a minimum of 5 minutes, up to 10 minutes if the separation is not complete.

16.3.5 Section 10.3 of the method calls for rinsing the sample collection bottle with the 60 mL solvent aliquot for the second and third extraction as well as the first extraction. This SOP calls for rinsing the sample collection bottle with only the first 60-mL methylene chloride aliquot.

16.3.6 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 3g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.4 Modifications from Method AK 102

16.4.1 Section 9.1.1.1 of the method calls for using no more than 1 liter of sample and to determine the volume either by measuring out exactly 1 liter or marking the meniscus on the sample container and later determining the volume of water required to fill the bottle back up to the mark. This SOP allows the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware. This SOP allows for the extraction of more than 1 L as it calls for the use of the entire sample volume.

16.4.2 Section 9.1.1.6 of the method says to allow the water and solvent layers to separate for approximately 10 minutes. This SOP calls for the allowing the organic layer to separate from the water phase for a minimum of 5 minutes, up to 10 minutes if the separation is not complete.

16.4.3 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 3g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.5 Modifications from Method NWTPH-Dx

16.5.1 The method calls for determining the initial volume of the sample my marking the meniscus on the bottle and later determining the volume of tap water required to fill the bottle back up to the mark. This SOP allows the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware.

16.5.2 The method calls for shaking the separatory funnel for one minute. This SOP calls for the separatory funnel to be shaken for at least three minutes.

16.5.3 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 3g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.6 Modifications from Oklahoma DRO

16.6.1 The method calls for aliquotting 800 mL to 900 mL of the sample volumetrically. This SOP calls for the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware. This SOP allows for the extraction of more than 1 L as it calls for the use of the entire sample volume.

16.6.2 The method calls for extracting using 50mL of solvent. This SOP calls for the extraction to be done using at least 60mL of solvent.

16.6.3 The method calls for shaking the separatory funnel for two minutes. This SOP calls for the separatory funnel to be shaken for at least three minutes.

16.6.4 The method calls for a method blank and LCS to be analyzed every 10 samples. This SOP calls for a method blank and LCS to be analyzed every batch of 20 samples.

16.6.5 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 3g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

17.0 Attachments

Table 1. Determinative Methods Using Separatory Funnel Extractions

18.0 Revision History

- **Revision 9.0, January 15, 2013**
  - Section 10.9 was updated to include note to eliminate use of salt in South Carolina samples.
- **Revision 8.0, September 25, 2012**
  - This procedure was updated to include instructions on how to extract 8270 water samples for Large Volume Injection.
- **Revision 7.0, January 31, 2012**
  - Annual Technical Review
  - Updated Section 6.2 to describe the requirements for computer software and hardware
  - Updated Section 7.0 to describe requirements for Reagents and Standards.
  - Updated Section 8.0 to state PCBs by method 8082 have no holding time as per SW-846 Update 4 and that samples for analysis by NW-TPH have a 7 day hold time, even if acid preserved.
  - Updated Section 9.1.4 and Section 10.1 to accurately describe the NCM notification system.

- Updated Section 10.4 and 10.6 to state the appropriate size of the graduated cylinders to be used to measure out 100mL and 200mL of leachate.
- Updated Sections 10.6.6 and 10.14 to give guidance to the analyst when a density check of a sample is required.
- Updated Section 10.9 to give more detail on how much sodium chloride should be added to the samples.
- Updated Section 16 to include the method modification of the sodium chloride addition.
- Updated Table 1 to reflect the current analytical SOPs.
- Corrected grammatical and formatting errors

- **Revision 6.0 dated 01/10/11**
  - Added note to Section 6 that sodium sulfate should be stored in tightly closed container.
  - Revised Section 7 to reference DV-OP-00020 for information about surrogate and spike standards.
  - Corrected Section 7.1 to indicate that the reagent water should be 18 to 18.2 Mohm/cm.
  - Revised procedure to include details on the extraction of Wyoming Leachates.
  - Added references to methods NWTPH-Dx, and Oklahoma DRO.
  - Added Section 6.2 computer software and hardware.
  - Section 8 was revised to give more detail on the preservation and hold times for methods AK102, AK103, NWTPH-Dx, and Oklahoma DRO.
  - Revised Section 9 to include more detail on QC requirements for methods AK102_103, NWTPH-Dx, and Oklahoma DRO.
  - Revised Section 10 to clarify that when 1 liter graduated cylinders are used to measure the initial volume of the water samples, that the volume should be recorded to the nearest 10mL.
  - Revised Section 10 to instruct that if samples for methods AK102_103, NWTPH-Dx, and Oklahoma DRO are received preserved, then the MB and the LCS samples should also be acidified with HCl. Otherwise the samples are extracted as received.
  - Revised Section 16 to include more detail on modification from methods AK102_103, NWTPH-Dx, and Oklahoma DRO
  - Revised the procedure to call for the 2nd fraction of 8270 TCLP leachates to be extracted at a pH of 14 instead of the pH 11 to 12 used in water samples. This was done to help the recovery of pyridine.

- **Revision 5.2 dated 9/30/09**
  - Added clarification for the criteria of surrogating and spiking samples directly into the original container.

- **Revision 5.1, dated 18 September 2009**
  - Added criteria for surrogating and spiking samples directly into the original container.
  - Added comments in Section 4 about phthalate contamination arising from gloves.
  - The procedure was revised to include the addition of approximately 3 grams of baked sodium chloride to every sample and QC sample in order to increase the ionic strength of QC samples and field QC samples to more closely match the ionic strength of typical samples and to aide in the extraction of the more polar compounds.
  - Eliminated the “short-list” 8270 LCS spike mix. All 8270 LCSs are spiked using the full list 8270/625 LCS mix, which was also revised to correct the analyte list.

- **Revision 5, dated 17 June 2009**
  - Updated Table 1 to include all determinative methods and SOPs used in conjunction with

this SOP.
- Revised Section 7.1 to define reagent water as 3 g of baked NaCl added to 1 L of water from the ELGA purification system. This was done to more closely mimic the ionic strength of environmental samples.
- Revised Table 2 to clarify how the motor oil LCS standard is prepared and to clarify that the standards are prepared as separate working level standards.
- Revised Table 3 to clarify that the toxaphene LCS standard is prepared as a separate standard from the organochlorine pesticide standard.
- Revised Table 4 to add the surrogate tetrachloro-m-xylene.
- Revised Table 5 to add compounds to the organophosphorus pesticide spike standard.
- Revised Section 7 to delete the method 625/AFCEE standard. The laboratory uses the standard referenced in Table 7 for all method 8270 procedures, except TCLP leachates.
- Revised the 8270 TCLP standard to correct the final concentrations.
- Removed Attachment 1 “Organic Extractions Checklist” and added references to WI-DV-009.
- Section 10.7 was revised to instruct the analyst to adjust the pH of samples logged in for method 8141 and 614 to a pH between 5 and 8.
- Section 10 was revised to instruct the analyst to solvent rinse the empty sample containers for all samples, not just samples logged in for 600 series tests.

- **Revision 4, dated 13 February 2008**
  - Added information in section 5 about safety latch on the rotator.
  - Updated section 7.9 to include the expiration dates of all standards.
  - The solvent used to prepare the method 8081 spike standard described in section 7.9.5.1 has been changed to methanol to prevent the breakdown of delta-BHC. This change required the standard to have a 1 week expiration date.
  - Section 9.0 was updated to clarify the frequency requirement for LCS/LCSDs in method 608, 610,and 614.
  - Section 9.0 was revised to instruct the lab that for SW-846 method batches if a MS/MSD is not performed a LCS/LCSD is needed for precision.
  - Section 10.3 was revised to give more detail on the labs procedure for aliquoting samples gravimetrically.
  - Table 3 was revised to include alpha-chlordane.
  - Table 6 was revised to include the concentrations of both the soil and water LCS standard.
  - Table 7 was revised to add additional compounds in the spike solution.
  - Section 16 was modified to include modifications from method 614.

- **Revision 1, dated 13 February 2008**
  - Integration for TestAmerica and STL operations.

TABLE 1.

Determinative Methods Using Separatory Funnel Extractions

Method Description	*Determinative Method*	*SOP*
Diesel Range Organics & Jet Fuels	SW-846 8015, California LUFT Method, Alaska Methods AK102 & AK103 SW-846 8015C	DV-GC-0027
Chlorinated Pesticides	SW-846 8081A SW-846 8081B EPA Method 608	DV-GC-0020 DV-GC-0016
Polychlorinated Biphenyls	SW-846 8082 SW-846 8082A EPA Method 608	DV-GC-0021 DV-GC-0016
Organophosphorus Pesticides	SW-846 8141A, & EPA Method 614	DV-GC-0017
Polynuclear Aromatic Hydrocarbons (PAH)	SW-846 8310 & EPA Method 610	DV-LC-0009
Semi-volatiles by GC/MS	SW-846 8270 SW-846 8270D	DV-MS-0011 DV-MS-0012
PAH by GC/MS SIM	SW-846 8270	DV-MS-0002

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Title: Concentration and Clean-up of Organic Extracts [SW-846, 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A, and EPA 600 Series Methods]

Approvals (Signature/Date):

Susan Oster 11/23/12 Susan Oster Date Technical Specialist	Adam Alban 03 Dec 12 Adam Alban Date Health & Safety Manager / Coordinator
12/3/12 John Morris Date Quality Assurance Manager	12/3/12 William S. Cicero Date Laboratory Director

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1.0 Scope and Application

1.1 This standard operating procedure (SOP) provides instructions for the concentration, and if necessary, cleanup, of solvent extracts of organic compounds from water samples, soil samples, TCLP leachates, and SPLP leachates. This SOP is based on SW-846 Methods 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A, and EPA 600 Series methods.

1.2 The determinative methods and extraction methods used in conjunction with this procedure are listed in Attachment 1.

NOTE: This SOP does not include the concentration steps of extracts for Herbicides by method 8151A or 615. See DV-OP-0011 instead.

1.3 This procedure does not include the extraction steps. See the following SOPs for the applicable extraction procedures:

DV-OP-0006:	Extraction of Aqueous Samples by Separatory Funnel, SW-846 3510C and EPA 600 Series
DV-OP-0008:	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0010:	Soxhlet Extraction of Solid Samples, SW-846 3540C
DV-OP-0015	Microwave Extraction of Solid Samples, SW-846 3546
DV-OP-0016:	Ultrasonic Extraction of Solid Samples, SW-846 3550B and 3550C
DV-OP-0021:	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C for Low-Level NDMA by GC/CI/MS/MS
DV-MS-0005, Appendix II:	Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C for Extended List PAHS for CSLP.

2.0 Summary of Method

Sample extracts are concentrated to a specific final volume using an S-EVAP, N-EVAP, or Turbo-Vap. Some methods require a solvent exchange. If necessary, various clean-up techniques are performed before the extract is sent for analysis.

3.0 Definitions

3.1 **Extraction Holding Time:** The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch:** A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards.

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. See WI-DV-0032

3.4 **Quality Assurance Summary (QAS):** Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the special instructions/Method Comments field in LIMS. In those situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

4.0 Interferences

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Visual interferences or anomalies (such as foaming, emulsions, odor, more than one layer of extract, etc.) must be documented.

4.3 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.4 Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by GC/CI/MS/MS must be concentrated in glassware designated for that method. K-D flasks, concentrator tubes, stem-less glass funnels, and snyder columns will be clearly marked and segregated for this purpose.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 In order to limit the emission of methylene chloride, TestAmerica Denver uses a solvent recovery system. The system condenses and collects methylene chloride that has been evaporated off the sample extracts while on the S-EVAP.

5.1.1.1 Each analyst must inspect the system before using it to ensure the collection tubes are in good condition, the in-process tanks are not full, and the chiller is operating correctly.

5.1.1.2 While concentrating methylene chloride or methylene chloride / acetone extracts on the S-Evap, the analyst will use a timer set at 30 minute intervals to help remind the analyst to check the level of the solvent collected in the in-process tanks. This will be done to ensure that the tanks are not over-filled. A tank will not be filled more than 90%.

5.1.1.3 The solvent recovery system will never be used for the collection of ether due to the potential danger to analysts if the system were to fail during operation.

5.1.2 Glasswool is a carcinogen and therefore should be handled in a hood to avoid inhalation of dust.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit [(1)]	Signs and Symptoms of Exposure
Acetonitrile	Flammable Irritant Poison	40ppm TWA	Exposure may cause cyanide poisoning resulting in reddening of the skin and eyes and pupil dilation. Effects of overexposure are often delayed due to the slow formation of cyanide ions in the body. May cause nose and throat irritation, flushing of the face, tightening of the chest. Also may cause headache, nausea, abdominal pain, convulsions, shock.
Hexane	Flammable Irritant	50ppm TWA	Causes irritation to eyes, skin and respiratory tract. Aspiration hazard if swallowed. Can enter lungs and cause damage. May cause nervous system effects. Breathing vapors may cause drowsiness and dizziness. Causes redness and pain to the skin and eyes.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Methanol	Flammable Irritant Poison	200 ppm TWA	Methanol evaporates at room temperature. Inhalation, ingestion and/or eye and skin contact can all possibly cause light-headedness, nausea, headache, and drowsiness. Prolonged exposure can lead to permanent blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Mercury	Corrosive Irritant Highly Toxic	0.05 mg/m3 TWA	May be fatal if inhaled. May cause respiratory tract irritation. May be harmful if absorbed through skin. May cause skin irritation.
Methylene Chloride	Irritant Carcinogen	25ppm TWA 125ppm STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

NOTE: All glassware used in this procedure is cleaned following SOP# DV-OP-0004. In addition, the glassware is rinsed with methylene chloride immediately prior to use.

NOTE: Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by GC/CI/MS/MS must be concentrated in glassware designated for that method. K-D flasks, glass funnels, concentrator tubes, and snyder columns will be clearly marked and segregated for this purpose.

- Kuderna-Danish (K-D) flasks.
- Concentrator tubes for K-D flasks, un-graduated, approximately 10 mL.
- Concentrator tubes for K-D flasks, graduated at 1mL, calibration checked before use following the steps detailed in DV-QA-0008.
- Snyder columns, 3-ball with ground glass joints at top and bottom
- Manual, adjustable positive-displacement pipette and bottle-top re-pipettor, used to dispense 1 to 20 mL. Calibration is checked following the steps detailed in DV-QA-0008.
- Extract Storage Vials – variety of sizes, clear and amber

- Pasteur pipettes – 6 inch and 9 inch in length.
- Stem-less glass funnels
- Glass wool, baked at 400°C for four hours.
- Boiling Chips – contaminant free, approximately 10/40 mesh Teflon®, PTFE. For concentrating extracts to a final volume greater than 1mL.
- Boiling Chips – contaminant free, carborundum #12 granules, for concentrating extracts to a 1mL final volume. These boiling chips are sufficiently small as to not add any error to the 1mL final volume.
- Solvent Recovery System – includes re-circulating chiller, set at 5°C, cooling condensers, Teflon® PTFE tubing and In-Process Tanks with quick-connect attachments
- S-Evap, thermostat controlled water bath
- N-Evap, thermostat controlled water bath with regulated nitrogen supply

7.0 Reagents and Standards

Reagents - All materials must be reagent grade or higher quality, unless otherwise specified

7.1 Methylene Chloride

Each lot of solvent is tested following CA-Q-S-001 or before it is put into use. QA personnel post the list of approved lots at solvent storage areas. For solvents packaged in CYCLETAINERS, that have not been previously tested per CA-Q-S-001, the first batch of samples prepared with a new lot of solvent is monitored and reported to the QA group per the instructions in CA-Q-S-001 DV-1. If any problems are identified, use of the solvent is suspended until further testing can be done and determines the solvent is acceptable.

7.2 Hexane

For solvents packaged in bottles, each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas. For solvents packaged in CYCLETAINERS, the first batch of samples prepared with a new lot of solvent is monitored and reported to the QA group per the instructions in CA-Q-S-001 DV-1. If any problems are identified, use of the solvent is suspended until further testing can be done and determines the solvent is acceptable.

7.3 Methanol, HPLC Grade

Each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.4 Acetone

Each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.5 Acetonitrile

Each lot of solvent is tested following CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.6 Baked Sodium Sulfate, 12-60 mesh
Heat sodium sulfate in a 400 °C oven for at least four hours. Each lot is tested as described in SOP CA-Q-S-001-DV-1

7.7 Sulfuric Acid, Concentrated –
For use in PCB extract clean-up.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample extracts waiting to be concentrated are stored refrigerated at 4°C ± 2°C in glass bottles or flasks and capped with Teflon-lined lids or aluminum foil. Final sample extracts are stored in glass vials with Teflon-lined lids. See Table 3 for details on storage vial types. Final concentrated extracts are stored refrigerated at 0°C – 6°C. Extracts have a holding time of 40 days from the date of extraction to the date of analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in DV-QA-003P, Quality Assurance Program.

Specific QC requirements for Federal programs, e.g., Department of Defense (DoD) Department of Energy (DoE), AFCEE etc., are desribed in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies
Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for batches of aqueous samples consists of reagent water, and for batches of soil samples, consists of Ottawa sand, both of which are free of any of the analyte(s) of interest. The method blank for batches of TCLP and SPLP leachates consists of leach fluid. The method blank is processed and analyzed just as if it were a field sample.

Acceptance Criteria: The result for the method blank must be less than the reporting limit for the analyte(s) of interest or less than 10% of the analyte concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: If target analytes in the blank exceed the acceptance limits, an unacceptable method blank must be re-prepared and reanalyzed. If the analyte was <u>not</u> detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.5 Laboratory Control Sample (LCS)

At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of reagent water to which the analyte(s) of interest are added at known concentration. For soil sample batches, the LCS consists of Ottawa sand to which the analyte(s) of interest are added at a known concentration. For TCLP and SPLP leachates, the LCS consists of leach fluid to which the analyte(s) of interest are added at known concentration. The LCS is carried through the entire analytical procedure just as if it were a sample.

EPA Methods 608, 610, and 625 require a LCS at a 10% frequency. In other words, one LCS is required for a batch of 10 or less samples. A LCSD is required for a batch of 11 or more samples.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at $\pm$ 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at $\pm$ 4 standard deviations around the mean of

historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Note: Marginal exceedances are not allowed for South Carolina work.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

EPA Methods 608, 610, and 625 require one matrix spike for every 10 samples. If the batch has more than 10 samples, then two matrix spikes must be performed. The two matrix spikes are to be performed on two different samples.

If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at ± 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Critical Procedural Considerations

10.2.1 As stated throughout this SOP, analysts must review Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).

10.2.2 Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces should be cleaned or disposed of before coming into contact with the sample.

10.2.3 According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to Attachment 3 for the appropriate final solvents and final volumes.

10.3 Refer to Attachment 3 to determine if the extract is to be concentrated by the Kuderna-Danish / N-Evap method described in Section 10.4 and 10.5, or the Turbo-Vap method described in Section 10.6

10.4 Concentration by the Kuderna-Danish Method

10.4.1 Refer to Attachment 3. If the extract is to be concentrated to a 1mL final volume, use a 1mL graduated concentrator tube. For extracts that are to be concentrated to any other final volume, use an un-graduated concentrator tube.

10.4.2 Assemble the Kuderna-Danish concentrator by attaching the appropriate concentrator tube to the 500 mL K-D flask with a clip. Make sure the attachment is firm by twisting the concentrator tube at the joint while wearing cut-resistant gloves. Refer to Attachment 4 for configuration of the Kuderna-Danish concentrator.

NOTE: Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by GC/CI/MS/MS must be concentrated in glassware designated for that method. K-D flasks, concentrator tubes, and snyder columns will be clearly marked and segregated for this purpose.

10.4.3 Rinse the apparatus with methylene chloride. Discard the rinse solvent into the appropriate waste container. Care should be taken to ensure all surfaces of the glass are coated with solvent.

10.4.4 If the extract is to be concentrated to a 1mL final volume, add 2-3 carborundum granules to the K-D concentrator. If the extract is to be concentrated to a final volume greater than 1mL, add 1-2 Teflon® boiling chips to each K-D concentrator.

10.4.5 If the sample extracts have not been filtered through sodium sulfate at the time of extraction, or if the sample extract has visible water, then the extract must be dried at this point. Plug a glass funnel with baked glass wool and add baked sodium sulfate. Rinse the funnel and the sodium sulfate with methylene chloride and place it on top of the K-D. During the quantitative transfer in section 10.4.6 the extract will be filtered through the sodium sulfate.

NOTE: Glass wool dust is a carcinogen and therefore glass wool should only be handled in a hood to avoid inhaling any glass particles. Once covered with sodium sulfate, it can be removed from the hood.

10.4.6 Quantitatively transfer the sample extract to the K-D flask. Transfer the sample label to the K-D flask. Perform a quantitative transfer of the extract by rinsing the sample extract container with methylene chloride and adding the rinse solvent to the K-D. If the extract is being filtered through sodium sulfate, be sure to rinse the sodium sulfate well to ensure no target compounds are left on the sodium sulfate. Allow the solvent to drain from the sodium sulfate into the K-D flask then discard the sodium sulfate.

10.4.7 Turn a three-ball Snyder column upside down and rinse with methylene chloride, then rinse the bottom joint with methylene chloride. Attach the Snyder column to the top of the K-D concentrator as shown in Attachment 4.

10.4.8 Place the K-D concentrator on a water bath so that the tip of the receiver tube is submerged. The water level should not reach the joint between the concentrator tube and the K-D flask. Refer to Attachment 3 for the correct water bath temperature. Record both the observed and the corrected temperature on the benchsheet.

10.4.9 For extracts that are methylene chloride or 50/50 methylene chloride/acetone, attach the solvent recovery system tube to the top of the Snyder column. At the appropriate rate of distillation, the balls will actively chatter but the chambers should not flood.

NOTE: For extracts for analysis for low-level NDMA by GC/CI/MS/MS, the solvent recovery system will not be used to avoid possible contamination.

NOTE: At this time, set a timer for no longer than 30 minutes as a reminder to check the in-process solvent tanks.

10.4.10 If the method does not require a solvent exchange~~,~~ skip to Section 10.4.12. If the method requires a solvent exchange, continue on to Section 10.4.11.

10.4.11 If the method requires a solvent exchange at this time, detach the solvent recovery system tube from the top of the Snyder column and add the appropriate exchange solvent through the top of the Snyder column. The exchange solvent should be added when the extract has concentrated to a level that it forms a quarter-sized pool of solvent in the bottom of the K-D. Refer to Attachment 3 for details of exchange solvents and volumes. Mark the K-D flask and sample label to indicate the exchange has been performed. There is no need to re-attach the solvent recovery system at this time as the majority of the methylene chloride has already been evaporated and collected.

10.4.12 Continue to concentrate the sample on the water bath back down to 10-15 mL, or just below the K-D and concentrator tube joint. At this point the boiling sample is just barely splashing above the top of the receiver tube.

NOTE: It is very important not to concentrate to dryness as analytes will be lost. Also, some of the analyses, especially for 8270 and 8015, are especially temperature sensitive and the sample should be taken off the water bath as soon as possible to avoid losing analytes. Also the 8081 surrogate TCMX is also fairly volatile and can be lost if the extract is allowed to concentrate too low either before or after hexane exchange.

10.4.13 Remove the K-D concentrator from the water bath. Rinse the

Snyder column down with a minimal amount of solvent. If the extract was exchanged, use the exchange solvent to perform the rinse, otherwise use methylene chloride.

10.4.14 Allow the extract to cool to room temperature, about 10 minutes.

10.4.15 After the extract is allowed to cool, if the level of the extract is above the level of the concentrator tube joint, add a fresh boiling chip and return the K-D concentrator to the water bath.

10.4.16 After the extract is cool, remove the snyder column. Remove the clip holding the K-D flask and concentrator tube together. Use a Kim-wipe to dry the water off of the joint area so that water does not get into the extract. Remove the concentrator tube from the K-D flask and rinse the lower K-D flask joint into the concentrator tube with methylene chloride or the appropriate exchange solvent.

10.5 Nitrogen Evaporation (N-Evap) to Final Concentration.

10.5.1 N-evap needles should be cleaned weekly by soaking overnight in methylene chloride. This is documented in the N-evap needle log-book.

10.5.2 At the beginning of each shift, the N-evap needles should be wiped clean with a Kim-wipe soaked in methylene chloride to remove any potential contamination. If a needle comes in contact with an extract, then it needs to be cleaned before being used on the next extract.

10.5.3 Place the concentrator tube on the nitrogen evaporator. The temperature of the water bath should be at least 5 °C below the boiling temperature of the solvent being evaporated (See Attachment 2). Lower the needle down to the sample so that a small dimple forms on the surface of the solvent. The stream of nitrogen should be gentle enough that it does not cause the extract to splash.

10.5.4 During the course of the evaporation, rinse the sides of the concentrator tube with approximately 1 mL of clean solvent. The rinse should occur when the solvent gets close to the final volume. Concentrate the solvent to just below the final volume and remove from the nitrogen evaporator.

10.5.5 Transfer the extract into the appropriate vial. Refer to Attachment 3 for the appropriate final volume and correct vial.

10.5.5.1 If the extracts are to have a final volume of 1mL, they should be in 1mL graduated concentrator tubes. Using a Pasteur pipette, add the appropriate solvent to the tube until the extract meniscus reaches the 1mL gradation. Then using the Pasteur pipette transfer the extract to a labeled 2mL amber glass vial.

10.5.5.2 For extracts with a final volume greater than 1mL, the vials should be calibrated using the manual, adjustable positive-displacement pipette or bottle-top re-pipettor. Pipette the correct

volume of clean solvent into the vial and mark the bottom of the meniscus with a thin marker. Discard the solvent. Transfer the extract into the vial using a Pasteur pipette and rinse the concentrator tube with solvent. Transfer the rinse to the vial. Bring the meniscus of the solvent up to the marked line. Cap with a Teflon-lined cap.

NOTE: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

NOTE: Some extracts might not concentrate down to the required final volume. If the extract is very dark and viscous, or an oil layer or precipitate starts to form, a higher final volume can be used. This should be documented in an NCM.

10.6 TurboVap Method

10.6.1 Turn on the TurboVap and adjust the water temperature to 40 °C. Turn the nitrogen supply on. Record both the observed and the actual temperature on the benchsheet.

10.6.2 Switch the endpoint sensor to “Manual”.

10.6.3 Adjust the water bath level. The water level should be above the extract level.

10.6.4 Turn on the nitrogen gas and adjust the gas pressure to approximately 12 psi. Lower pressure may be used if needed to prevent samples from splashing out of the TurboVap tubes.

10.6.5 Rinse the TurboVap tube with methylene chloride or the solvent the extract is in. Discard the waste.

10.6.6 Transfer the sample to the TurboVap tube. For 8141 soils extracted by soxhlet, dry the extract first by filtering through a funnel with baked sodium sulfate. Rinse the sample extract container with clean solvent and transfer to the TurboVap tube. Do not fill the TurboVap tubes over the fill line or approximately ¾ full.

10.6.7 Place the TurboVap tube into the TurboVap and turn on nitrogen to the position the tube is in.

10.6.8 Close the lid. You should be able to see the sample extracts swirling in the tubes**.**

NOTE: If the extract splashes when the nitrogen flow starts, transfer a portion of the extract back into the original extract container, or lower the gas pressure.

10.6.9 As the extract concentrates, transfer the remainder of the extract in to the

appropriate Turbovap tube. Rinse the sample container with a few milliliters of methylene chloride or appropriate solvent and transfer to the Turbovap tube.

10.6.10 During the concentration rinse the Turbovap tube walls with a few milliliters of solvent 1 or 2 times.

10.6.11 If a solvent exchange is required, concentrate to about 5 mL and add the exchange solvent. After the exchange solvent is added, swirl the extract to make sure the extract is well mixed. Concentrate back down to slightly less than the appropriate volume. Refer to Attachment 3 for details of exchange solvents and final volumes.

10.6.12 Transfer the extract into the appropriate vial.

10.6.12.1 Currently, the TurboVap is only used to concentrate extracts with final volumes greater than 1mL. Ask the QA Manager or the supervisor for guidance if a project requires a 1mL final volume by TurboVap.

10.6.12.2 For extracts with a final volume greater than 1mL, the vials should be calibrated using the manual, adjustable pipette or bottle-top re-pipettor. Pipette the correct volume of clean solvent into the vial and mark the bottom of the meniscus with a thin marker. Discard the solvent. Transfer the extract to the vial using a Pasteur pipette and rinse the concentrator tube with solvent. Transfer the rinse to the vial. Bring the meniscus of the solvent up to the marked line. Cap with a Teflon-lined cap.

10.6.12.3 Rinse the Turbovap tube with methylene chloride 2-3 times before washing. Turbovap tubes are not baked. They are cleaned in accordance with DV-OP-0004. If the Turbovap tubes need to be used again before they are dry, rinse with acetone to dry the Turbovap tube.

10.7 Cleanup Techniques

NOTE: If any sample in a batch requires a clean-up, the batch QC must also undergo the same clean-up technique.

10.7.1 Florisil Cartridge Cleanup

Florisil can be used to remove low-medium molecular weight polar hydrocarbon interfering compounds from pesticide extracts. The laboratory will use Florisil cleanups whenever water extracts have any color, whenever soil extracts have any color darker than a Post-It® Note, or whenever there is clear evidence of interferences, such as significant interfering peaks in the RT range for the target pesticide compounds or failing sample surrogate recoveries. Extracts that are to be analyzed for kepone will not be florisil cleaned, because florisil will remove kepone from the extract.

NOTE: Florisil cartridge performance checks are conducted for every lot of Florisil before use. Add 1.0 mL of the Florisil check solution described in Attachment 5 to a pre-rinsed Florisil cartridge. Following the procedure described below, load and elute the 1mL of check solution through the Florisil cartridge. Bring the final volume back down to 1.0 mL in hexane. The test sample must show 80-120 % recovery of all pesticide analytes with < 5% trichlorophenol recovery, and no peaks interfering with target compounds can be detected.

10.7.1.1 Clean the manifold and ports

Prior to each use, the top and underside of the manifold lid must be wiped down with hexane and a Kim-wipe to prevent any cross-contamination. The manifold ports must be dis-assembled and placed in a jar with fresh acetonitrile, in a sonication bath for a minimum of 30 minutes. The jar used in the soak and sonication of the ports must be replaced weekly to ensure it does not spread contamination. This is documented in the Organic Extraction Weekly Cleaning Logbook.

10.7.1.2 Place one Florisil cartridge into the vacuum manifold for each sample extract. Make sure all valves are closed.

10.7.1.3 Add 5 mL of hexane to each cartridge.

10.7.1.4 Slowly open the valves to allow a few drops of hexane to pass through, then close the valve and allow the hexane to soak the cartridge for at least 5 minutes.

10.7.1.5 Slowly open the valves again and allow the hexane to drain through the cartridge but close the valve when the solvent level is right above the glass frit. Do not allow the cartridges to go dry. If cartridges go dry, repeat the conditioning step.

10.7.1.6 Remove the manifold top and place one clean, labeled 16 × 125 mm disposable glass test tube in each position for each of the samples. Replace the vacuum manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.

10.7.1.7 Add exactly 2.0 mL of the concentrated extract to the appropriate Florisil cartridge. Turn the valve to the on position.

10.7.1.8 Allow the extract to gravity drip through the cartridge. The flow through the cartridges should be approximately 2 mL/minute.

10.7.1.9 Just before the Florisil cartridge goes, dry add 5 mL of hexane:acetone (90:10). Allow this to pass through the cartridge, then just before it goes dry again, add another 5 mL of hexane:acetone (90:10).

10.7.1.10 Allow the Florisil cartridge to go dry after the second addition of hexane:acetone (90:10). Turn the vacuum pump on after all of the cartridges have gone dry to recover any remaining solvent.

10.7.1.11 Remove the tubes from the vacuum manifold and concentrate them back down to just below 2.0 mL on the nitrogen evaporator. Quantitatively transfer the extract to a 4mL vial that has been calibrated to hold 2.0mL and bring the extracts up to the 2.0 mL calibration mark with hexane.

10.7.1.12 Discard the used cartridges.

10.7.2 Sulfur Removal

Sulfur can be removed by one of three methods: mercury, copper, or tetrabutylammonium sulfite (TBA), according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

NOTE: Some programs (e.g., South Carolina) do not allow the use of elemental mercury. Copper or TBA will be used as an alternative.

10.7.2.1 Sulfur Removal with Elemental Mercury

NOTE: Use Mercury in a hood and sparingly in order to minimize exposure and disposal costs.

10.7.2.1.1 Transfer approximately 2 mL of sample extract into a clean Teflon-sealed vial.

10.7.2.1.2 Add one to three drops of mercury to the extract vial and seal.

10.7.2.1.3 Shake well for 15-30 seconds. If prolonged shaking is required, use a mechanical shaker.

10.7.2.1.4 Remove the extract from the mercury using a disposable pipette and transfer to a clean vial.

10.7.2.1.5 If the mercury turns black, sulfur was present. Decant or pipette off the extract to a clean vial and repeat the procedure by adding one to three drops of fresh mercury. Do this until the mercury does not turn black.

10.7.2.1.6 If the extract is cloudy, filter the extract through a 1um disposable syringe filter.

10.7.2.1.7 Properly dispose of the mercury waste.

10.7.2.2 Sulfur Removal with Copper Powder

NOTE: This technique requires the copper powder to be very reactive, as demonstrated by a bright and shiny appearance. A pre-cleaned, activated copper may be purchased from a valid vendor. If manual preparation of reactive copper is performed, take care to remove all traces of acid in order to prevent degradation of some analytes.

10.7.2.2.1 Weigh out copper into a 20ml VOA VIAL assuming two grams of copper needed per sample.
10.7.2.2.2 Remove oxides by treating with 10% nitric acid.
10.7.2.2.3 Rinse the copper with DI organic-free water three times to remove all traces of acid.
10.7.2.2.4 Rinse the copper with acetone and dry under a stream of nitrogen.
10.7.2.2.5 Add approximately 2 grams of the copper powder to a 2ml vial with approximately 1ml of sample extract and shake vigorously on a mechanical shaker for at least one minute.
10.7.2.2.6 After phase separate, draw off extract and transfer to a clean vial.

10.7.3 Sulfuric Acid Cleanup

10.7.3.1 Add 1 mL of concentrated sulfuric acid to approximately 1 mL of sample extract in a Teflon capped vial.

CAUTION: There must be no water or acetone present in the extract or the reaction may shatter the sample container.

10.7.3.2 Vortex for about 5 seconds and allow to settle. (Centrifuge if necessary)

10.7.3.3 Remove the sample extract (top layer) from the acid using a Pasteur pipette and transfer to a clean vial.

CAUTION: It is not necessary to remove all the extract since the final volume is already determined. Transferring any amount of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column

10.7.3.4 If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.

10.7.3.5 Properly dispose of the acid waste.

11.0 Calibration

Not applicable to this procedure. See the determinative methods for calibration of the analytical instrumentation.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Before analyzing samples, the laboratory must establish a method detection limit (MDL). The laboratory also operates under programs that require instrument detection limits (IDLs). See DV-QA-005P, "Determination of Method Detection Limits", for more information on the method detection limit studies.

12.2 Demonstration of Capabilities

An initial demonstration of capability (IDOC) must be performed by each analyst. On-going proficiency must be demonstrated by each analyst on an annual basis. See SOP DV-QA-0024, "Employee Training", for more information on the IDOCs.

12.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and who has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health & Safety Manual, and DV-HS-001P, "Waste Management Plan."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methylene chloride – Waste Stream B
14.2.2 Flammable Solvents – Waste Stream C
14.2.3 1:1 MeCl2:Acetone – Waste Stream CA
14.2.4 Solid waste/sodium sulfate – Waste Stream D

14.3 Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

15.1.1 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

15.1.2 Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.

15.1.3 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

15.1.4 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

15.1.5 Method 3540C, Soxhlet Extraction, Revision 3, December 1996.

15.1.6 Method 3546, Microwave Extraction, Revision 0, February 2006.

15.1.7 Method 3620C, Florisil Cleanup, Revision 3, February 2007.

15.1.8 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

15.1.9 Method 3660A, Sulfur Cleanup, Revision 1, July 1992.

15.1.10 Method 3665A, Sulfuric Acid/Permagante Cleanup, Revision 1, December 1996.

15.2 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater

15.2.1 Method 608, Organochlorine Pesticides and PCBs.

15.2.2 Method 610, Polynuclear Aromatic Hydrocarbons.

15.2.3 Method 614, The Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater

15.2.4 Method 625, Base/Neutrals and Acids.

16.0 Method Modifications:

16.1 Method 3665A calls for the clean-up to be performed using 1:1 Sulfuric Acid:H2O. This procedure calls for the clean-up to be performed using concentrated sulfuric acid.

Attachments

Attachment 1: Determinative and Extraction Methods Used in Conjunction with this SOP.
Attachment 2: Boiling Points of Solvents
Attachment 3: Concentration Summary
Attachment 4: Kuderna-Danish Concentrator
Attachment 5: Florisil Check Solution

17.0 Revision History

- Revision 7 dated 30 November 2012
  - Section 5 and Section 10.4.5 were revised to instruct the analysts to handle glass wool in a hood to avoid breathing in the dust.
  - Revised Section 10.4.8 to instruct the analysts to document both the observed and corrected temperatures.
  - Section 10.7.1.11 was revised to describe in more detail how the florisiled extracts are taken to the 2 mL final volume.
  - Section 14.2 was revised to include the waste stream for 1:1 MeCl2:Acetone – Waste Stream CA.
  - Attachment 1 was revised to include DV-OP-0015 as an acceptable extraction for Diesel Range Organics.
  - Attachment 3 was revised to include details on 8081/3510_LL concentration steps.
- Revision 6.0 dated 14 October 2011
  - The procedure was revised to remove instructions on how to concentrate and clean up extract for method 8070 and 607. TestAmerica Denver no longer supports these methods.
  - Section 1.3 was corrected to give the correct SOP number to Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C for Low-Level NDMA by GC/CI/MS/MS.
  - Section 7.5 was revised to state acetonitrile is tested before use. Previously this solvent was not tested before use.
  - The procedure was revised to include instructions that all extracts for analysis by method 8081, 8082, or 608 to be hexane exchanged only after concentration on the S-Evap. Previously the SOP instructed analysts to add the hexane exchange before the S-Evap for extracts that were concentrated by microwave extraction. This resulted in poor hexane exchanges, therefore the extracts are now concentrated before the exchange.
  - The procedure was revised to instruct analysts not to use the solvent recovery system when concentrating samples for analysis of low-level NDMA by GC/CI/MS/MS. This was done to eliminate a possible source of contamination in this ppt level analysis.
  - The procedure was revised to instruct analysts to use concentrated sulfuric acid in the acid clean up of PCB extracts.
  - The procedure was revised to clarify the exact steps used in the sulfur removal with mercury.
- Revision 5 dated 07/20/10
  - Note added to section 9.5 to not allow marginal exceedances for South Carolina work.
  - Updated to reflect changes to the LIMS system.
  - Updated Attachment 1 and Section 1.3 to include the most recent extraction and analysis SOPs.

    - Added procedures to concentrate microwave extracts by K-D.
- Revision 4, dated 26 August 2009
    - Added instructions on the concentration of extracts from microwave extraction, SW846 3546.
    - Added clarification that the solvent recovery system is only to be used with extracts containing methylene chloride.
    - Added instructions on the use of 1mL graduated concentrator tubes to determine 1mL final volumes.
    - Changed the required temperature of the re-circulating chiller used in the solvent recovery system from 10°C to 8°C.
    - Added instructions on how to properly clean the manifold and valves used in florisil clean-up.
    - Added guidance on when samples should be taken through the florisil clean-up.
    - Change to the use of 1:1 Sulfuric Acid in the clean-up procedure.
- Revision 3.1, dated 10 October 2008
    - Added references to method 3550C throughout SOP.
- Revision 3, dated 25 April 2008
    - Integration for TestAmerica and STL operations

Attachment 1.

Determinative and Extraction Methods Used in Conjunction with this SOP

Method Description	Determinative Method	Determinative Method SOP	Extraction Method	Extraction Method SOP
Diesel Range Organics & Jet Fuels	SW-846 8015B, 8015C, California LUFT Method, & AK102 & AK103, NW-TPH, OK DRO	DV-GC-0002 DV-GC-0027	WATER: SW-846 3510C, AK102 AK103 NW-TPH OK DRO SOIL: SW-846 3550B/C SW-846 3546 AK102, AK103 NW-TPH OK DRO	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Chlorinated Pesticides	SW-846 8081A, 8081B & EPA Method 608	DV-GC-0020 DV-GC-0016 DV-GC-0026	WATER: SW-846 3510C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Polychlorinated Biphenyls	SW-846 8082, 8082A EPA Method 608	DV-GC-0021 DV-GC-0016 DV-GC-0030	WATER: SW-846 3510C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Organo-phosphorus Pesticides	SW-846 8141A, 8141B, & EPA Method 614	DV-GC-0017	WATER: SW-846 3510C SOIL: SW-846 3540C	WATER: DV-OP-0006 SOIL: DV-OP-0010
Polynuclear Aromatic Hydrocarbons	SW-846 8310 & EPA Method 610	DV-LC-0009	WATER: SW-846 3510C SOIL: SW-846 3550B/C	WATER: DV-OP-0006 SOIL: DV-OP-0016
Semi-volatiles by GC/MS	SW-846 8270C, 8270D & EPA 625	DV-MS-0011 DV-MS-0012	WATER: SW-846 3510C SW-846 3520C SOIL: SW-846 3550B/C	WATER: DV-OP-0006 or DV-OP-0008 SOIL: DV-OP-0016
Low-Level Semi-Volatiles by GC/MS	SW-846 8270C	DV-MS-0011	WATER: SW-846 3520C	WATER: DV-OP-0008
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270C SIM	DV-MS-0002	WATER: SW-846 3520C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0008 SOIL: DV-OP-0016 or DV-OP-0015
n-Nitrosodimethylamine by GC/CI/MS/MS	SOP	DV-LC-0019	WATER: SW-846 3520C SOIL: SW-846 3550B/C	WATER: DV-OP-0021 SOIL: DV-OP-0016
Extended List PAHs by GC/MS SIM for CSLP and Full Scan	SW-846 8270C	DV-MS-0005	WATER: SW-846 3520C	WATER: DV-MS-0005, Appendix II

Attachment 2.

Boiling Points of Solvents

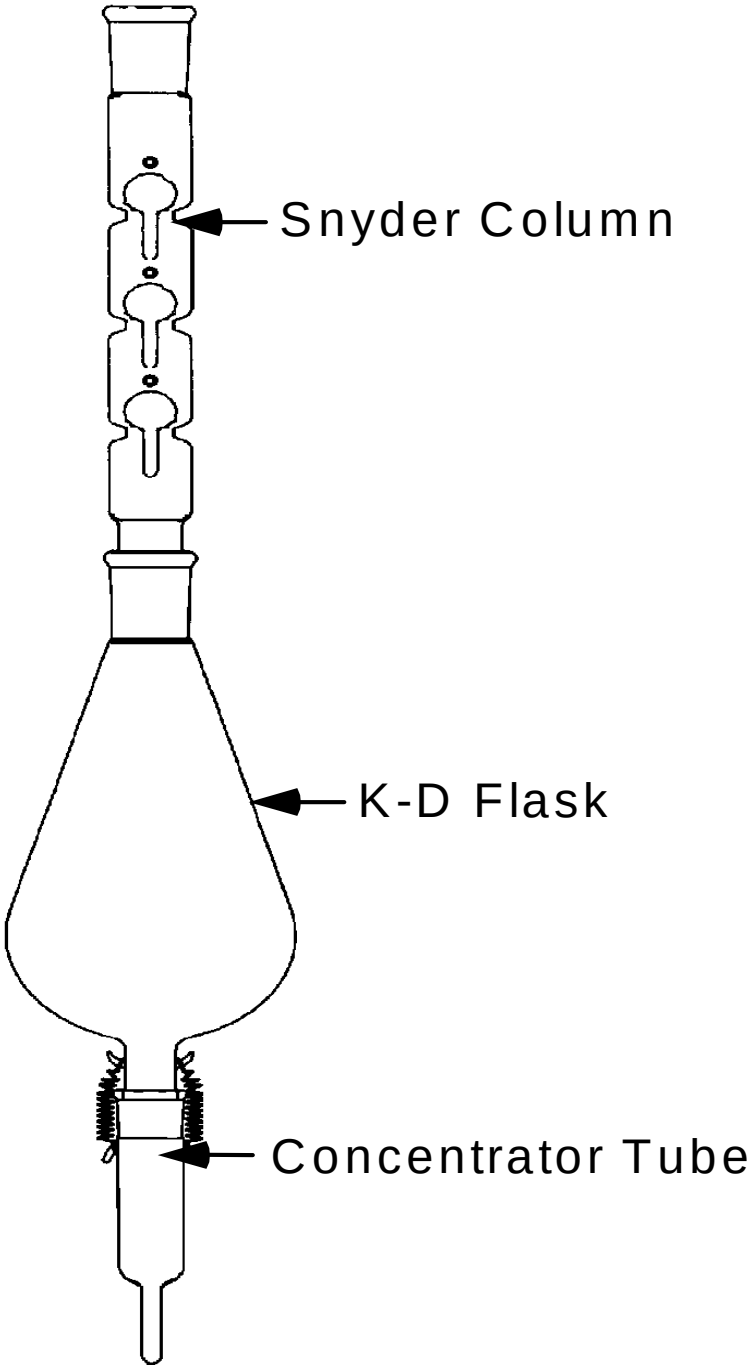
Solvent	Boiling Point (°C)
Methylene chloride	40
Acetone	56
Hexane	69
Methanol	65
Acetonitrile	82

Attachment 3 - Concentration Summary
(This table is included in controlled Work Instruction WI-DV-009)

Concentration Summary								
Analytical Method	**Matrix**	**Extraction Solvent**	**Bath Temp (C)**	**Exchange Solvent**	**Exchange Vol. (mL)**	**Final Vol. (uL)**	**Vial Type**	**Cleanup Steps**
625 8270C 8270D 8270_AFCEE 8270_DoD 8270C_UTS 8270C_FS_CSLP 8270C_SIM 8270D_SIM 8270_SIM_AFCEE 8270_SIM_DoD 8270C_SIM_CSLP 8270C_SIM_LL	Soil	$MeCl_2$:Acetone	S-Evap 88			1000	2 mL amber	
	Water and Leachate	$MeCl_2$	S-Evap 84			1000	2 mL amber	
8270C_LL	Water	$MeCl_2$	S-Evap 84			2000	4 mL amber	
8015B_DRO 8015C_DRO 8015D_DRO AK102_103 NWTPH_Dx Okla_DRO 8015B_Terp	Water or Soil	$MeCl_2$	S-Evap 84			1000	2 mL amber	
NDMA_CIMSMS	Soil/Water	$MeCl_2$	S-Evap 84			1000	2 mL amber	Designated glass!
608 8081A 8081B 8082 8082A	Soil by 3550C or 3546	$MeCl_2$:Acetone	S-Evap 88	Hexane	50	10,000	12 mL clear	Florisil if needed
	Soil by 3550C_LL or 3546_LL	$MeCl_2$:Acetone	S-Evap 88	Hexane	50	5000	12 mL clear	Florisil if needed
	Wipes by 3550C or 3546	Hexane	S-Evap 88	NA		10,000	12 mL clear	Florisil if needed
	Water by 3510C	MeCl2	S-Evap 88	Hexane	50	10,000	12 mL clear	Florisil if needed.
	8082 Water by 3510C_LL	$MeCl_2$	S-Evap 88	Hexane	50	1000	2 mL amber	
	8081 Water by 3510C_LL	$MeCl_2$	S-Evap 88	Hexane	50	5000	12 mL clear	Florisil if needed
610 8310	Soil	$MeCl_2$:Acetone	S-Evap 88	ACN	10	4000	4 mL amber	
	Water	$MeCl_2$	S-Evap 88	ACN	10	1000	2 mL amber	
614 8141A 8141B	Soil	$MeCl_2$:Acetone	Turbo-Vap 40	Hexane	50	2000	4 mL amber	
	Water	$MeCl_2$	Turbo-Vap 40	Hexane	50	2000	4 mL amber	
8321A_Herb	Soil	Ether/Acetone	Turbo-Vap 40	ACN	2	5000	8 mL amber	Enter in 10mL Vf

Attachment 4.

Kuderna-Danish Concentrator



Attachment 5.

Florisil Check Solution
Prepared in Hexane

Compound	Concentration
2,4,5-Trichlorophenol	0.1ug/mL
Alpha-BHC	0.05ug/mL
Alpha-Chlordane	0.05ug/mL
Aldrin	0.05ug/mL
Beta-BHC	0.05ug/mL
Dieldrin	0.05ug/mL
Endosulfan I	0.05ug/mL
Endosulfan II	0.05ug/mL
Endosulfan sulfate	0.05ug/mL
Endrin	0.05ug/mL
Endrin Aldehyde	0.05ug/mL
Endrin Ketone	0.05ug/mL
Gamma-BHC	0.05ug/mL
Gamma-Chlordane	0.05ug/mL
Heptachlor	0.05ug/mL
Heptachlor expoxide	0.05ug/mL
Methoxychlor	0.05ug/mL
4,4-DDD	0.05ug/mL
4,4-DDE	0.05ug/mL
4,4-DDT	0.05ug/mL

Electronic Copy Only

Title: Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607

Approvals (Signature/Date):

Susan Decker 7.28.10
Susan Decker Date
Organic Extractions Manager

Adam W. Alban 28 July 10
Adam Alban Date
Health & Safety Manager / Coordinator

Karen Kuoppala 7-28-10
Karen Kuoppala Date
Quality Assurance Manager

Robert C. Hanisch 7/30/10
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

This Standard Operating Procedure (SOP) is applicable to the solvent extraction of organic compounds from aqueous samples, TCLP leachates, and SPLP leachates using a continuous liquid/liquid extractor (CLLE). This SOP is based on SW-846 Method 3520C and EPA Methods 625 and 607.

The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate pH and spiking mixtures are used.

This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, "Concentration and Clean-up of Organic Extracts", for details concerning the concentration and cleanup of extracts.

This procedure does not include the extraction of samples for low-level NDMA analysis by GC/CI/MS/MS. The CLLE extraction procedure utilized for that analysis is described in DV-OP-0020.

2.0 Summary of Method

A measured volume of sample, usually 1 liter, is placed in a continuous liquid/liquid extractor (CLLE). The pH is adjusted, as required, for the efficient extraction of specific compounds. The organic compounds are extracted with methylene chloride for 18 to 24 hours. A second extraction at a different pH may be also required. The water phase is discarded.

3.0 Definitions

3.1 Aliquot: A part which is a definite fraction of a whole; as in a "sample aliquot for testing or analysis." "Aliquot" is also used as a verb meaning to take all or part of a sample for preparation, extraction, and/or analysis.

3.2 Extraction Holding Time: The elapsed time, expressed in days, from the date of collection of the sample to the date of extraction, i.e., the date solvent comes in contact with the sample. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.3 Preparation Batch: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards.

3.4 Quality Assurance Summary (QAS): Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the special instructions/client requirements field in LIMS. In those situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all laboratory employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.3 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.4 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. Especially take note of the possibility of phthalate contamination from gloves. Gloves should be changed out frequently and whenever they come in contact with solvent. Glassware should be handled in a fashion that keeps gloves away from the interior and mouth of the glassware.

4.5 The decomposition of some analytes has been demonstrated under basic extraction conditions. Organochlorine pesticides may dechlorinate, phthalate esters may exchange, and phenol may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510C. Method 3510C is preferred over Method 3520C for the analysis of these classes of compounds. However, using Method 3520C and performing the initial extraction at the acid pH optimizes the recovery of phenols.

4.6 The recovery of some target analytes and the surrogate 2-fluorobiphenyl can be reduced if proper care isn't taken to target the pH during the base extraction to 11-12. It is not recommended to add excess base that will cause the sample pH to be greater than 12.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 CLLE glassware is often times fragile and awkward to use. Care should be taken to prevent breakage and injury. During the course of the procedure it might be necessary to adjust the height of the heating mantle. This will cause the CLLE body to tilt. Do not tilt the CLLE body more than 15 degrees to avoid stressing the glass and making the CLLE unstable. See Attachment 1 for troubleshooting.

5.1.2 The procedure requires that the ground glass joints are fitted tightly to prevent loss of the extract. Cut resistant gloves will be worn when assembling or dis-assembling the ground glass joints to protect against cuts if the glass were to break.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Serious or Significant Hazard Rating

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, & headache. Causes irritation, redness, & pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 mg/m3	Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, and runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and can cause burns that may result in permanent impairment of vision, even blindness with greater exposures.
Sulfuric Acid	Corrosive Carcinogen	1 mg/m3	Inhalation may cause irritation of the respiratory tract with burning pain, coughing, wheezing, shortness of breath, and pulmonary edema. Causes chemical burns to the respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Causes skin & severe eye burns. May cause irreversible eye injury, blindness, permanent corneal opacification.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit

6.0 Equipment and Supplies

NOTE: All glassware used in this procedure is cleaned following SOP DV-OP-0004. In addition, the glassware is rinsed with methylene chloride immediately prior to use.

6.1 Supplies

- Continuous Liquid-Liquid Extractor (CLLE), equipped with ground glass joints and polytetrafluoroethylene (PTFE) stopcock. (See Figure 1.)
- 250mL boiling flask with ground glass joint.
- Boiling Chips, contaminant free, approximately 10/40 mesh, Teflon®, PTFE.
- Cooling Condensers
- Re-circulating Chiller – kept at 5°C to 10°C
- Heating Mantle, Rheostat controlled, or Hotplate with temperature control. If hotplates are used then a metal cup must also be used to even heat the boiling flasks.
- Balance, ≥ 1600 g capacity, accurate to ± 1 g, calibration checked daily per SOP DV-QA-0014.
- pH indicator paper, wide range.
- Class A Graduated Cylinder, sizes ranging from 50 mL to 1 L.
- Teflon® stir rods
- Mechanical pipette, 1 mL, positive displacement, with disposable tips, calibration checked daily per SOP DV-QA-0008.
- Aluminum foil.
- Disposable Pasteur Pipettes – used to take initial pH of sample.

6.2 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

Reagents - All materials must be reagent grade or higher quality, unless otherwise specified

7.1 Reagent Water

Testamerica Denver has three ELGA water purification systems. The water coming from the ELGA system should be 18-18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026.

7.2 Methylene Chloride

Each lot of solvent is tested following Corporate SOP CA-Q-S-001 or TestAmerica Denver SOP CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.3 Acids and Bases

7.3.1 Sulfuric Acid (H2SO4), 1:1

Place an ice water bath on a stir plate. Place a container with a magnetic stir bar in the bath. While stirring, slowly add 1 part concentrated reagent grade sulfuric acid (36N) to 1 part water from the ELGA purification system. Assign a 1 year expiration date from the date made or the vender expiration date, whichever is shorter.

7.3.2 Sodium Hydroxide (NaOH), 10N

Purchased at ready-to-use concentration from commercial vendors. Assign a 1 year expiration date from the date opened or the vender expiration date, whichever is shorter.

7.4 Sodium Thiosulfate, 0.08 g/mL solution

Dissolve 8 grams of sodium thiosulfate in 100mL of water from the ELGA system. Assign a 1 year expiration date from the date made or the vender expiration date, whichever is shorter.

7.5 Baked Sodium Chloride

Bake in 400 °C oven for at least 4 hours. Assign an expiration date of 1 year after date opened, unless vender expiration date is shorter.

Standards - Stock standards are obtained from commercial sources.

7.6 Verification

All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

7.7 Storage Requirements

All standards must be refrigerated at 4 ± 2 °C and protected from light. Standard solutions are allowed to adjust to room temperature before using.

7.8 Expiration Date of Standards

7.8.1 Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of opening or earlier if the vendor indicates an earlier date.

7.8.2 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions.

7.9 Working Spike Solutions for LCS and MS/MSD

7.9.1 If a project requires a non-routine set of spike compounds, the current working spiking standard is reviewed to see if the required compounds are already included. A new standard does not always have to be made if more compounds are included than the project requires. That is to say, the LCS and MS/MSD do not have to be evaluated for all compounds in the spike solution if only a subset of compounds is needed. It depends on how the PM and QA set up the work in LIMS based on client instructions.

7.9.2 TestAmerica Denver's QC Policy DV-QA-003P requires that the same spike solution and spike amount are used for LCSs and MS/MSDs.

7.9.3 The following sections describe the standard solutions used for preparing spikes and surrogates for each determinative method used with this SOP. Except where noted in this SOP or in WI-DV-0009, each standard is designed so that 1 mL of spike solution is added to make the LCS, MS, and MSD, and 1 mL of surrogate solution is added to all samples and QC in the batch.

7.9.4 Standards for Semi-Volatile Organics in Waters and SPLP Leachates by 8270C, 8270D, and 625.

7.9.4.1 8270 standard solutions are prepared in purge and trap grade methanol as shown in Table 2. The standards are given a 1 year expiration date.

7.9.4.2 A separate benzidine spiking solution is prepared in purge and trap grade methanol as described in Table 3. The expiration date for this standard is one week from the date of preparation. 1 mL of this standard is added to every LCS/LCSD and MS/MSD for method 625 as needed. This standard can also be added to method 8270 LCSs upon client request.

7.9.5 Standards for Low-Level Semi-Volatile Organics in Waters by Low-Level 8270C (8270_LL).

7.9.5.1 For LCSs and MS/MSDs by Method 8270_LL, 0.125mL of the Method 625 LCS spiking solution described in Table 2 is added. This produces a sample concentration for each analyte at 10µg/L.

7.9.5.2 A method 8270_LL surrogate solution is prepared in purge and trap grade methanol. It contains the same compounds as the 8270 Surrogate standard described in Table 2 but at concentrations of 5µg/mL and 7.5µg/mL. It is given a 1 year expiration date.

7.9.6 Standards for Semi-Volatile Organics in TCLP Leachates by 8270

7.9.6.1 The method 8270 spiking solution for TCLP leachates is prepared in purge and trap grade methanol as shown in Table 4. The standard is given a 1 year expiration date.

7.9.6.2 TCLP leachates are extracted with the 8270 surrogate solution described in Section 7.9.4.1 and in Table 2.

7.9.7 Standards for Polynuclear Aromatic Hydrocarbons (PAHs) in Waters by 8270 SIM

7.9.7.1 PAH standard spike solution is prepared by performing diluting the Method 8270/625 spike solution described in Table 2 in 90:10 purge and trap methanol : methylene chloride to reach a final concentration of 0.9µg/mL. The standard is given a 1 year expiration date.

7.9.7.2 PAH standard surrogate solution is prepared by performing a 1:200 dilution of the 8270 standard surrogate solution described in Table 2 in 90:10 purge and trap methanol : methylene chloride. The standard is given a 1 year expiration date.

7.9.8 Standards for NDMA by 8070A and 607

7.9.8.1 NDMA standard solutions are prepared in purge and trap grade methanol as described in Table 5. The standards are given a 6 month expiration date.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Amber Glass	1000 mL	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
TCLP Leachates	Glass	200 mL	Cool 4 ± 2°C	7 Days from the completion of the leach	SW-846 1311
SPLP Leachates	Glass	1000 mL	Cool 4 ± 2°C	7 Days from the completion of the leach	SW-846 1312

[1] Exclusive of analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the Method Comments to determine specific QC requirements that apply. See WI-DV-0032 for more information on Method Comments.

The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for batches of aqueous samples consists of 1 L of reagent water, which is free of any of the analyte(s) of interest. The method blank for batches of TCLP leachates consists of 200 mL of leach fluid. The method blank for batches of SPLP leachates consists of 1 L of leach fluid. The method blank is processed and analyzed just as if it were a field sample.

Acceptance Criteria: The result for the method blank must be less than the reporting limit for the analyte(s) of interest or less than 10% of the analyte concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: If target analytes in the blank exceed the acceptance limits, an unacceptable method blank must be re-prepared and reanalyzed. If the analyte was not detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.5 Laboratory Control Sample (LCS)

At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of 1 L of reagent water to which the analyte(s) of interest are added at known concentration. For TCLP leachates, the LCS consists of 200 mL of leach fluid to which the analyte(s) of interest are added at known concentration. For SPLP leachates, the LCS consists of 1 L of leach fluid to which the analyte(s) of interest are added at known concentration. The LCS is carried through the entire analytical procedure just as if it were a sample.

Methods 625 and 607 requires a LCS at a 10% frequency. In other words one LCS is required for a batch of 10 or less samples. A LCSD is required for a batch of 11 or more samples.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be

random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Note: Marginal exceedances are not allowed for South Carolina work.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

Methods 625 and 607 requires one matrix spike for every 10 samples. If the batch has more than 10 samples, then two matrix spikes must be performed. The two matrix spikes are to be performed on two different samples.

If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.7 Surrogate Spikes
Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at $\pm$ 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision and the approval of the Quality Assurance Manager (or designee) to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.1 Critical Procedural Considerations

10.1.1 As stated throughout this SOP, analysts must review Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).

10.1.2 Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any piece of glassware other than the designated one should be cleaned or disposed of before coming into contact with the sample.

10.1.3 If problems arise during the extraction, see Attachment 1: Troubleshooting Guide.

10.2 Assemble and Clean the Glassware Immediately Before Use

10.2.1 For each sample and QC sample, rinse a 250 mL boiling flask with methylene chloride. Add a few boiling chips and fill with about 150 mL of methylene chloride.

10.2.2 For each sample and QC sample, rinse a CLLE once with acetone to ensure the CLLE is dry, and then rinse two times with methylene chloride. Make sure the stop-cock is open during the rinses. Then fill the bottom of the CLLEs with methylene chloride to a level approximately 2 cm above the opening of the solvent flow arm. (See Figure 1.)

10.2.3 Set the CLLE with the stop-cock open on a stand under a slot hood and in front of a heating mantle or hotplate with a metal cup. Attach the 250 mL boiling flask containing methylene chloride and boiling chips to the side arm of the CLLE and place a heating mantle or hotplate with metal cup under the flask.

10.2.4 Cap the CLLE with aluminum foil to prevent contamination or solvent evaporation.

10.2.5 Label each boiling flask with the sample ID or batch QC ID. Also include on the label what fraction (acid or base) if the method calls for extraction at a secondary pH. (See Section 10.13).

10.3 Prepare LCS and Method Blank Samples

NOTE: For SW-846 methods if there is not a MS/MSD pair in the batch then perform a LCS/LCSD. Methods 607 and 625 require a LCS and LCSD in batches of 11 samples or more.

10.3.1 For aqueous sample batches, dissolve approximately 3g (approximately ½ tsp.) of baked NaCl to 1 liter of reagent water. With the stopcock of the CLLE open, pour this water into the CLLEs marked for the LCS's and MB until the methylene chloride in the bottom of the CLLE starts to spill over through the solvent flow arm and into the boiling flask. Be careful not to allow water to spill over. You may need to add additional methylene chloride to the CLLE to prevent water from entering the solvent flow arm.

NOTE: If any sample in the batch is preserved with sodium thiosulfate, then the LCS's and MBs should also contain sodium thiosulfate. Add 1mL of the 0.08 g/mL solution to the water in the CLLE.

10.3.2 For TCLP batches, prepare the LCS's and method blanks using 200 mL of the appropriate leach fluid. For SPLP batches, prepare the LCS using 1 liter of the appropriate leach fluid. Add reagent water as necessary to the CLLE to cause the methylene chloride to begin to pour through the solvent flow arm and into the boiling flask. Place the leachate bottle beside the CLLE so a second analyst can check that the correct blank fluid was used. This is documented on the Organic Extraction Worksheet (WI-DV-0009).

10.4 Prepare the samples and the MS/MSDs

10.4.1 Measure the initial sample pH with wide-range pH paper and record the pH on the extraction bench sheet. If the sample is a leachate, compare the current pH against the pH recorded on the TCLP worksheet. Note any discrepancies in an NCM.

10.4.2 For TCLP leachates, use a Class A graduated cylinder to measure out 200 mL into the labeled CLLE. Add reagent water to the CLLE to ensure proper flow. For SPLP leachates, use a Class A graduated cylinder to measure out 1000mL into the labeled CLLE. Place the leachate container beside the CLLE so that a second analyst can check the labels. This is documented on the Organic Extraction Worksheet (WI-DV-0009).

10.4.3 For aqueous samples, it should be noted that TestAmerica Denver routinely aliquots samples gravimetrically. This is done to prevent cross-contamination due to volumetric glassware and to provide a more accurate initial volume measurement. However, some clients and regulatory programs require the laboratory to aliquot samples volumetrically. The client requirements and QASs must be read before samples are aliquotted to check for this requirement. If samples are to be aliquotted

volumetrically, use Class A volumetric glassware only and proceed to Section 10.4.5

10.4.4 Weigh the bottle containing the approximately 1 liter sample and record the gross weight to the nearest gram.

10.4.5 Inspect the samples for large amounts of sediment that may interfere with the extraction of the sample by clogging the solvent flow arm.

10.4.5.1 If the sample contains so much sediment that the entire sample volume cannot be extracted, decant the sample into the CLLE (or a graduated cylinder if volumetric aliquotting is required), careful not to transfer the sediment. Write a NCM to document the sediment and that it prevented the entire sample volume from being extracted and the sample container from being rinsed.

10.4.5.2 If the sample does not contain a significant amount of sediment, then the entire sample volume will be used in the extraction. Do not pour the sample into the CLLE (or into the graduated cylinder if volumetric aliquotting is required) until after the surrogates and any necessary spikes have been added to the samples.

10.4.6 Place the sample containers in front of the CLLE labeled for that sample. A second analyst should then check the labels to make sure the correct sample is being extracted. This check is documented in the Organic Extraction Checklist (WI-DV-0009)

10.5 Add Surrogates to All Field Samples and QC Samples

10.5.1 Add 1.0 mL of the appropriate working surrogate standard to the sample container for each sample and MS/MSD. Add the surrogate standard to the MB and the LCS's in the CLLEs. Record the ID of the standard used on the bench sheet. Note that the standard should be allowed to come to room temperature before spiking the samples.

Note: If the sample contains an amount of sediment that has been deemed to interfere with the extraction process then the surrogate standard is added to the sample in the CLLE or in the graduated cylinder. This is considered a deviation and must be documented in a NCM.

10.6 Add Spikes to all LCS's and MS/MSDs

10.6.1 Add the appropriate working spike standard to the MS/MSD sample containers and the CLLEs for the LCS and/or LCSD samples. Record the ID of the standard used on the bench sheet. Note that the standard should be allowed to come to room temperature before spiking the samples.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-009.

10.7 Add NaCl to all samples and all QC samples

10.7.1 Add approximately 3 grams (approximately ½ tsp.) of baked NaCl to each sample container and each MS/MSD container. Cap the container and

shake gently to dissolve the salt. If the sample is in a graduated cylinder or in a CLLE at this point because of significant sediment, then add the salt to the graduated cylinder or CLLE and stir to dissolve.

10.8 If volumetric aliquotting is required, transfer the entire sample into a Class A graduated cylinder and record the volume on the benchsheet. Then pour the sample into the labeled CLLE on top of the methylene chloride. Rinse the sample container and the graduated cylinder with methylene chloride and add the rinse to the CLLE.

10.9 If volumetric aliquotting is not required, pour the sample directly into the CLLE on top of the methylene chloride. Rinse the sample container with methylene chloride and add the rinse to the CLLE.

10.10 Be careful to allow only the methylene chloride in the bottom of the CLLE to spill over into the boiling flask, and not to allow water to spill over. Add additional methylene chloride or reagent water to the CLLE if needed to ensure proper solvent flow.

10.11 After the labels have been checked by a second analyst, reweigh the bottle and calculate the initial sample volume by subtracting the empty bottle's weight from the full bottle's weight, assuming a density of 1g=1mL. If there is any indication that the sample's density is not 1g=1mL, then measure the density of the sample and correct the calculated initial volume accordingly. See Section 11 for the calculation. Document abnormal sample density in an NCM.

10.12 If the initial volume is less than 800 mL the sample reporting limits and method detection limits will be elevated accordingly. Document this in a NCM.

10.13 Adjust pH of Field Samples and QC Samples

Adjust the sample pH as indicated in the chart below using a minimum amount of 1:1 sulfuric acid or 10 N sodium hydroxide, as necessary.

When adjusting neutral samples to a pH of 1-2, start by adding 2mL of the 1:1 sulfuric acid. Use a Teflon® stir rod to mix the sample and check the pH using wide-range pH paper. Record the adjusted pH and the lot number of the acid on the bench sheet. If more than 2mL of the acid is required, continue adding the acid in 2mL increments until the proper pH is achieved and document on the benchsheet and in an observation NCM how many milliliters of acid were required.

When adjusting neutral samples to a pH of 14 for 8270_LL 1,4-Dioxane, start by adding 5mL of the sodium hydroxide. Use a Teflon® stir rod to mix the sample and check the pH using wide range pH paper. Record the adjusted pH and the lot number of the base on the bench sheet. If more than 5mL of sodium hydroxide is required, continue adding the base in 1mL increments until the proper pH is achieved and document in an observation NCM how many milliliters of base were required. It is important to try to achieve a pH of 14 in order to most effectively extract the 1,4-dioxane.

Method	Initial Extraction pH	Secondary Extraction pH
SW-846 8270 and Method 625 Method Codes: 625 8270_AFCEE 8270_DoD 8270C 8270C_DNB 8270_UTS 8270D	1 – 2	11 - 12
SW-846 8270 SIM PAHs Method Codes: 8270_SIM_AFCEE 8270_SIM_DoD 8270C_SIM	As Received	None
SW-846 8270_LL Semi-Volatile Organics Method Code 8270C_LL with a prep of 3520C	1 – 2	11-12
SW-846 8270_LL 1,4-Dioxane Only Method Code 8270C_LL with a prep of 3520C_Base	14	None
SW-846 8070A and Method 607	As Received	None

10.14 Start the Initial Extraction

10.14.1 Attach the cold condenser (chilled at 5°C to10°C). Check that the hoses are not kinked. Check the temperature and water level of the chiller.

10.14.2 Turn on the heating mantle or hotplate. Check again for boiling chips in the boiling flask and inspect joints for leaks once solvent has begun cycling. The methylene chloride in the round bottom flask should be boiling steadily, but not too rapidly that the condenser cannot condense the vapor as fast as it is being generated.

10.14.3 Record the date and the time the extraction started on the bench sheet.

10.14.4 For methods all methods except "8270_DoD" and "625", extract for 18-24 hours. For method 625, extract for 24 hours. In order to consistently meet the DoD QSM control limits for the method 8270 surrogate 2-fluorobiphenyl, a 24 hour extraction is required for method "8270_DoD".

10.14.5 At the end of the extraction, turn off the heating mantle or hotplate and allow the extractor to cool. Record the date and time the extraction stopped on the bench sheet.

10.14.6 Remove the boiling flask, cap tightly with aluminum foil, and store refrigerated until concentration. If a second extraction at basic pH is required, continue on to Section 10.15. If a second extraction is not required, go to Section 10.16.

10.15 Start the Secondary Base Extraction

10.15.1 Attach a boiling flask with fresh methylene chloride and boiling chips to the CLLE. Label each boiling flask with the sample ID or batch QC ID.

10.15.2 Remove the condenser. Using a minimum amount of sodium hydroxide, adjust the pH of the sample in the extractor body to a pH of 11-12 for the methods indicated in Section 10.13. Usually 5-6 mL of base is needed. Use a Teflon ® stir rod to mix the sample. Measure with wide-range pH paper and record the adjusted pH and the lot number of the base used on the bench sheet. If more base is needed add it in 1mL increments until a pH of 11-12 is reached. Document in an observation NCM how many milliliters of base were required. It is important to try to achieve a pH between 11-12. A pH above 12 might be detrimental to the recovery of some target analytes.

10.15.3 Re-attach the cold condenser, turn on the heating mantle or hotplate, and inspect the joints for leaks. Record the date and time the extraction started on the bench sheet.

10.15.4 For methods all methods except "8270_DoD" and "625", extract for 18-24 hours. For method 625, extract for 24 hours. In order to consistently meet the DoD QSM control limits for the method 8270 surrogate 2-fluorobiphenyl, a 24 hour extraction is required for method "8270_DoD".

10.15.5 At the end of the second extraction, turn off the heating mantle or hotplate and allow the extractor to cool. Record the date and time the extraction stopped on the bench sheet.

10.15.6 Remove the boiling flask, cap tightly with aluminum foil, and store refrigerated until concentration.

10.16 Disposal of Waste

10.16.1 Dispose of the methylene chloride remaining in the CLLE body in the methylene chloride Waste Stream B.

10.16.2 Dispose of the solvent-saturated water remaining in the CLLE in Waste Stream X if pH is neutral or basic.

10.17 Initial weights and volumes of samples and all extraction dates/times and reagents are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklist (see WI-DV-0009).

10.18 Wash glassware following SOP DV-OP-0004.

11.0 Data Analysis and Calculations

$$InitialVolume(mL) = \frac{FullBottle(g) - EmptyBottle(g)}{Density(g/mL)}$$

12.0 Method Performance

12.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL). See Policy DV-QA-005P, "Determination of Method Detection Limits", for more information on the method detection limit studies.

12.2 An initial demonstration of capability (IDOC) must be performed by each analyst. On-going proficiency must be demonstrated by each analyst on an annual basis. See DV-QA-0024, "Employee Training", for more information on the IDOCs.

12.3 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

12.4 Calibration
N/A

12.5 Sample Analysis
N/A

13.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

The laboratory currently purchases only low-solvent extractors that allow the procedure to be performed with approximately 50mL of methylene chloride in the bottom of the extractor body.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and HS-001, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methylene chloride – Waste Stream B

14.2.2 Neutral aqueous sample waste saturated with methylene chloride – Waste Stream X.

14.2.3 Basic aqueous sample waste saturated with methylene chloride – Waste Stream X.

14.2.4 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005, Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.

15.2 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 625.

15.3 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 607.

16.0 Modifications:

16.1 Modifications from SW-846 Method 3520C

16.1.1 Section 7.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

16.1.2 Section 7.7 of the method states that if the acid and base extracts are not to be analyzed separately, then the boiling flask and solvent does not need to be changed, but may be used for the second pH extraction. This is currently not the practice here at TestAmerica Denver in order to prevent loss of some of the compounds that would extract in the first 18-hour extraction.

16.1.3 Table 1 of the method calls for the secondary extraction pH for method 8270C to be >11. This SOP calls for the secondary extraction pH for method 8270C to be between 11 and 12. Guidance is given in order to prevent the pH of the secondary extraction from being greater than 12 as this has been demonstrated to produce lower recoveries of some target compounds including the surrogate 2-flurobiphenyl.

16.2 Modifications from 40 CFR Method 625

16.2.1 Section 2.1 of the method calls for extracting the method at a pH greater than 11 and then again at a pH less than 2. This SOP calls for the acid extraction to be performed first followed by the base extraction. This is done to improve the recoveries of the phenols.

16.2.2 Section 11.2 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

16.2.3 The Method calls for the acid and base extracts to be concentrated and analyzed separately. This SOP calls for the extracts to be combined, then concentrated and analyzed.

16.3 Modifications from 40 CFR Method 607

16.3.1 The method calls for the extraction to be performed by separatory funnel. However it is the laboratory's experience that CLLE extraction produces better recoveries.

16.3.2 Section 10.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

17.0 Attachments

Figure 1. Continuous Liquid-Liquid Extractor (CLLE)

Table 1. Determinative Methods Using CLLE Extractions

Table 2. Working Standards for Waters and SPLP Leachates by 8270.

Table 3. Working Benzidine Spike Standards for 625

Table 4. Working Standards for TCLP 8270.

Table 5. Working Standards for 8070A and 607

Attachment 1. Trouble-shooting Guide

18.0 Revision History

Revision 5.0 dated 08/02/10

- This procedure was revised to remove the steps required for the extraction of samples for low-level NDMA analysis by GC/CI/MS/MS. The CLLE extraction procedure utilized for that analysis is described in DV-OP-0020.
- This procedure was revised to incorporate the requirements of TestAmerica Denver's new LIMS "TALS".
- Section 5.1.2 was added to discuss the hazards of ground glass joints and required PPE for tightening and loosening the joints.
- Section 6.1 and Section 10 were revised to state that the chiller temperature should be set at 5°C to 10°C instead of 8°C to 10°C.
- Section 6.1 was revised to stat the capacity of the balance should be greater than or equal to 1600g, not 1400g.

- Section 7.1 was revised to state that he lab has 3 ELGA analytical units instead of 2 and that the water should be at or above 18 Mohm-cm instead of 17 Mohm-cm.
- Section 7 was revised to clarify the expiration dates of the reagents.
- Section 7.9.5 was revised to state that instead of preparing a separate LCS standard for method 8270C_LL, 0.125mL of the method 8270 standard will be used.
- Note added to section 9.5 to not allow marginal exceedances for South Carolina work.
- The 8270/625 standard (8270LCS80ppm) described in Table 2 was revised to be made at a concentration of 80ug/mL instead of 100ug/mL. The surrogate compounds were corrected to remove compounds that are not used in the surrogate recovery evaluations.
- The TCLP spike standard (8270TCLPSpike) described in Table 4 was corrected to accurately reflect the concentrations of 3-Methylphenol and 4-methylphenol.

Revision 4.3 dated 01/04/10

- Basic Annual Review

Revision 4.2 dated 9/30/09

- Added clarification for the criteria of surrogating and spiking samples directly into the original container.

Revision 4.1 dated 9/18/09

- Added criteria for surrogating and spiking samples directly into the original container.
- Added comments in Section 4 about phthalate contamination arising from gloves.
- Removed the Organic Extractions Checklist. The checklist is now included in work instruction WI-DV-0009.
- The procedure was revised to include the addition of approximately 3 grams of baked sodium chloride to every sample and QC sample except those being extracted for low-level NDMA. This was done in order to increase the ionic strength of QC samples and field QC samples to more closely match the ionic strength of typical samples and to aide in the extraction of the more polar compounds.
- Changed the concentration of the benzidine LCS spike to 200µg/mL and the volume used to 1mL per liter of sample.
- Eliminated the "short-list" 8270 LCS spike mix. All 8270 LCSs are spiked using the full list 8270/625 LCS mix, which was also revised to correct the analyte list.
- Eliminated the acid only extraction for pentachlorophenol by 8270C SIM.

Revision 4 dated 12/20/08

- Added Section 4.6 to discuss the importance of targeting the pH of the base extraction.
- Added the option for hotplates in Section 6.
- Section 7.1.2 was revised to change the amount of sodium chloride used in the preparation of reagent water from 1g per liter to 3g per liter. This was done to compensate for seasonal changes in the water quality.
- Section 7.3.1 was revised to reflect actual laboratory practice. It was changed to state that the water used in the preparation of the 1:1 sulfuric acid is "water from the ELGA purification system" instead of "reagent" water. The laboratory's practice has been to use water from the purification system in the making of the reagent, not water from the purification system that has had salt added.

- Section 10.2.2 and 10.3.2 were revised to give better guidance on the glassware rinsing. This was done to match the instructions given in DV-OP-0004 "Glassware Washing for Organic Analysis Applications".
- Sections 10.4.3.4 and 10.4.4.6 were revised to include a label check after the sample transfer into the CLLE body.
- Section 10.8 was revised to give more guidance on the pH adjustment by instructing the analyst to add 2mL of acid when adjusting neutral samples to a pH of 1-2 with additional acid added in 1mL increments. The section was also revised to instruct the analyst to add 10mL of base when adjusting neutral samples to a pH of 14 wth additional base added in 1mL increments. The table was revised to change all base extractions to a pH of 11-12, except 8270 Best Practice, Low-Level 1,4-Dioxane which requires a pH of 14.
- Section 10.10.2 was revised to change the amount of base added to adjust an acidic sample to a pH of 11-12. The previous version of this SOP stated that normally 10mL of sodium hydroxide is to be used. This was revised to state 5-6 mL of sodium hydroxide should be used.
- Section 13 was revised to include the laboratory's current practice of purchasing only low-solvent extractor bodies in order to reduce the amount of methylene chloride needed.
- Section 16.1.4 was revised to document the differences between the basic pH range used in the SW-846 method 3520C and the range used in this procedure.

Revision 3, dated 20 March 2008

- Integration for TestAmerica and STL operations.

FIGURE 1.

Continuous Liquid-Liquid Extractor (CLLE)

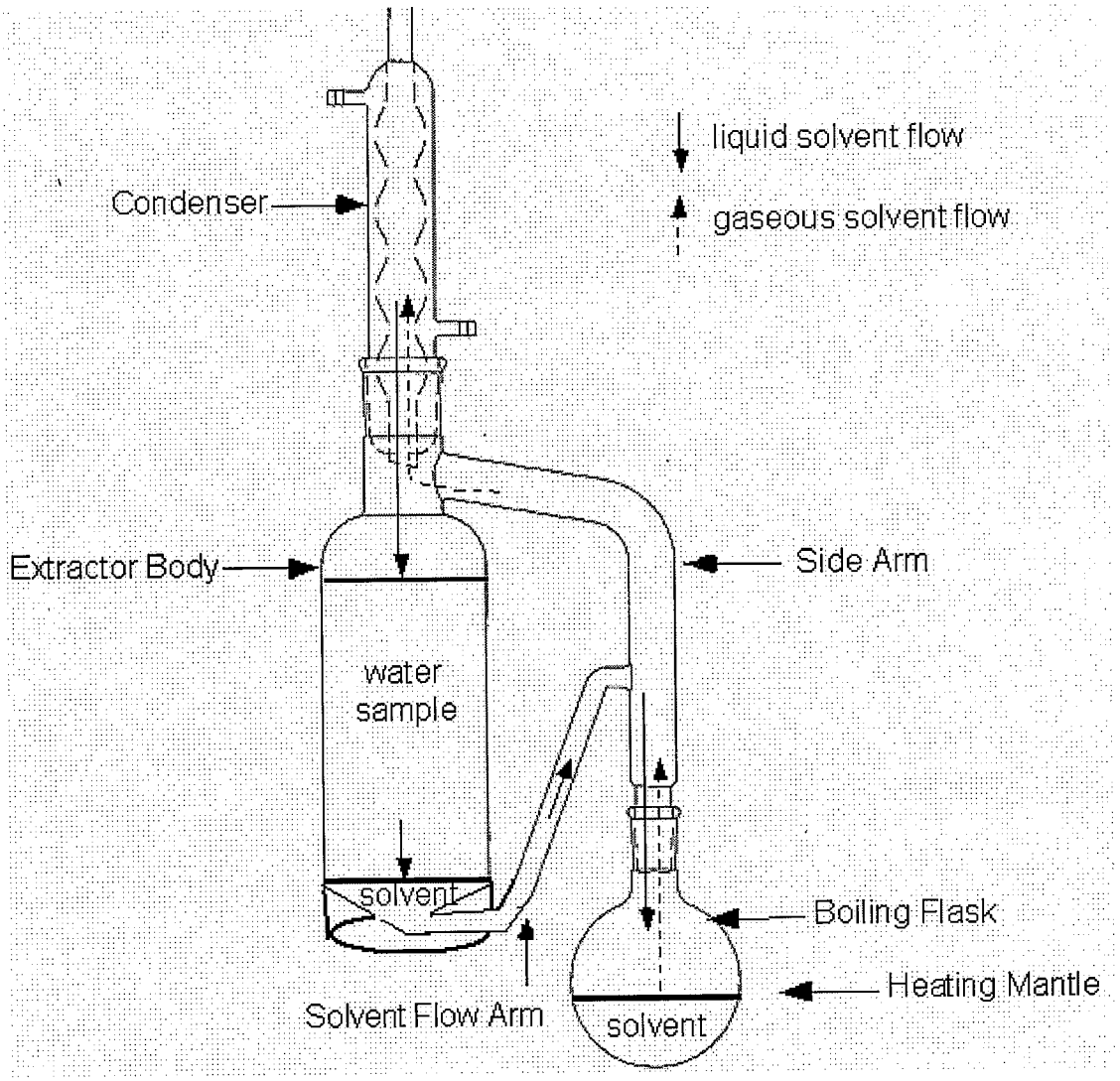


TABLE 1.

Determinative Methods Using CLLE Extractions

Method Description	*Determinative Method*	*SOP*
Semi-Volatiles by GC/MS	SW-846 8270C EPA Method 625	DV-MS-0011
Semi-Volatiles by GC/MS	SW-846 8270D	DV-MS-0012
Low-Level Semi-Volatiles by GC/MS Best Practice	SW-846 8270	DV-MS-0011
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270 SIM	DV-MS-0002
n-Nitrosodimethylamine by GC	SW-846 8070A EPA Method 607	DV-GC-0018

TABLE 2.

Working Standards for Waters & SPLP Leachates by Method 8270/625

Spike Solution (8270LCS80ppm)	
1,2-Dinitrobenzene	80
1,2,4-Trichlorobenzene	80
1,2-Dichlorobenzene	80
1,3-Dichlorobenzene	80
1,3-Dinitrobenzene	80
1,4-Dichlorobenzene	80
1,4-Dinitrobenzene	80
1,4-Dioxane	80
1-Methylnaphthalene	80
2,3,4,6-Tetrachlorophenol	80
2,3,5,6-Tetrachlofophenol	80
2,4,5-Trichlorophenol	80
2,4,6-Trichlorophenol	80
2,4-Dichlorophenol	80
2,4-Dimethylphenol	80
2,4-Dinitrophenol	80
2,4-Dinitrotoluene	80
2,6-Dinitrotoluene	80
2-Chloronaphthalene	80
2-Chlorophenol	80
2-Methyl-4,6-Dinitrophenol	80
2-Methylnaphthalene	80
2-Methylphenol	80
2-Nitroaniline	80
2-Nitrophenol	80
3,3'-Dichlorobenzidine	80
3-Methylphenol	Prepared at 80ug/mL, but co-elutes with 4-Methylphenol for a total concentration of 160ug/mL
3-Nitroaniline	80
4-Bromophenylphenyl ether	80
4-Chloro-3-Methylphenol	80
4-Chloroaniline	80
4-Chlorophenyl phenyl ether	80
4-Methylphenol	Prepared at 80ug/mL, but co-elutes with 3-Methylphenol for a total concentration of 160ug/mL
4-Nitroaniline	80
4-Nitrophenol	80

TABLE 2 Continued.

Working Standards for Waters & SPLP Leachates by Method 8270/625

Acenaphthene	80
Acenaphthylene	80
Aniline	80
Anthracene	80
Azobenzene	80
Benzo(k)fluoranthene	80
Benzo(a)anthracene	80
Benzo(a)pyrene	80
Benzo(b)fluoranthene	80
Benzo(g,h,i)perylene	80
Benzoic Acid	80
Benzyl Alcohol	80
Benzyl butyl phthalate	80
Bis(2-Chloroethyoxy)methane	80
Bis(2-Chloroethyl) ether	80
Bis(2-ethylhexyl) phthalate	80
Bis(2-ethylhexyl) adipate	80
Carbazole	80
Chrysene	80
Di-n-butyl phthalate	80
Di-n-octyl phthalate	80
Dibenz(a,h)anthracene	80
Dibenzofuran	80
Diethyl phthalate	80
Dimethyl phthalate	80
Fluoranthene	80
Fluorene	80
Hexachlorobenzene	80
Hexachlorobutadiene	80
Hexachlorocylclopentadiene	80
Hexachloroethane	80
Indeno(1,2,3-cd)pyrene	80
Isophorone	80
N-Nitrosodi-n-propylamine	80
N-Nitrosodimethylamine	80
N-Nitrosodiphenylamine	80

TABLE 2 Continued.

Working Standards for Waters & SPLP Leachates by Method 8270/625

Naphthalene	80
Nitrobenzene	80
Pentachlorophenol	80
Phenanthrene	80
Phenol	80
Pyrene	80
Pyridine	80
Surrogate Solution: (8270Surrogate)	
2-Fluorobiphenyl	100
2-Fluorophenol	150
Nitrobenzene-d5	100
Terphenyl-d14	100
Phenol-d5	150
2,4,6-Tribromophenol	150

TABLE 3.

Working Benzidine Spike Standard for Method 625

(Benzidine LCS)

Spike Solution:	
Compound	**Concentration (µg/mL)**
Benzidine	200

TABLE 4.

Working Standards for TCLP 8270

(8270TCLPSpike)

Spike Solution:	
Compound	**Concentration (µg/mL)**
1,4-dichlorobenzne	50
2,4,5-trichlorophenol	50
2,4,6-trichlorophenol	50
2,4-dinitrotoluene	20
2-methylphenol	50
3-methylphenol	100 (prepared at 50ug/mL, but co-elutes with 4-methylphenol)
4-methylphenol	100 (prepared at 50ug/mL, but co-elutes with 4-methylphenol)
Hexachlorobenzene	20
Hexachlorobutadiene	50
Hexachloroethane	50
Nitrobenzene	50
Pentachlorophenol	100
Pyridine	50

TABLE 5.

Working Standards for NDMA by Method 8070A and 607

Spike Solution: (8070LCS)	
Compound	**Concentration (µg/mL)**
n-Nitrosodimethylamine	10
Surrogate Solution: (8070 Surr)	
n-Nitrosodiethylamine	10
n-Nitrosodiphethylamine	10

ATTACHMENT 1.

Trouble-shooting Guide

Burn-ups - *Whenever one of these things happen, document it in an NCM, and on the benchsheet!*

- **Is the stopcock closed?** If yes, then the sample never extracted. The solvent just went up the arm, condensed, and collected in the body of the CLLE. At this point the boiling flask can be very hot because it was boiled dry, so turn off the heating mantle. It is best if you replace the boiling flask with a new empty flask with new boiling chips because the old boiling chips could be ruined. Once the new empty boiling flask is on the arm, open the stopcock and the solvent will spill over the arm and into the flask. Turn on the heating mantle and start the 18 or 24 extraction clock over.
- **Is the condenser warm?** If yes, then the extraction is most likely ruined, especially if the boiling flask was dry. If there is sample volume and hold time remaining, then the sample should be re-extracted. If there is no sample volume remaining, then get a cold condenser on the CLLE, add more solvent and continue with the extraction. If there is sample remaining, but hold time has expired we need to save this extraction because it is the only one in hold. So get a cold condenser on the CLLE, but start the re-extraction ASAP.
- **Is there a loose joint?** If yes, then the extraction is most likely ruined. Follow corrective actions for warn condenser above.
- **Was the CLLE not topped off?** If the answers to numbers 1, 2, and 3 above is no, then look at the solvent return arm. If the return arm is not full, then maybe the CLLE was never topped off and the solvent just went up the arm, condensed, and collected in the body of the CLLE. Follow corrective actions for closed stopcock above.

Flooded Arms

- If the arm is equipped with a Snyder column, the arm might have become flooded because the solvent was boiling too fast and flooded the chambers of the Snyder column. When this happens, the solvent level in the round bottom flask might actually be low because the solvent is boiling out of the flask faster than it can flow back in. If this is the case, turn down the temperature of the heating mantle.
- If the flask and arm contain only solvent, just let the extraction finish. At the end of the extraction time, close the stopcock while the heating mantle is still on. Monitor the CLLE closely as the solvent boils and collects in the body of the CLLE. When the solvent level is low enough to remove the flask, turn off the heating mantle and let it cool. Remove the flask and cap with foil. If a second extraction is needed, place an empty boiling flask on the arm and open the stopcock. The excess solvent from the first extraction will flow into the empty boiling flask. Add more solvent if needed.
- If the flask and arm contain solvent and water, turn off the heating mantle and let the boiling flask cool. If there remains enough solvent in the boiling flask, close the stopcock and boil off enough solvent so you can remove the boiling flask. Once solvent level is low enough, then turn off heating mantle and allow it to cool. Then remove boiling flask and pour the water and solvent from the flask into the CLLE body while the stopcock is closed. Re-attach the boiling flask, open the stopcock, and allow the solvent to flow into the flask. If this doesn't work, the sample and all the solvent needs to be transferred to a larger CLLE.

- If the flask and arm contains solvent and water, and the step above didn't work or there is not enough solvent left in the boiling flask to attempt the step above, turn off the heating mantle and let the boiling flask cool. Rinse a 2L separatory funnel with methylene chloride. When the boiling flask is cool, remove the condenser from the CLLE, grasp the CLLE at the body and at the joint with the boiling flask and pour the sample and the solvent into the rinsed separatory funnel. This needs to be done in a hood and you will need to have someone help you. Add a few more boiling chips to the boiling flask and place the empty CLLE back on the stand under the slot hood. Drain the methylene chloride from the separatory funnel into the body of the CLLE. Once all the methylene chloride has been transferred, continue to drain the water into the CLLE body on top of the methylene chloride. The water level in the CLLE will reach a high enough level that it will force the solvent over into the boiling flask. If the solvent level in the body of the CLLE gets too low, stop and add more solvent to the CLLE body to prevent water from going into the boiling flask. Rinse the separatory funnel with methylene chloride and add it to the CLLE.

Stuck Joints

- **Stuck Condenser** If the condenser is stuck, first close the stopcock and remove the boiling flask. Put on cut-resistant gloves. Try to loosen the joint with the cut-resistant gloves. Try both clock-wise and counter-clock-wise. If this doesn't work, heat the outside of the joint with the torch, but be sure not to keep the flame on one part of the glass, instead keep moving the flame. Watch for vapor bubbles between the glass joints as an indication the joint is loosening. Try to loosen the joint again. Repeat a few times. If heating the outside of the joint is not working, let the glass cool for about 30 minutes then come back and try again. If it is still stuck, unplug the condenser and empty the CLLE. Then soak the stuck joint in soapy water overnight.
- **Stuck Boiling Flask** If the boiling flask is stuck, first close the stopcock and remove the condenser. Put on cut-resistant gloves. Try to loosen the joint with the cut-resistant gloves. Try to loosen the joint both clock-wise and counter-clock-wise. If this doesn't work, tap the joint downward with a heavy object and try again. A plastic media-bottle cap can be used.
- **Stuck CLLE Body** If the CLLE body is stuck to the stand, close the stopcock and remove the condenser and the boiling flask. If possible, remove the plastic support from the rack with by removing the screws that hold it in place. Then lift the CLLE and the plastic support off the rack and empty the CLLE. Run hot water over the CLLE and plastic support to separate the glass from the plastic.

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Title: Microwave Extraction of Solid Samples by Method [SW-846 3546]

Approvals (Signature/Date):

Susan H. Oster 1/24/13	Adam W Alban 30Jan13
Susan Oster Date	Adam Alban Date
Technical Specialist	Health & Safety Manager / Coordinator
1/30/13	1/30/13
John Morris Date	William S. Cicero Date
Quality Assurance Manager	Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This SOP is applicable to the solvent extraction of organic compounds from solid samples using microwave energy to produce elevated temperature and pressure conditions in a closed vessel containing the sample and organic solvent. This procedure achieves analyte recoveries equivalent to those from soxhlet or sonications methods, but uses less solvent. This SOP is based on SW-846 Method 3546.

1.2 The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate pH and spiking mixtures are used.

1.3 This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, Concentration of Organic Extracts, for those details.

2.0 Summary of Method

A measured weight of sample, typically 30 g, is solvent extracted using a microwave extractor.

3.0 Definitions

3.1 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. Please reference WI-DV-0032 for details on Method Comments.

3.4 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.5 **Aliquot**: A part that is a definite fraction of a whole; as in “take an aliquot of a sample for testing or analysis.” In the context of this SOP, “aliquot” is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Sodium sulfate is not used in the extraction vessel. This is because salts are known to super heat when exposed to microwave energy. Samples are extracted without the addition of sodium sulfate, but the extracts are dried with sodium sulfate after the extraction, before concentration of the extracts. If the sample is excessively wet the aliquot can be divided among two or three extraction vessels and the extracts combined prior to concentration.

4.3 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of this SOP (Section 9). Specific selection of reagents may be required to avoid introduction of contaminants.

4.4 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.5 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.6 Paint chips are an especially difficult matrix to extract. Oftentimes the paint chips dissolve or partially dissolve in solvents and therefore can ruin glassware and extraction vessels. It is the laboratory's experience that paint chips are best extracted by method SW-846 3580 instead of 3550C or 3546.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 A post-run cool down must be used after each extraction to prevent the possibility of operator burns. Pressure builds up in the closed vessel at high temperatures. Care should be taken when opening the vessel when it is above room temperature.

5.1.2 Samples that contain metal fragments or metal components of any kind should not be extracted by this procedure. These samples should be

extracted by method SW-846 3550C instead. Care should be taken to inspect samples carefully as they are aliquotted.

Eye protection that satisfies ANSI Z87.1 (as described in the Corporate Safety Manual), laboratory coat, and appropriate gloves must be worn while performing this procedure. Nitrile gloves shall be worn when handling solvents; latex gloves may be worn when handling samples only; and cut resistant gloves shall be worn when washing glassware.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous. It is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Hexane	Flammable	50 ppm (TWA)	Prolonged or repeated contact with skin can cause defatting and dermatitis. Contact with eyes can cause redness, tearing, and blurred vision. Exposure can cause lung irritation, chest pain, and edema, which may be fatal.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

- Microwave extractor. CEM MARS®

  At least once a year, power measurement calibration should be performed at 400W, 800W, and 1600W. This calibration can be performed by the vender or by TestAmerica staff following the instructions in the Operations Manual for the microwave.
- Microwave extraction vessels. 75mL Teflon™ Express vessels with stopper and cap (CEM Corp.)
- Hand wrench to tighten the caps on the extraction vessels.
- MARS 40 position carrousel (CEM Corp)
- Balance, >1400-g capacity, accurate to ± 0.1 g, calibrated daily per SOP DV-QA-0014.
- Media bottles, 100 mL or 250mL with Teflon™-lined caps or capped with aluminum foil.
- Stainless steel conical funnels
- Ashless cellulose filter paper – Whatman Grade 41 or Ahlstrom Grade 54
- Pipetter with disposable 1.0-mL tips, calibrated daily per SOP DV-QA-0008.
- Metal spatulas or tongue depressors.
- Solvent dispenser pump.
- Filter flask.
- Vacuum pump.
- Washing tool for Teflon™ extractor vessels. This tool is a long thin sponge-like brush.

6.1 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on

Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Methylene chloride – Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.2 Acetone - Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.3 Hexane - Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.4 Baked Sodium Sulfate, 12-60 mesh - Heat sodium sulfate in a 400 °C oven for at least four hours. Each lot is tested following CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.5 Baked Ottawa Sand – Heat Ottawa sand in a 400 °C oven for at least four hours.

7.6 35% Nitric Acid – Dilute 70% Nitric Acid 1:1 in water.

Standards

7.7 Please reference SOP DV-OP-00020 and WI-DV-009 for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils for Method 8082A	Glass with Teflon-lined lids	30 grams	Cool, $\leq 6^{o}C$	None	SW-846
Wipes for Method 8082A	Glass with Teflon-lined lids	N/A	Cool, $\leq 6^{o}C$	None	SW-846
Soils for all other Methods, including 8082	Glass with Teflon-lined lids	30 grams	Cool, $\leq 6^{o}C$	14 days	SW-846
Wipes for all other Methods, including 8082	Glass with Teflon-lined lids	N/A	Cool, $\leq 6^{o}C$	14 days	SW-846

[1] Exclusive of analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, reference the Method Comments and QAS to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Assurance Program.*

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, *Requirements for Federal Programs*.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS and in the Quality Assurance Summaries (QAS) available in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank is processed and analyzed just as if it were a field sample.

The method blank consists of 30g of baked Ottawa sand free of any of the analyte(s) of interest.

Acceptance Criteria: The result for the method blank must be less than the reporting limit for the analyte(s) of interest or less than 10% of the analyte concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: If target analytes in the blank exceed the acceptance limits, an unacceptable method blank must be re-prepared and reanalyzed. If the analyte was not detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.5 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

At least one LCS must be processed with each preparation batch. The LCS is carried through the entire analytical procedure just as if it were a sample.

The LCS consists of 30g of baked Ottawa sand to which the analyte(s) of interest are added at known concentration.

Method AK102 requires LCS and a LCSD for every batch for every spike compound.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

Method NWTPH-Dx requires a matrix spike and a matrix spike duplicate for every 10 samples. If insufficient sample volume is available for MS/MSD, a NCM must be written and a LCS and LCSD must be performed for every 10 samples.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at $\pm$ 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Critical Procedural Considerations

10.2.1 As stated throughout this SOP, analysts must review the LIMS Method Comments, and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-009).

10.2.2 Analysts must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other beaker or media bottle than the designated one should be cleaned or disposed of before coming into contact with the sample.

10.3 Periodic cleaning.

10.3.1 Once a week the extraction vessels must be cleaned using a “Clean Method” on the microwave. The method is under the User Directory with the settings that follow:

Sample Type: Inorganic
Control Type: Ramp to Temperature
Power: 100%
Ramp: 5 minutes to 180 °C
Hold: 10 minutes

10.3.2 Fill each tube with 30mL of the nitric acid solution described in Section 7 and cap tightly. Place the tubes in the carousel, then run the “Clean Method”

10.3.3 Allow the vessels to cool, and then dispose of the nitric acid in waste stream J. Rinse the vessel with DI water three times.

10.3.4 Fill each tube with 30mL of 1:1 Methylene Chloride : Acetone solution and cap tightly. Place the tubes in the carousel, then run the “Clean Method” again.

10.3.5 Allow the vessels to cool, and then dispose of the solvent in waste stream C. Allow the vessels to air dry.

10.4 Assemble and Clean the Extraction Tubes Immediately Before Use.

10.4.1 If the microwave tube, cap, or plugs are wet, pre-rinse with acetone.

10.4.2 Rinse the microwave tube, cap and plug with methylene chloride. The plugs can be placed in a large Büchner funnel to help facilitate the rinse.

10.4.3 Discard the solvent in the correct waste stream.

10.5 Aliquot Samples

10.5.1 If the sample is a soil, mix and homogenize samples according to the instructions provided in SOP DV-QA-0023, Subsampling. If the sample is a wipe, transfer the wipe to the extraction vessel.

10.5.2 Label microwave vessel with the sample ID, method, batch number, and date. The label needs to be flat and placed close to the bottom of the vessel.

10.5.3 For each MB and LCS sample, weigh 30 to 33 g of baked Ottawa sand into labeled microwave vessels. Record the weight to the nearest 0.1 g directly into LIMS or hand record the weight on the benchsheet.

10.5.4 For each sample and MS/MSD, weigh 30 to 33 g of sample into the labeled microwave vessel. Record the weight to the nearest 0.1 g directly into LIMS or hand record the weight on the benchsheet.

NOTE: If the sample matrix appears to be unusual, or especially wet, the 30 g aliquot can be equally divided between two or three separate microwave extraction vessels. The vessels will be extracted independently, but the extracts will be re-combined before concentration. This will prevent the extraction vessels from over-heating and venting if the sample is unusually wet, oily, or bulky (if a 30 g aliquot would fill the tube more than ¾ full). If the sample is split into two or three separate vessels, prepare an NCM.

NOTE: Care should be taken to ensure that the top lip of the tube is clean of any sample material or debris so that the plug will fit tightly later.

10.5.5 Place the microwave vessel on a cart next to the sample container so that a second analyst can check the labels. This is documented on the Organic Extraction Checklists (See WI-DV-009).

10.6 Prepare a bottle with a bottle-top dispenser with the appropriate solvent.

- Methylene Chloride is used for soil and wipe samples for the following methods:
  - SW-846 8015B (8015B_DRO)
  - SW-846 8015C (8015C_DRO)
  - Alaska Methods AK102 and AK103 (AK102_103)
  - NWTPH DRO (NWTPH_Dx)

- **o** Oklahoma DRO Method (Okla_DRO)

- For soil extraction by all other methods, the solvent used is a 1:1 mixture of methylene chloride and acetone.

- For wipe samples by method 8081 and 8082, the solvent used is hexane.

- For wipe samples by method 8270 SIM, the solvent used is a 1:1 mixture of methylene chloride and acetone.

10.7 Add Surrogate and Spike Solutions

NOTE: The standards should be allowed to come to room temperature before spiking the samples.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-009.

10.7.1 Only one batch should be surrogated at a time to ensure the correct standards are used and to ensure the solvent is added as soon as possible to the samples.

10.7.2 Using a calibrated pipette, add the appropriate volume of the appropriate working surrogate standard (see WI-DV-009) to the microwave vessel for each field sample and QC sample. Record the ID of the standard used on the benchsheet.

NOTE: If the sample aliquot was split into two or three separate tubes in Section 10.5.4 above, split the surrogate volume into the separate tubes as well.

10.7.3 Using a calibrated pipette, add the appropriate volume of the appropriate working spike standard (see DV-OP-009) to the microwave vessel containing any LCS, LCSD, MS, and MSD samples. Record the ID of the standard used on the benchsheet.

NOTE: If the MS or MSD aliquot was split into two or three separate tubes in Section 10.5.4 above, split the spike volume into the separate tubes as well.

10.8 Making sure not to overflow the vessel, slowly add approximately 25-30 mL of the appropriate solvent to the vessel. See Section 10.6 above for the appropriate solvent. Note that the solvent should be added as soon as possible after the addition of the surrogate and spiking standards to prevent loss of the more volatile compounds.

NOTE: For wipe samples add the solvent to the container that the wipe was received in and then transfer it to the microwave vessel. This is done to ensure a quantitative transfer of any solvent and material in the wipe sample container.

NOTE: The solvent should completely cover and saturate the sample so additional solvent may be needed depending on the matrix of the individual sample. The sample and solvent must not fill more than 2/3 of the vessel.

10.9 Seal the vessels by placing the plug on top of the vessel, small side down, and hand tighten the cap over the plug.

NOTE: Care should be taken to ensure that the plug, the cap, and the threads of the vessel are clean of any material or debris.

10.10 After sealed, the vessels must be inverted several times to ensure that the material is well mixed and saturated. It is recommended that when extracting with 100% methylene chloride to vent and re-cap the vessels before continuing to relieve excess pressure and thereby preventing the vessels to vent during the extraction.

10.11 Load vessels into the carrousel.

10.11.1 There must be at least 8 vessels in the carrousel. Adding blank vessels with sand and solvent may be necessary.

10.11.2 Balance the tubes around the carrousel to ensure that all samples are exposed to an equal amount of energy during the extraction. For 8-16 samples, use only the inside ring. For 17-24 samples use only the outer ring. For more than 24 samples the inner ring should be filled completely first and additional samples balanced around the outer ring.

10.11.3 Only samples using the same extraction solvent should be placed in the same carrousel and ran at the same time.

10.11.4 For the vessels to be correctly loaded in the carrousel the cap should completely touch the top of the carrousel with nothing else of the extraction vessel visible.

10.12 Place the carrousel into the microwave, making sure that it sits on the turning apparatus correctly. The carrousel should be able to rotate. Close the door.

10.13 The Method Menu screen should indicate “Start Current Method” as being 3546 Full Xpress. Press the green “Start/Pause” button to begin the extraction.

NOTE: If a different method is shown, go to the “Load Method” on the menu screen. Choose “User directory” and place the cursor on the desired method. Press the “Home” button to return to the main menu, where the test highlighted will appear under the “Start Current Method”.

The method is under the User Directory with the settings that follow:

Sample Type: Organic
Control Type: Ramp to Temperature
Power: 100% (1600W)
Ramp: 20 minutes to 115 °C
Hold: 10 minutes

10.13.1 When the extraction is complete, the vessels will need to return to room temperature prior to opening the vessels. The microwave will indicate the approximate temperature of the vessels.

CAUTION: If the carousel is removed from the microwave before the vessels are at room temperature, do NOT open the vessels. The vessels may be placed in a rack outside of the microwave to cool down.

10.13.2 The microwave contains a solvent sensor that will indicate the presence of solvent in the microwave and will stop the extraction. To minimize this, care needs to be taken not to overfill the vessel and to properly cap and tighten the vessel prior to extraction. If the solvent sensor indicates the presence of solvent, open the door and inspect the tops of the tubes for evidence of a solvent leak. If solvent has vented or leaked out of an extraction vessel, the sample must be re-aliquotted and the extraction started over. It is best to re-aliquot the sample into two or three separate extraction vessels to prevent over-heating again. Document this in an NCM.

10.14 Assemble and Clean Filter Funnels and Media Jars.

10.14.1 Without gloves on, fold a 15cm or 18cm diameter cellulose filter paper in quarters. Open the folds to create a cone. Place the filter paper in the bottom of a conical stainless steel funnel. Place the funnel on a 100mL or 250-mL media bottle.

10.14.2 Place approximately 1 tablespoon of baked sodium sulfate in the funnel. Rinse all surfaces of the funnel, the filter and the sodium sulfate with the extraction solvent (see Section 10.6), so all surfaces of the funnel, filter, and sodium sulfate are rinsed.

NOTE: When preparing glassware for the extraction of wipe samples, sodium sulfate is not necessary and the solvent used in the rinse should be the solvent used in the extraction of the wipe samples. (Normally hexane for methods 8081 and 8082).

10.14.3 Allow the solvent to drain completely into the media bottle. Swirl the media bottle to ensure all surfaces come into contact with the solvent. Add additional solvent to the rinse if necessary.

10.14.4 Pour the solvent out of the media bottle over the stem of the stainless steel funnel to rinse the funnel stem.

10.14.5 Discard the solvent in the correct waste stream.

10.15 Filter the Extracts

10.15.1 After the extraction method is complete and the vessels reach room temperature, quantitatively transfer the entire sample through solvent rinsed sodium sulfate funnels and into the media jar. The quantitative transfer is performed by rinsing the microwave extraction vessel at least

three times with solvent.

NOTE: The quantitative rinse is vital in order to achieve good recoveries. The rinses should be significant enough that when done, the extract volume is between 75mL and 100mL.

NOTE: If the sample aliquot was split between two or three tubes, the extracts from all the tubes shall be combined at this time. Filter all of the extracts through the same sodium sulfate funnel and collect in the same media jar.

10.15.2 Once the solvent has completely drained into the collection apparatus, rinse the funnel contents with 10 to 20 mL of additional solvent. Dispose of the solid sample and sodium sulfate into Waste Stream D and cap the media jar with the extract with a Teflon-lined lid or aluminum foil.

10.16 If the extract contains visible solids, it will be necessary to filter the extract again prior to concentration.

10.17 Store the extract refrigerated 4 ± 2 °C until concentration.

10.18 Handwritten notes on the benchsheet are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklists (see WI-DV-009).

10.19 All glassware and microwave tubes, plugs, and caps are washed according to DV-OP-0004.

11.0 Calibration

Not applicable to this procedure.

12.0 Calculations / Data Reduction

Not Applicable.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

Before analyzing samples, the laboratory must establish a method detection limit (MDL). See DV-QA-005P, Determination of Method Detection Limits, for more information on the method detection limit studies.

13.2 Demonstration of Capabilities

An initial demonstration of capability (IDOC) must be performed by each analyst. On-going proficiency must be demonstrated by each analyst on an annual basis. See M-Q-001, TestAmerica Quality Management Plan, and the TestAmerica Denver Laboratory Quality Assurance Manual (QAM) for more information on the IDOCs.

13.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is

performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

14.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method, the policies in section 13 of the Environmental Health and Safety Manual for "Waste Management and Pollution Prevention", and the Waste Management procedure, DV-HS-001P.

15.2 Waste Streams Produced By This Method

15.2.1 Methylene chloride – Waste Stream B

15.2.2 1:1 MeCl2:Acetone – Waste Stream CA

15.2.3 Flammable solvent – Waste Stream C

15.2.4 Solid waste/sodium sulfate – Waste Stream D

15.2.5 Nitric Acid Waste – Waste Stream J

15.2.6 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

16.0 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3456 Microwave Extraction, Revision 0, February 2007.

17.0 Method Modifications:

17.1 SW-846 Method 3546 calls for samples to be either air-dried and ground or mixed with sodium sulfate prior to extraction. This procedure does not call of the air-drying of samples unless requested by the client as this may lead to loss of the more volatile compounds.

17.2 SW-846 Method 3546 calls for samples to be aliquoted on a balance capable to weighing to 0.01 g. This SOP calls for a balance capable to weighing to 0.1 g as this is sufficient to report data to 3 significant figures.

17.3 SW-846 Method 3546 Section 1.4 states "2-20 g of material is usually necessary and can be accommodated by this extraction procedure." This SOP calls for 30-33 g of material.

17.4 SW-846 Method 3546 Section 11.7 states "Add approximately 25 mL of the appropriate solvent system to the vessel." This SOP calls for the addition of 30mL of solvent.

17.5 Method NWTPH-Dx calls for samples to be extracted by method SW-846 3550C. Valid MDLs and IDOCs have been completed using both method SW-846 3550C and SW-846 3546 and they are comparable therefore method NWTPH-Dx is a possible determinative method by this procedure.

17.6 Method AK102 and AK103 calls for samples to be extracted by soxhlet. Valid MDLs and IDOCs have been completed using this procedure, therefore method AK102 and AK103 are listed as a possible determinative methods by this procedure.

17.7 Oklahoma Department of Environmental Quality DRO Method calls for samples to be extracted by sonication or soxhlet. Valid MDLs and IDOCS have been completed using both method SW-846 3550C and SW-846 3546 and they are comparable therefore method Oklahoma Dept. of Environmental Quality DRO Method is a possible determinative method by this procedure.

17.8 Washington State Dept. of Ecology Method for the Determination of Extractable Petroleum Hydrocarbons Fractions calls for samples to be extracted SW-846 3550C, 3540C, or 3545. Valid MDLs and IDOCs have been completed using both method SW-846 3550C and SW-846 3546 and they are comparable, therefore Method WA EPH is a possible determinative method by this procedure.

18.0 Attachments

Table 1: Determinative Methods Using Ultrasonic Extraction

19.0 Revision History

- Revision 3, January 31, 2013
  - Annual Technical Review
  - Sections 4.2 and 10.5.4 were revised to remove the optional addition of sodium sulfate to the samples before extraction. It was determined that the better option when dealing with wet samples is to split the sample into two or three tubes and re-combine the extracts before concentration.
  - Section 4 was revised to add instructions on how to deal with paint chip samples.
  - Section 5 was revised to add comments about the dangers of metal fragments in samples.
  - Section 6 was revised to include the requirement that the Power Measurement Calibration procedure be performed on the unit every year.
  - Section 8 was revised to update the hold times for Method SW-846 8082A.
  - Section 10.8 was revised to give more detail on how full the extraction vessel should be once solvent has been added.
  - Section 10.13.1 was revised to allow the carousel to be removed from the microwave unit before the vessels are cool so long as the vessels are not opened.
  - Section 10.15.1 was revised to add a note about the importance of quantitative transfers and rinses while filtering the extracts.
  - Section 10.15.1 was revised to add instructions to combine all extracts from samples that were originally split across two or three tubes.
  - Section 15 was revised to include the waste stream CA.

- Added the Note to Table 1

- Revision 2.0, January 31, 2012
  - Annual Technical Review
  - Updated Section 4.2 and Section 10.5.4 to describe when sodium sulfate should be used in the extraction vessel.
  - Updated Section 6.0 to allow the use of aluminum foil to cap 100mL and 250mL media jars.
  - Updated Section 6.1 to include details on computer software and hardware.
  - Updated Section 7.0 to include details on the purity of reagents and standards.
  - Updated Section 9.1.4 and Section 10.1 to more accurately reflect the NCM process.
  - Corrected grammatical and formatting errors
  - Updated Section 10.3 to include a solvent cleaning after the weekly acid cleaning.
  - Updated Section 10.5.4, Section 10.7.2, and Section 10.7.3 to include an option to split the sample aliquot into two separate microwave vessels.
  - Updated Section 10.10 and 10.13.2 to give details on how to prevent vessels from over-heating and venting and steps to be taken if venting does occur.
  - Updated Section 10.16 to accurately reflect how the laboratory handles extracts with suspended sediment.
  - Updated Section 10.19 to reference SOP DV-OP-0004 on how to clean the microwave vessels.

- Revision 1 dated 01 Jan 2011
  - Added 8270C SIM as a valid determinative method by microwave extraction.
  - Changed the procedure to call for the extract to be filtered thru a conical steel funnel lined with cellulose filter paper instead of a glass funnel with glass wool. This was done to help remove sediment from the extracts.
  - Removed details about the surrogate and spike standards used in the extraction. This information can now be found in DV-OP-0020.
  - Added instructions to Section 7 on how to prepare the nitric acid solution used in the weekly cleaning of the tubes.
  - Changed the solvent used in the extraction of samples for method 8081 and 8082. The samples are now extracted in a 1:1 Mixture of MeCl2:Acetone instead of a 1:1 Mixture of MeCl2:Hexane.
  - Revised the procedure in Section 10.5 for aliquotting samples to state that 30 to 33g of sample should be used instead of 30±2g and that the weight should be recorded to the nearest 0.1g instead of the nearest mg.

- Revision 0.1 dated 12 March 2010
  - Updated implementation date
  - Added section 6.1

TABLE 1.

Determinative Methods Using Ultrasonic Extraction

Method Description	Determinative Method	SOP
Chlorinated Pesticides	SW-846 8081A SW-846 8081B	DV-GC-0020
Polychlorinated Biphenyls (PCBs)	SW-846 8082 SW-846 8082A	DV-GC-0021 DV-GC-0030
Polynuclear Aromatic Hydrocarbons by HPLC	SW-846 8310	DV-LC-0009
Diesel and Residual Range Organics	SW-846 8015B SW-846 8015C NWTPH-Dx AK102 AK103 OK Dept. of Environ. Quality DRO Method	DV-GC-0002 DV-GC-0027
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270C SIM	DV-MS-0002

Note: The IS, ICV, and CCV QC elements are addressed in the analytical SOPs.

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Title: Ultrasonic Extraction of Solid Samples [SW-846 3550B & 3550C]

Approvals (Signature/Date):

Susan Oster 11/29/12 Susan Oster Date Technical Specialist	Adam W Alban 03 Dec 12 Adam Alban Date Health & Safety Manager / Coordinator
12/3/12 John P. Morris Date Quality Assurance Manager	12/3/12 William S. Cicero Date Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This SOP is applicable to the solvent extraction of organic compounds from solid samples, including wipes, using sonication (i.e., ultrasonic extraction). This SOP is based on SW-846 Method 3550B and 3550C.

1.2 The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate pH and spiking mixtures are used.

1.3 This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, Concentration of Organic Extracts, for those details.

2.0 Summary of Method

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This mixture is solvent extracted three times using an ultrasonic horn.

3.0 Definitions

3.1 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client.

3.4 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.5 **Aliquot**: A part that is a definite fraction of a whole; as in “take an aliquot of a sample for testing or analysis.” In the context of this SOP, “aliquot” is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 In order to extract especially wet solids, the initial sample weight might have to be reduced in order to achieve a free-flowing mixture with the sodium sulfate. This can raise the reporting limits and method detection limits.

4.3 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of this SOP (Section 9). Specific selection of reagents may be required to avoid introduction of contaminants.

4.4 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.5 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.6 There are many sources of phthalate contamination in the laboratory. The most common of which are nitrile gloves. The analyst should never touch the inside of glassware with gloves. For the analysis of low-level phthalates by method 8270C SIM, common filter paper can introduce phthalate contamination. Therefore when samples are extracted for this analysis, the Method Comments will instruct the analyst that only glass wool can be used.

4.7 It has been observed that 8270 compounds benzoic acid, 2,4-dinitrophenol, and 4,6-dinitro-2-methylphenol will not recover well if the extract does not drain completely and quickly through the sodium sulfate. Therefore it is very important that a thorough rinse is performed – especially after the 1st sonication. Recoveries will also be improved if the filter paper and funnels used allow for quick drainage. It has been observed that Büchner funnels and glass fiber filter paper will slow drainage.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Ultrasonic disrupters can produce high intensity noise and must be used in an area with adequate noise protection. During operation, the horns will be kept in a sound enclosure inside the fume hood to protect the analyst. If a sound enclosure is not used, then hearing protection is required when within 10 feet of an operating ultrasonic disrupter and the analyst must be

in the Hearing Protection Program per DV-HS-0010, Hearing Conservation Program.

5.1.2 Eye protection that satisfies ANSI Z87.1 (as described in the Environmental Health and Safety Manual), laboratory coat, and appropriate gloves must be worn while performing this procedure. Nitrile gloves shall be worn when handling solvents; latex gloves may be worn when handling samples only; and cut resistant gloves shall be worn when washing glassware.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant Poison	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable	50 ppm (TWA)	Prolonged or repeated contact with skin can cause defatting and dermatitis. Contact with eyes can cause redness, tearing, and blurred vision. Exposure can cause lung irritation, chest pain, and edema, which may be fatal.
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Sonicator, at least 300 watts.

6.2 Sonicator horn, ¾ inch

6.3 Balance, >1400-g capacity, accurate to ± 0.1 g, calibrated daily per SOP DV-QA-0014.

6.4 Beakers, 400 mL.

6.5 Media bottles, 250 mL with Teflon-lined caps.

6.6 Stainless steel conical funnels

6.7 Ashless cellulose filter paper – Whatman Grade 41 or Ahlstrom Grade 54

6.8 Glass wool - For the analysis of low-level phthalates by method 8270 SIM.

6.9 Pipetter with disposable 1.0-mL tips, calibrated daily per SOP DV-QA-0008.

6.10 Aluminum foil.

6.11 Wooden tongue depressors

6.12 Metal spatulas.

6.13 Solvent dispenser pump.

6.14 Filter flask.

6.15 Vacuum pump.

6.16 Computer Software and Hardware

Please refer to the master list of documents and software located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or subsequent revision) for the current software to be used for data processing.

7.0 Reagents and Standards

Reagents - All materials must be reagent grade or higher quality, unless otherwise specified

7.1 Methylene chloride – Each lot of solvent is tested following SOP CA-Q-S-001 or CA-Q-W-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.2 Acetone - Each lot of solvent is tested following SOP CA-Q-S-001 or CA-Q-W-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.3 Hexane - Each lot of solvent is tested following SOP CA-Q-S-001 or CA-Q-W-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.4 Baked Sodium Sulfate, 12-60 mesh - Each lot is tested following CA-Q-W-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas. Heat sodium sulfate in a 400°C oven for at least four hours. Cool, covered tightly with foil, and store in tightly closed jars.

7.5 Baked Ottawa Sand – Heat Ottawa sand in a 400°C oven for at least four hours.

Standards

7.6 Please reference SOP DV-OP-0020 for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils for Method 8082A	Glass with Teflon-lined lids	30 grams	Cool, $\leq 6^{o}C$	None	SW-846
Wipes for Method 8082A	Glass with Teflon-lined lids	N/A	Cool, $\leq 6^{o}C$	None	SW-846
Soils for all other Methods, including 8082	Glass with Teflon-lined lids	30 grams	Cool, $\leq 6^{o}C$	14 days	SW-846
Wipes for all other Methods, including 8082	Glass with Teflon-lined lids	N/A	Cool, $\leq 6^{o}C$	14 days	SW-846

[1] Exclusive of analysis.

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in DV-QA-003P, Quality Assurance Program.

9.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD) Department of Energy (DoE), AFCEE etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be

notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.5 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.6 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.7 Method Blank (MB)

At least one method blank must be processed with each preparation batch.

The method blank for batches of soil samples consists of 30 grams of baked Ottawa sand, which is free of any of the analyte(s) of interest.

TestAmerica Denver typically provides clients with clean filter paper or sterile gauze to use as wipes. In these cases, the laboratory prepares wipe-matrix MBs by spiking clean filter paper or gauze (of the same type that is provided to the client) with the surrogate compounds to be used for analysis. If the client uses a different type of material for the wipes, the client should provide a clean specimen of that material to be used for the MB. If the client does not provide a blank wipe in this case, the laboratory will prepare the MBs from filter paper or gauze, spiked with the surrogate compounds.

Acceptance Criteria: The result for the method blank must be less than the reporting limit for the analyte(s) of interest or less than 10% of the analyte concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: If target analytes in the blank exceed the acceptance limits, an unacceptable method blank must be re-prepared and reanalyzed. If the analyte was <u>not</u> detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.8 Laboratory Control Sample (LCS)

At least one LCS must be processed with each preparation batch. Some projects require two LCSs (LCS and LCSD) in every batch, therefore it is important to check special project instructions for each sample. Specifically, Alaska Methods AK102 and AK103 require an LCS and LCSD.

For soil sample batches, the LCS consists of 30 g of reagent sand to which the analyte(s) of interest are added at a known concentration.

LCSs for wipe-matrix samples are prepared by spiking the compounds of interest and surrogate compounds onto a piece of clean filter paper or sterile gauze. If the client uses a different type of material for the wipes, the client should provide blank wipe material to the laboratory for use in preparing the LCS. If the client does not provide blank wipe material, the laboratory will prepare LCS using clean filter paper or sterile gauze spiked with the compounds of interest and surrogate compounds.

The LCS is carried through the entire analytical procedure just as if it were a sample.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis. MS/MSDs are not performed on wipe samples.

If insufficient sample volume is available for MS/MSD, an NCM must be written. For SW-846 methods a LCS/LCSD will be required in this case with the exception of work done under the AFCEE and DoD programs which allows precision to be calculated using LCSs from different batches over the duration of the project.

Acceptance Criteria: The recovery and RPD results for the MS and MSD must fall within the established control limits. Control limits are set at $\pm$ 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.10 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at $\pm$ 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in

the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control.

9.11 Sample Duplicates

A sample duplicate is a second aliquot of an environmental sample that is processed with the first aliquot of that sample. Sample duplicates are processed as independent samples within the same batch. The sample and duplicate results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample duplicate precision results are not necessarily representative of the precision for other samples in the batch. Sample duplicates are performed when requested by the client.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Critical Procedural Considerations

10.2.1 As stated throughout this SOP, analysts must review the Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).

10.2.2 Analysts must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other beaker or media bottle than the designated one should be cleaned or disposed of before coming into contact with the sample. Gloves should

never come into contact with the inside of beakers, media jars, or steel funnels.

10.2.3 Sodium sulfate should be kept in closed containers when not in use. It is important to close the container when not actively using the sodium sulfate.

10.3 Sonicator Tuning and Horn Inspection

10.3.1 Every week the sonicator horns are inspected for pitting and are replaced if excessive wear is observed.

10.3.2 If the sonicator is not self-tuning, the sonicator must be tuned once a week or whenever a new horn is installed. Tuning is documented in the sonicator maintenance log.

10.3.3 Starting at a power setting of 1, tune the sonicator so that the output is less than 20%.

10.3.4 Repeat the tune at a power setting of 5 and 10. At each power setting, tune the sonicator so that the output is less than 20%.

10.3.5 If the output is over 20%, consult your supervisor and the manufacturer's manual for troubleshooting help.

10.4 Assemble and clean the glassware immediately before use.

10.4.1 Rinse 400-mL thick-walled beakers with methylene chloride.

NOTE: In order to prevent phthalate contamination, never touch the inside of a beaker with gloves on. When rinsing beakers be sure to keep gloves away from the mouth of the beaker.

10.4.2 Without gloves on, fold a 15 cm diameter cellulose filter paper in quarters. Open the folds to create a cone. Place the filter paper in the bottom of a conical stainless steel funnel. Place the funnel on a 250-mL media bottle.

NOTE: For low-level phthalate analysis by 8270 SIM, use glass wool. Check the Method Comments to determine if this is necessary and see Section 4.6.

10.4.3 Place approximately 1 tablespoon of baked sodium sulfate in the funnel. Rinse all surfaces of the funnel, the filter and the sodium sulfate with methylene chloride or acetone/methylene chloride (depending on the extraction solvent, see Section 10.6) so all surfaces of the funnel, filter, and sodium sulfate are rinsed.

NOTE: When preparing glassware for the extraction of wipe samples, sodium sulfate is not necessary and the solvent used in the rinse should be the solvent used in the extraction of the wipe samples. (Normally hexane for methods 8081 and 8082).

10.4.4 Allow the solvent to drain completely into the media bottle. Swirl the media

bottle to ensure all surfaces come into contact with the solvent. Add additional solvent to the rinse if necessary.

10.4.5 Pour the solvent out of the media bottle over the stem of the stainless steel funnel to rinse the funnel stem.

10.4.6 Discard the solvent in the correct waste stream.

10.5 Aliquot Samples

10.5.1 If the sample is a wipe, the sonication can be performed with the wipe in its original container if the original container is large enough. Otherwise, transfer the wipe and any solvent from the original container to a clean beaker.

10.5.2 For each MB and LCS, place a clean wipe into a labeled beaker and proceed to section 10.6.

10.5.3 If the sample is a soil, mix and homogenize samples according to the instructions provided in SOP DV-QA-0023, Subsampling. Use a disposable wooden spatula or a metal spatula that has been rinsed with methylene chloride and dried with a lab tissue.

10.5.4 Break the sample aliquot up into small pieces. The aliquot must not contain particles or clumps bigger than ½ inch in diameter in order to facilitate a complete extraction.

10.5.5 Label a 400-mL beaker with the sample ID, method, and batch number.

10.5.6 Weigh 30 to 33 g of sample into the labeled beaker. Record the weight to the nearest 0.1 g directly into the LIMS or hand record the weight on the benchsheet.

NOTE: Some clients may require the initial aliquot to be adjusted based on the percent moisture of the sample. In those cases, it might be necessary to aliquot more than 33 g of sample. If this is required, the Method Comments will state “Perform Calculation”. The laboratory’s LIMS (TALS) will calculate the required initial weight of wet sample needed to ensure at least 30 g of dry sample is included in the initial aliquot. In TALS, under the Batch Notes, enter a “1” in the “Perform Calculation” field. TALS will then calculate the required initial weight of wet sample needed under the “Target Amount” field in the Worksheet tab. Weigh out at least that mass of wet sample.

10.5.7 Add approximately 1 tablespoon of baked sodium sulfate to the beaker and mix well. If the sample is especially wet, more sodium sulfate will be needed to ensure the sample is free-flowing. If the sample is extremely wet, the initial sample weight might have to be reduced in order to keep the volume of sample and sodium sulfate in the beaker to a level that the horn can still thoroughly disrupt.

10.5.8 For each MB and LCS sample, weigh 30 to 33 g of baked Ottawa sand into labeled beakers. Add 1 tablespoon of baked sodium sulfate to the beaker and mix well.

10.5.9 Cap the beaker tightly with aluminum foil.

10.5.10 Place the beaker on a cart next to the sample container so that a second analyst can check the labels. This is documented on the Organic Extraction Worksheet (See WI-DV-0009).

10.6 Prepare a bottle with a bottle-top dispenser with the appropriate solvent.

10.6.1 Methylene Chloride is used for soil and wipe samples for the following methods:

- SW-846 8015B (8015B_DRO)
- SW-846 8015C (8015C_DRO)
- Alaska Methods AK102 and AK103 (AK102_103)
- NWTPH DRO (NWTPH_Dx)
- Oklahoma DRO Method (Okla_DRO)
- Low-level NDMA by GC/CI/MS/MS (NDMA_CIMSMS)

10.6.2 For soil extraction by all other methods, the solvent used is a 1:1 mixture of methylene chloride and acetone.

10.6.3 For wipe samples by method 8081 and 8082, the solvent used is hexane.

10.6.4 For wipe samples by method 8270, the solvent used is a 1:1 mixture of methylene chloride and acetone.

10.7 Add Surrogate, Spikes, and Solvent to Field Samples and all QC samples.

10.7.1 The standards should be allowed to come to room temperature before spiking the samples. Record the ID of the standard used on the benchsheet.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-009.

10.7.2 Only one batch should be surrogated at a time to ensure the correct standards are used and to ensure the solvent is added as soon as possible to the samples.

10.7.3 Using a calibrated pipette, add the appropriate volume of the appropriate working surrogate standard to the beaker for each field sample and method blank. Do this by punching a hole in the aluminum foil cap with the pipette tip.

10.7.4 Using a calibrated pipette, add the appropriate volume of the appropriate

working spike standard to the beaker for each LCS, LCSD and MS/MSD. Do this by punching a 2nd hole in the aluminum foil cap with the pipette tip.

10.7.5 Immediately after the addition of the spike standard to the LCS, MS, & MSD sample, add approximately 100 mL of the appropriate solvent. Note that the solvent should be added as soon as possible after the addition of the spiking standards to prevent loss of the more volatile extractables. Sufficient solvent should be added so that the solvent level is at least ¾ inch above the solids.

NOTE: When hexane is used as the extraction solvent, use only enough to cover the wipe, i.e., approximately 50 mL. This will help facilitate the concentration of the extract later.

10.8 Rinse the disrupter horn with methylene chloride and wipe down with a clean laboratory tissue.

10.9 Place the bottom surface of the disrupter horn tip just below the surface of the solvent, but above the sediment layer.

10.10 Sonicate for three minutes, making sure the entire sample is agitated. The output should be set at 10 for the ¾-inch standard horn. The mode switch should be set on pulse, and the percent-duty cycle knob at 50%.

10.11 Decant and filter the extract through the prepared stainless steel funnel into the media bottle. Immediately rinse the sodium sulfate in the funnel with at least 50 mL of solvent. **This is a critical step and must be performed as soon as the extract has drained from the funnel and must be done with at least 50 mL of solvent.**

10.12 Repeat the extraction two more times with the appropriate solvent. Each time add sufficient solvent so that the solvent level is at least ¾ inch above the solids. If wipes are being extracted with hexane, then repeat two or more times with additional 50-mL portions of solvent.

10.13 Decant off the solvent after each sonication. After the third and final sonication, pour the entire extract into the funnel. Do not attempt to decant at this step but make every effort to recover all solvent from the beaker. If sufficient room in the media jar exists, rinse the beaker and/or the funnel with an additional 10 to 20 mL of solvent and add the rinse to the funnel.

10.14 Once the solvent has completely drained into the media bottle, dispose of the solid sample and the sodium sulfate into Waste Stream D and cap the media bottle containing the extract with a Teflon-lined lid or with aluminum foil.

10.15 Be sure to rinse the disrupter horn between samples following the procedure in Section 10.8.

10.16 If the extract contains visible solids, it will be necessary to filter the extract again. This filtration can be performed immediately before the concentration step by filtering the extract through another filter paper and funnel directly into the K-D

apparatus. If the extract clogs the filter or filtration is extremely slow, the filter and funnel can be placed on a filter flask and a vacuum can be applied.

10.17 Place the extract in a refrigerator until concentration. Document on the benchsheet in which refrigerator the extracts are stored and the total extract count for the batch.

10.18 Handwritten notes on the benchsheet are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklist (see WI-DV-009).

11.0 Calibration

Not applicable to this procedure.

12.0 Calculations / Data Reduction

Not Applicable.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

Before analyzing samples, the laboratory must establish a method detection limit (MDL). See DV-QA-005P, Determination of Method Detection Limits, for more information on the method detection limit studies.

13.2 Demonstration of Capabilities

An initial demonstration of capability (IDOC) must be performed by each analyst. On-going proficiency must be demonstrated by each analyst on an annual basis. See M-Q-001, TestAmerica Quality Management Plan, and the TestAmerica Denver Laboratory Quality Assurance Manual (QAM) for more information on the IDOCs.

13.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

14.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees

will abide by this method, the policies in section 13 of the Environmental Health and Safety Manual for "Waste Management and Pollution Prevention", and the Waste Management procedure, DV-HS-001P.

15.2 Waste Streams Produced By This Method

15.2.1 Methylene chloride – Waste Stream B

15.2.2 Flammable solvent – Waste Stream C

15.2.3 1:1 MeCl2:Acetone – Waste Stream CA

15.2.4 Solid waste/sodium sulfate – Waste Stream D

15.2.5 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

16.0 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3550C Ultrasonic Extraction, Revision 3, February 2007.

16.2 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3550B Ultrasonic Extraction, Revision 3, December 1996.

16.3 Alaska Method AK102, "For the Determination of Diesel Range Organics", Version 04/08/02.

16.4 Alaska Method AK103, "For the Determination of Residual Range Organics", Version 04/08/02.

16.5 Oklahoma Department of Environmental Quality, Methods 8000/8100 (modified) Diesel Range Organics (DRO), October 22, 1997 Rev. 4.1.

16.6 NWTPH-HCID "Hydrocarbon Identification Method for Soil and Water," Manchester Environmental Laboratory, Dept. of Ecology, State of Washington.

17.0 Method Modifications:

17.1 SW-846 Method 3550C Section 11.3 instructs that the surrogate and spike compounds should be added to the sample before the sample is mixed with sodium sulfate. This SOP calls for the sample to be mixed thoroughly with sodium sulfate before the surrogate and spike compounds are added. This is done per EPA Memo dated August 5, 2010 titled "Spiking (Prior To vs. After Sample Drying) Issue in SW-846 Organic Extraction Methods."

17.2 SW-846 Method 3550C calls for the use of Büchner funnels and vacuum filtration of all extracts. This SOP calls for the use of conical funnels. This was done to

prevent the extract from becoming trapped in the sodium sulfate in the Büchner funnel and specifically to improve the recoveries of benzoic acid, 2,4-dinitrophenol, and 4,6-dinitro-2-methylphenol.

17.3 Oklahoma Department of Environmental Quality method calls for the aliquot not to exceed 20 g. This procedure calls for the soil aliquot to be 30 g to 33 g.

17.4 Oklahoma Department of Environmental Quality DRO method calls for solvent to be added to the sample in a 1:1 ratio (milliliters of solvent to grams of sample). This procedure calls for 100 mL of solvent to be added to 30 g of sample.

17.5 Method from the state of Washington uses a 10 g soil sample that is shaken and processed in a sonic bath. This procedure calls for the soil aliquot to be 30 g to 33 g and is processed directly with a sonicator horn.

17.6 Methods 3550B and 3550C instruct the lab to determine the dry weight of the sample. This is performed according to SOP DV-WC-0023 and is not included in this SOP.

17.7 The medium/high concentration extraction procedure described in Methods 3550B and 3550C is not addressed in this SOP.

18.0 Revision History

18.1 Rev 4, dated 30 November 2012

- Updated Section 8 to indicate per SW-846 Revision 4, soils and wipes for analysis under Method 8082A do not have a holding time.
- Updated Section 9.9 to indicate that the DoD does not require LCSD.
- Updated Section 10.5.7 to indicate that the initial sample weight might have to be reduced for extremely wet samples.
- Section 10.5.5 was revised to remove the requirement to document the extraction date on the extract label.
- Updated Section 15 to include Waste Stream CA.

18.2 Rev 3.1, dated 30 November 2011

- Source method review
- Removed references to Method 8070; method no longer active at lab.
- Updated Section 9 to state that MBs and LCSs for wipe samples are created either from filter paper or sterile gauze.
- Added Section 9.11 to include definition and requirements for sample duplicate.
- Added a Note to Section 10.5.6 to describe how to adjust the initial aliquot mass to compensate for percent moisture.
- Updated Section 17 to exclude dry weight determination, high concentration method and Method NWTPH-HCID.
- Updated method references to include NWTPH-HCID.
- Updated SOP references in Table 1 to reflect active SOPs.
- Formatting and grammatical changes throughout

18.3 Rev 3 Dated 29 October 2010

- The procedure was revised to reference both method 3550B and 3550C.
- Section 4.6 was revised to change the requirement for binder free filter paper to glass wool when performing the extraction for low-level phthalate analysis by 8270 SIM. A note was added to Section 10.4.2 as well.
- Section 4.7 was added and Section 10 was revised to call for the use of stainless steel conical funnels and cellulose filter paper instead of Büchner funnels and glass fiber filter paper. This was done to prevent the extract from becoming trapped in the sodium sulfate in the Büchner funnel and specifically to improve the recoveries of benzoic acid, 2,4-dinitrophenol, and 4,6-dinitro-2-methylphenol.
- Instructions were added to Section 10.11 to emphasize the importance of rinsing the funnel and sodium sulfate immediately after the first sonication.
- Section 10.3 was revised to include requirements for inspecting the horns on a weekly basis.
- This procedure was revised to instruct the analyst to mix the sample thoroughly with sodium sulfate before adding the surrogate and spike compounds. This change was made per EPA Memo dated August 5, 2010 titled “Spiking (Prior To vs. After Sampling Drying) Issue in SW-846 Organic Extraction Methods.
- Details about the surrogate and spike standards used in this procedure have been moved to SOP DV-OP-0020

18.4 Rev 2 Dated 14 December 2009

- Section 1.2 was revised to state that this procedure can be used for additional methods when appropriate solvents and spiking mixtures are used instead of different pH and spiking mixtures as this procedure does not require any pH adjustments.
- Section 4 was revised to include notes about phthalate contamination.
- Section 6 was revised to include special glass fiber filter paper that should be used for the extraction of low-level phthalates.
- Section 7 was revised to remove the reference to the AFCEE spike mix for method 8270. The laboratory now only maintains one standard spike mix for method 8270.
- Section 7 was revised to include the benzidine LCS standard.
- Section 7 was revised to correct the expiration date for standards for NDMA by GC/CI/MS/MS from 6 months to 1 year. This was done to match the requirements in DV-QA-0015
- Section 10.5 was revised to change the initial aliquot from 30 +/- 2.0 grams to 30 to 33 grams. This was done to ensure that all samples have reporting limits and method detection limits no higher than the laboratory’s standard reporting limits and method detection limits.
- Section 10 was revised to stress how important it is that the solvent is added to the sample as soon as possible after the addition of the surrogate and spike compounds. This was done by only requiring samples to be stirred with a spatula if they were too wet to form a free-flowing mixture with the sodium sulfate after swirling the beaker.

18.5 Rev 1 Dated 31 January 2009

- Section 7.10.11 was corrected. This section had incorrectly described the making of the SIM PAH working standards.

19.0 Attachments

Table 1: Determinative Methods Using Ultrasonic Extraction

TABLE 1.

Determinative Methods Using Ultrasonic Extraction

Method Description	Determinative Method	SOP
Diesel Range Organics, Jet Fuels, Motor Oil, Residual Range Organics	SW-846 8015B SW-846 8015C Alaska Methods AK102 & AK103	DV-GC-0027
Chlorinated Pesticides	SW-846 8081A SW-846 8081B	DV-GC-0020
Polychlorinated Biphenyls	SW-846 8082 SW-846 8082A	DV-GC-0021 DV-GC-0030
Polynuclear Aromatic Hydrocarbons	SW-846 8310	DV-LC-0009
Semi-volatiles by GC/MS	SW-846 8270C SW-846 8270D	DV-MS-0011 DV-MS-0012
Polynuclear Aromatic Hydrocarbons by GC/MS	SW-846 8270D SIM	DV-MS-0002
N-Nitrosodimethylamine by GC/CI/MS/MS	TestAmerica Denver SOP	DV-LC-0019

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THE LEADER IN ENVIRONMENTAL TESTING

Title: Extraction of Aqueous Samples by Microextraction, SW846 3511

Approvals (Signature/Date):

Susan Oster — Date 8/1/12
Organic Extractions Manager

Adam Alban — Date 01 Aug 12
Health & Safety Manager / Coordinator

John Morris — Date 8/1/12
Quality Assurance Manager

Robert C. Hanisch for — Date 8.1.12
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

This Standard Operating Procedure (SOP) is applicable to the solvent extraction of organic compounds from water samples using a 35mL microextraction. This SOP based on SW-846 Method 3511.

The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate pH and spiking mixtures are used.

This procedure does not include the cleanup steps. See SOP DV-OP-0007, "Concentration of Organic Extracts", for details concerning the concentration and cleanup of extracts.

2.0 Summary of Method

Samples are collected in 40mL vials. Approximately 5mL of sample volume is removed from the vial to make room for reagents. Sodium chloride, surrogates, and solvent are added to the vial which is then placed on an orbital shaker for 10 minutes. The solvent is then collected and dried and sent on for analysis. The water phase is discarded. The microscale approach minimizes sample size and solvent usage, thereby reducing the supply costs, health and safety issues, and waste generated.

3.0 Definitions

3.1 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. Please reference WI-DV-0032 for details on Method Comments.

3.4 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.5 **Aliquot**: A part that is a definite fraction of a whole; as in "take an aliquot of a sample for testing or analysis." In the context of this SOP, "aliquot" is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.3 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented in an NCM.

4.4 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. Especially take note of the possibility of phthalate contamination from gloves. Gloves should be changed out frequently and whenever they come in contact with solvent. Glassware should be handled in a fashion that keeps gloves away from the interior and mouth of the glassware.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Secure tumbler and extraction apparatus before starting rotary agitation apparatus.

5.1.2 Do not attempt to manually stop a rotating piece of equipment. Keep all hanging objects, such as ties, hair, necklaces, etc., away from rotating equipment. Guards must be used when the apparatus is rotating to prevent loose clothing or limbs from getting caught.

5.1.3 Glass vials can break when the caps are being tightened. Cut resistant gloves should be worn whenever caps are being tightened.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the

method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Serious or Significant Hazard Rating

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Hexane	Flammable Irritant	50ppm TWA	Causes irritation to eyes, skin and respiratory tract. Aspiration hazard if swallowed. Can enter lungs and cause damage. May cause nervous system effects. Breathing vapors may cause drowsiness and dizziness. Causes redness and pain to the skin and eyes.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit			

6.0 Equipment and Supplies

6.1 Supplies

- pH indicator paper, wide range.
- Disposable Pasteur pipettes.
- Autosampler vials, glass, crimp top cap with PTFE-faced septa.
- Sample Extraction Vials – 40mL VOA vials with PTFE-lined septum screw top.
- Class A Graduated cylinder, 50mL in size.

6.2 Equipment

- Orbital shaker table, capable of maintaining 200 rpm for 10 minutes.
- Balance, ≥ 1400 g capacity, accurate to ± 1 g, calibration checked daily per SOP DV-QA-0014.
- Mechanical pipette, 100 uL, positive displacement, with disposable tips, calibrated per SOP DV-QA-0008.
- Manual, adjustable positive-displacement pipette or bottle-top re-pipettor, used to dispense 2 mL. Calibration is checked following the steps detailed in DV-QA-0008.
- Manual, adjustable positive-displacement pipette or bottle-top re-pipettor, used to dispense 35mL. Calibration is checked following the steps detailed in DV-QA-0008.

6.3 Computer Software and Hardware

- Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagent Water

7.1.1 TestAmerica Denver has two ELGA water purification systems. The water coming from the ELGA system should be 18-18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026.

7.2 Hexane

For solvents packaged in bottles, each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas. For solvents packaged in CYCLETAINERS, the first batch of samples prepared with a new lot of solvent is monitored and reported to the QA group per the instructions in CA-Q-S-001 DV-1. If any problems are identified, use of the solvent is suspended until further testing can be done and determines the solvent is acceptable.

7.3 Baked Sodium Sulfate, 12-60 mesh

Heat sodium sulfate in a 400 °C oven for at least four hours. Store in tightly closed container.

7.4 Baked Sodium Chloride

Bake in 400 °C oven for at least 4 hours.

7.5 Standards

Please reference SOP DV-OP-00020 and WI-DV-009 for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix and Method	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water for Method 8082 or 8082A	40mL Glass	35 mL	Cool 4 ± 2°C	None [2]	SW-846 Chapter 4, Revision 4, Feb 2007
Water for Method 8081B or 8081C	40mL Glass	35 mL	Cool 4 ± 2°C	7 Days	SW-846 Chapter 4, Revision 4, Feb 2007

[1] Exclusive of analysis.
[2] Some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require a 1 week hold time for method 8082 and 8082A. The states of California, South Carolina, Pennsylvania, and Connecticut require a 1 week hold time.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, refer to the Method Comments and QAS to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Assurance Program.*

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, *Requirements for Federal Programs*.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS and in the Quality Assurance Summaries (QAS) available in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank is processed and analyzed just as if it were a field sample.

The method blank for batches of aqueous samples consists of 35mL of reagent water free of any of the analyte(s) of interest. The method blank for batches of TCLP leachates consists of 20mL of the blank leachate. The method blank for batches of SPLP leachates consists of 35mL of the blank leachate.

Acceptance Criteria: The result for the method blank must be less than the reporting limit for the analyte(s) of interest or less than 10% of the analyte concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: If target analytes in the blank exceed the acceptance limits, an acceptable method blank must be re-prepared and reanalyzed. If the analyte was not detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.5 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

At least one LCS must be processed with each preparation batch. The LCS is carried through the entire analytical procedure just as if it were a sample.

For aqueous sample batches, the LCS consists of 35mL of reagent water to which the analyte(s) of interest are added at known concentration. The LCS for batches of TCLP leachates consists of 20mL of the blank leachate to which the analyte(s) of interest are added at known concentration. The LCS for batches of SPLP leachates consists of 35mL of the blank leachate to which the analyte(s) of interest are added at known concentration.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at $\pm$ 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at $\pm$ 4 standard deviations around the mean of

historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared unless Method Comments indicate otherwise.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at ± 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Critical Procedural Considerations

10.2.1 As stated throughout this SOP, analysts must review the Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).

10.2.2 Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other vial than the designated one should be cleaned or disposed of before coming into contact with the sample.

10.3 Prepare MB and LCS and LCSD samples

10.3.1 For each water MB, LCS and LCSD sample required, place 35mL of reagent water in a 40mL glass VOA vial. Use a Class A graduated cylinder or a bottle-top pump that has been calibration checked to measure the 35mL of reagent water

10.3.2 For TCLP leachates, use a 50mL or Class A graduated cylinder to measure out 20mL to 21mL of the appropriate leach fluid for each MB and LCS and LCSD. Alternatively, the 20mL of leach fluid can be aliquoted gravimetrically by taring a vial on a balance and pouring blank fluid into the

vial until the balance reads 20.0g to 21.0g. Record the volume to the nearest mL. Place the leachate bottle beside the vial so a second analyst can check that the correct leach fluid was used.

10.3.3 For SPLP leachates, use a 50mL Class A graduated cylinder to measure out 35mL of the appropriate leach fluid for each MB and LCS and LCSD. Alternatively, the 35mL of leach fluid can be aliquoted gravimetrically by taring a vial on a balance and pouring blank fluid into the vial until the balance reads 35.0g to 36.0g. Record the volume to the nearest 1 mL. Place the leachate bottle beside the vial so a second analyst can check that the correct leach fluid was used.

10.4 Aliquot and measure the initial sample pH of TCLP or SPLP Leach samples.

10.4.1 Measure the initial sample pH with wide-range pH paper and record the pH on the extraction bench sheet.

10.4.2 For TCLP leachates, use a 50mL Class A graduated cylinder to measure out 20mL of the leachate. Record the volume to the nearest 1mL. Place the leachate bottle beside the vial so a second analyst can check that the correct leach fluid was used.

10.4.3 For SPLP leachates, use a 50mL Class A graduated cylinder to measure out 35mL of the leachate. Record the volume to the nearest 1mL. Place the leachate bottle beside the vial so a second analyst can check that the correct leach fluid was used.

10.5 Aliquot and measure the initial sample pH of water samples.

10.5.1 For water samples, place the sample vials in a vial holder and remove the caps from the sample VOA vials. Be sure to place the cap next to the vial so caps are returned to the correct vial later.

10.5.2 Using a new disposable serological pipette for each sample, remove approximately 5mL of sample from each vial. If the vial was not received completely full remove only enough volume to allow approximately 5mL of headspace in the vial.

10.5.3 Measure the initial sample pH with wide-range pH paper and record the pH on the extraction bench sheet.

10.5.4 Re-cap the vials.

10.5.5 Weigh the vial containing the approximately 35mL of sample and record the gross weight to at least the nearest 0.1 gram.

10.6 Add Surrogates to All Field Samples and QC Samples

10.6.1 Place the vials for all samples and all QC samples in a vial holder and remove the caps from the vials. Be sure to place the cap next to the vial so caps are returned to the correct vial later.

10.6.2 The standards should be allowed to come to room temperature before spiking the samples. Record the ID of the standard used on the benchsheet.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-009.

10.6.3 Only one batch should be surrogated at a time to ensure the correct standards are used.

10.6.4 Add the appropriate volume of the appropriate working surrogate standard to all field samples and QC samples. Record the ID of the standard used on the bench sheet. Reference work instruction WI-DV-009 to determine the appropriate standard and the appropriate volume.

10.7 Add Spikes to all LCS's and MS/MSDs

10.7.1 Add the appropriate volume of the appropriate working spike standard to the MS/MSD, LCS and/or LCSD samples. Record the ID of the standard used on the bench sheet. Reference work instruction WI-DV-009 to determine the appropriate standard and the appropriate volume.

10.8 Adjust pH of Field Samples and QC Samples

10.8.1 If the samples have a pH less than 5 or greater than 9, adjust the sample pH using a minimum amount of 1:1 sulfuric acid or 10 N sodium hydroxide, as necessary. Record the adjusted pH and the lot number of the acid or base on the bench sheet.

10.9 Add 10g of sodium chloride to each sample and QC sample

10.9.1 Add 10 – 10.25g of NaCl to all samples and all QC samples. Record the lot number of the sodium chloride on the bench sheet.

10.10 Add Solvent to all Field Samples and QC Samples

10.10.1 Using a calibrated pipette or bottle-top pump, add exactly 2mL of hexane to each vial. Record the lot number of the hexane used on the benchsheet.

10.10.2 Cap the vials tightly while wearing cut-resistant gloves.

10.11 Vortex each vial for 1-3 seconds.

10.12 Shake the sample vials for at least 10 minutes.

10.12.1 Place the sample vials in a box and attach the box to an orbital shaker as described in Section 6.2. Shake the samples at 200 rpm for at least 10 minutes. At the end of the 10 minutes, check to see that all the sodium chloride has dissolved. If the sodium chloride has not completely dissolved, shake the samples for an additional 5 minutes. If the sodium chloride has not dissolved completely after 15 minutes of total shaking time, document this anomaly in an NCM.

10.12.2 Allow the organic phase and water phase to separate for at least 5 minutes. If the phases do not separate completely, the vials can be centrifuged to facilitate the phase separation.

10.13 Dry the extracts.

10.13.1 For each sample and QC sample, prepare a 2mL clear auto-sampler vial by adding a very small amount of sodium sulfate and labeling the vial with the sample ID.

10.13.2 Using disposable Pasteur pipettes, transfer the hexane extract into the prepared 2mL vial. Take care not transfer water into the 2mL vial.

10.13.3 Cap and crimp the vials closed. Shake to mix the sodium sulfate and the extract.

10.14 Calculate the initial sample volume.

10.14.1 Empty the remaining sample from the vial into waste stream X or Y. Place the vials up-side down in a vial rack to drip dry. Replace the caps and weigh the empty capped vial and record the weight to the nearest 0.1g.

10.14.2 Calculate the initial sample volume by subtracting the empty bottles weight from the full bottles weight, assuming a density of 1g=1mL.

11.0 Data Analysis and Calculations

$$InitialVolume(mL) = FullBottle(g) - EmptyBottle(g)$$

12.0 Method Performance

12.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL). See Policy DV-QA-005P, “Determination of Method Detection Limits”, for more information on the method detection limit studies.

12.2 An initial demonstration of capability (IDOC) must be performed by each analyst. On-going proficiency must be demonstrated by each analyst on an annual basis. See DV-QA-0024, “Employee Training”, for more information on the IDOCs.

12.3 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

13.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Neutral aqueous sample waste – Waste Stream X or Waste Stream Y.

14.2.2 Hexane waste – Waste Stream C.

14.2.3 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 SW-846, Method 3511, Organic Compounds in Water by Microextraction, Revision 0, November 2002.

16.0 Modifications:

16.1 Modifications from SW-846 Method 3511

16.1.1 Section 11.5 and Section 11.7 of the source method instructs the analyst to work with a single field or QC sample at a time in order to avoid volatile constituent losses. This procedure is not written for the extraction and analysis of volatile constituents, therefore this requirement has been removed.

16.1.2 Section 11.9 of the source method instructs the analyst to shake the vials for 5 minutes or until the sodium chloride has completely dissolved. This procedure calls for the sample to be shaken for 10 minutes or until the sodium chloride has dissolved.

16.1.3 Section 11.10 of the source method instructs the analyst to centrifuge the vials to get the phases to completely separate. This procedure instructs the analyst to centrifuge only if an emulsion forms.

17.0 Attachments

Table 1. Determinative Methods Using Micoextraction

18.0 Revision History

- **Revision 1, August 1, 2012**
  - The procedure was revised to instruct the analysts to shake the samples for 10 minutes on an orbital shaker at 200 rpm instead of rotating the sample end over end for 10 minutes. This was done to improve overall method performance.
  -
- **Revision 0, May 25, 2012**

TABLE 1.

Determinative Methods Using Microextraction

Method Description	*Determinative Method*	*SOP*
Chlorinated Pesticides	SW-846 8081A SW-846 8081B	DV-GC-0020
Polychlorinated Biphenyls	SW-846 8082 SW-846 8082A	DV-GC-0021

Electronic Copy Only

Title: Chemical Oxygen Demand by Method 410.4

[EPA 410.4]

Approvals (Signature/Date):

Dave Elkin — Date: 10/4/10 Wet Chemistry Supervisor	Adam Alban — Date: 04Oct10 Health & Safety Manager / Coordinator
Karen Kuoppala — Date: 10/6/10 Quality Assurance Manager	Robert C. Hanisch — Date: 10/7/10 Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

- This standard operating procedure (SOP) provides instructions for the determination of chemical oxygen demand (COD) in aqueous samples. The test determines the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant.
- This method is applicable to surface waters, and domestic and industrial wastes.
- This method covers a practical COD concentration range of 10 - 200 mg/L. Higher concentrations can be determined by dilution of the sample. The reporting limit is 10 mg/L.

2.0 Summary of Method

2.1 This method is based on reduction of Cr+6 to Cr+3. The COD tubes contain a pre-measured concentration of Cr+6. COD in the samples causes Cr+6 to be reduced, so that the Cr+6 concentration decreases with increasing COD concentration. The absorbance at 420 nm is proportional to the Cr+6 concentration and inversely proportional to COD concentration.

2.2 COD is related to TOC and BOD in typical samples consisting of readily oxidized materials. COD should be higher than BOD, and BOD should be higher than TOC. The exact ratios depend on the actual composition of the samples. For EPA and similar QC samples, the ratios are approximately BOD = 0.6 x COD and TOC = 0.4 X COD.

3.0 Definitions

3.1 **COD:** "Chemical Oxygen Demand" is defined as the amount of a specified oxidant that reacts with the sample under controlled conditions.

3.2 **TOC:** "Total Organic Carbon" is a measure of organic carbon in a sample.

3.3 **BOD:** "Biochemical Oxygen Demand" is a measure of oxygen consumed by microorganisms under specific conditions.

4.0 Interferences

4.1 Organic contamination in the glassware or water used for dilutions will cause high results. Contamination of the calibration standards will cause low results. Even low concentrations of organic material will cause detectable errors.

4.2 Some inorganic compounds will contribute to COD. If COD is meant to be an indicator of organic contamination in these cases, the result will be too high. Reduced inorganic species such as ferrous iron, sulfide, and manganous manganese are oxidized quantitatively under the test conditions.

4.3 Pyridine and related compounds resist oxidation.

4.4 Volatile materials may be lost during sampling and in subsequent handling. Volatile organics are present in the vapor state under test conditions and do not come into contact with the oxidizing liquid.

4.5 Ammonia, present either in the waste or liberated from nitrogen-containing organic matter, is not oxidized in the absence of significant concentrations of free chloride ions.

4.6 Nitrite in concentrations of more than 2 mg/L is a positive interference.

4.7 Chloride is quantitatively oxidized by dichromate and will cause high results. Mercuric sulfate is present in the tubes to prevent interference from up to a maximum chloride concentration of 2000 mg/L. Samples containing more chloride than this must be diluted prior to analysis.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The COD digestion tubes used in this analysis contain a proprietary mixture of compounds of mercury, silver, and chromium, which are highly toxic. Wear gloves and handle the tubes carefully. DO NOT POUR USED TUBES INTO SINKS. Take care not to overheat the tubes. This may cause them to leak or break. The block digester must be placed in a fume hood. The tubes will get very warm when sample is added. Carefully inspect tubes to ensure that they are not leaking before handling.

5.1.3 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.4 The block digester is set to a maximum of 152 °C. Take care not to touch the hot surfaces directly to avoid burns.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

HACH COD Reagent	Corrosive Poison	0.1 mg/m^3 (Hg) 0.01 mg/m^3 (Ag) 1 mg/m^3 (H_2SO_4)	Toxic by inhalation. Causes severe burns and may cause difficult breathing, mouth soreness, and teeth erosion. Causes severe burns in contact with skin. Easily absorbed through the skin and may cause abdominal pain, circulatory disturbances, diarrhea, loosening of the teeth, nausea, vomiting, rapid pulse rate, toxic nephritis, shock, collapse, kidney damage, and death. Causes severe burns in contact with eyes.
1 – Always add water to acid to avoid violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Analytical balance capable of accurately weighing to the nearest 0.0001 g. Verify the accuracy of the balance each day it is used as described in SOP DV-QA-0014.
- Block digestor for 16 mm tubes, set at 150 ± 2 °C.
- Thermometer to measure block digestor temperature. Thermometers require periodic calibration checks against NIST thermometers as described in SOP DV-QA-0001.
- Spectrophotometer with adapter for 16 mm tubes, for absorbance measurements at 420 nm.

6.2 Supplies

- Volumetric Flasks (Class A): varying volumes.
- Eppendorf Pipettes, varying volumes
- Chloride test strips.

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

7.1 Premixed Low Level COD tubes, Hach (Method 8000) Corporation #21258-15.

7.2 Deionized Water

Deionized water must be free of significant organic carbon. Do not use water that has been stored in plastic squirt bottles; obtain fresh water from the tap. The TestAmerica Denver house deionized water meets the requirements of ASTM Type II water with a minimum resistivity of 10 Megohm-cm. Deionized water straight from the tap is used instead of ELGA water as the cartridges in the ELGA system may contribute small amounts of COD to the water.

7.3 COD Spike Solution, 10,000 mg/L

Dry reagent grade potassium hydrogen phthalate (KHP) to a constant weight at 120 °C. Dissolve 0.8503 g of KHP in water and dilute to 100 mL in a volumetric flask. If the final

concentration varies from 10,000 mg/L, subsequent dilutions must be adjusted accordingly. This solution may be purchased commercially.

7.4 COD Stock Standard, 1,000 mg/L

Use a certified standard purchased from a commercial source.

7.5 COD Working Calibration Standards

Dilute the 1,000 mg/L COD Stock Standard (Section 7.4) according to the table below to obtain the indicated concentrations. The working standards must be prepared fresh before each analysis.

Level	Volume of Stock (mL)	Final Volume (mL)	Final Conc (mg/L)
1	1.0	100	10
2	2.0	100	20
3	5.0	100	50
4	10	100	100
5	15	100	150
6	20	100	200

7.6 COD Laboratory Control Sample (LCS), 100 mg/L

The LCS is prepared in the same manner as the Level 4 calibration standard (100 mg/L) as described in Section 7.5.

7.7 COD Initial Calibration Verification (ICV) Stock Standard, 1000 mg/L

The ICV standard solution is a second-source standard that is obtained from a commercial source that is different from the source of the COD Stock Standard (Section 7.4)

7.8 Working ICV Standard, 100 mg/L

Dilute 10 mL of the 1000 mg/L ICV Stock Standard (Section 7.7) to 100 mL with deionized water.

7.9 Matrix Spike and Matrix Spike Duplicate Samples (MS/MSD), 50 mg/L

Matrix spikes are prepared by diluting 0.5 mL of the 10,000 mg/L COD Spike Solution (Section 7.3) to 100 mL with the sample selected for spiking.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Amber Glass	250 mLs	H_2SO_4, pH < 2; Cool 4 ± 2°C	28 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Control Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

9.1.1 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See Policy DV-QA-003P for further details.

9.1.2 Method Blank

A method blank is required with every batch of 20 or fewer samples. The blank is deionized water taken through the procedure as if it were a sample.

Acceptance Criteria: The method blank must not contain COD above the reporting limit or above one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL).

Corrective Action: If the method blank exceeds allowable levels, all associated samples must be re-digested and reanalyzed

9.1.3 Laboratory Control Sample (LCS)

One LCS is required with each analytical batch. The LCS is prepared at a concentration of 100 mg/L as described in Section 7.6.

Acceptance Criteria: The recovery of the LCS must be within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean percent recovery, and must be no wider than 90 to 110 %.

Corrective Action: If the LCS recovery falls outside of the established limits, all associated samples must be re-digested and reanalyzed

9.1.4 Matrix Spike and Matrix Spike Duplicate Samples (MS/MSD)

One MS/MSD pair is required for every 10 field samples. MS/MSDs are prepared to contain an additional 50 mg/L COD above the native COD concentration of the parent sample as described in Section 7.9.

Acceptance Criteria: The recovery of the MS and MSD must be within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean percent recovery, and must be no wider than 90 to 110 %.The relative percent difference (RPD) between the MS and MSD must be no greater than 20%.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.2 Instrument QC

All calibration standards, QC solutions, and field samples are analyzed using a 2 mL portion of sample that is digested.

9.2.1 Initial Calibration (ICAL)

The six calibration concentration levels 10 to 200 mg/L as shown in Section 7.5.

9.2.2 Initial Calibration Verification (ICV)

The second-source ICV standard (Section 7.8) is analyzed immediately following the ICAL.

Acceptance Criteria: The COD recovery in the ICV standard must be 90 - 110 %.

Corrective Action: If the recovery is outside of the acceptance limits, investigate the problem and repeat the ICAL. If the problem is traced to the digested standards, the entire batch must be re-prepared and reanalyzed.

9.2.3 Continuing Calibration Verification (CCV)

Analyze the CCV standard after every 10 or fewer samples and after the last sample. The CCV standard is prepared by digesting 2 mL of the 100 mg/L Level 4 calibration standard listed in Section 7.5.

Acceptance Criteria: The COD recovery in the CCV standard must be 90 - 110%.

Corrective Action: If the recovery is outside of the acceptance limits, repeat the ICAL and reanalyze all samples analyzed since the last successful CCV.

9.2.4 Continuing Calibration Blank (CCB)

Analyze a CCB is after each CCV. The CCB consists of 2 mL of deionized water that is digested and analyzed in the same manner as a sample.

Acceptance Criteria: The result for the CCB must be less than the reporting limit.

Corrective Action: If the blank is above the acceptance limit, check for carryover or the need for instrument maintenance. Recalibrate the instrument and reanalyze all samples analyzed since the last successful CCV.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 All glassware must be thoroughly rinsed with deionized water to remove traces of detergents and other potential contaminants.

10.1.2 Turn on the block digestor and preheat to 150 ± 2 °C.

10.1.3 Prior to starting the digestion, screen all samples for chloride.

NOTE: The chloride screen may be omitted only when chloride data for the samples are available or when comparable results, such as TDS or conductivity, indicate it is not possible for the samples to contain > 2000 mg/L of chloride.

10.1.4 Use chloride test strips to screen for chloride. Record the chloride result on the bench sheet. Samples that are high in chloride are also noticeable for the large amount of precipitate that is formed when the sample is added to a COD tube.

10.1.5 Samples containing more than 2000 mg/L chloride must be diluted in order to bring the chloride concentration below this level. Do not over-

dilute the samples. Generate an NCM anomaly for any sample that requires dilution to reduce chloride interference.

10.1.5.1 When possible do not take sample aliquots for dilution that are less than 1.0 ml. It is preferable to take larger sample aliquots when possible to get a proper homogeneous sample.

Example: For a 2x dilution take 1 mL of your sample and dilute it to a final volume of 2 mL using DI water. Sample is ready for analysis

Example: For a 100x dilution take 1 mL of your sample and dilute it to a final volume of 100 mL using DI water. A 2 mL aliquot taken from the 100 mL is then used for analysis.

Standard COD Dilutions

Sample Aliquot	Final volume	Dilution
1.0 mL	2 mL	2x
1.0 mL	5 mL	5x
1.0 mL	10 mL	10x
1.0 mL	20 mL	20x
1.0 mL	50 mL	50x
1.0 mL	100 mL	100x

10.1.5.2 When samples require serial dilutions, typically greater than 100x, try to limit the amount of dilutions required to get to the final dilution.

Example: For a 1000x dilution take 1 mL of your sample and dilute it to a final volume of 100 mL using DI water. From the 100 mL take an aliquot of 1.0 mL and bring it up to a final volume of 10 mL. A 2 mL aliquot taken from the 10 mL is then used for analysis.

Example: For a 10,000x dilution take 1 mL of your sample and dilute it to a final volume of 100 mL using DI water. From the 100 mL take an aliquot of 1 and bring it up to a final volume of 100 mL. A 2 mL aliquot taken from the 100 mL is then used for analysis.

Standard Serial Dilutions

Sample Aliquot	Final Volume	Initial Dilution	Aliquot from initial dilution	Final Volume	Final Dilution
1.0 mL	100 mL	100x	1.0 mL	10 mL	1000x

1.0 mL	100 mL	100x	1.0 mL	20 mL	2000x
1.0 mL	100 mL	100x	1.0 mL	50 mL	5000x
1.0 mL	100 mL	100x	1.0 mL	100 mL	10,000x

10.1.6 Inspect each tube and discard any that are discolored. The presence of a precipitate in the tubes is normal.

10.1.7 Prepare the calibration standards for analysis by pipetting 2.0 mL of each standard solution into separate COD tubes.

10.1.8 Prepare the method blank by pipetting 2.0 mL of deionized water into a COD tube.

10.1.9 Prepare the LCS by pipetting 2.0 mL of the LCS solution (Section 7.6) into a COD tube.

10.1.10 Pipette 2.0 mL each of each field sample into separate COD tubes. Samples containing solid material should be homogenized first.

10.1.11 Using the sample selected for spiking, prepare the MS and MSD as described in Section 7.9, and pipette 2.0 mL of each into separate COD tubes.

10.1.12 Tighten the cap on each COD tube securely, but do not over-tighten. Label the tubes on the space provided with the appropriate identification.

10.1.13 Mix the contents in each tube by slowly inverting two or three times. At this point, the tube will become warm. If any of the tubes leak, the samples affected must be re-aliquotted.

10.1.14 Place the COD tubes in the heated block digestor for two hours and record the start time and the temperature on the bench sheet.

10.1.15 Remove the tubes from the block digestor after digesting for 2 hours and record the end time and temperature on the bench sheet.

10.1.16 Wait 20 minutes to allow the tubes to cool to 120 °C or less, then invert each tube several times while still warm. Place the tubes into a rack.

10.1.17 Wait until the tubes have cooled to room temperature before proceeding.

10.1.18 Inspect each tube and note any sample that shows signs of leakage (reduced volume, salts and/or charring on the outside of the tube). Re-digest any samples showing these signs.

10.1.19 Allow any precipitate in the samples to settle out fully before analysis.

10.2 Calibration

10.2.1 Calibrate the instrument using the six calibration concentration levels (10 to 200 mg/L as shown in Section 7.5).

NOTE: It is generally **NOT** acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest

or lowest on the curve **AND** the reporting limit and/or linear range is adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason is clearly documented, for example a cracked tube, and a minimum of five levels remain.

10.2.2 Digest and analyze the calibration standards in exactly the same manner as the samples as described in Section 10.1 and 10.3.

10.2.3 Use linear least squares regression to calculate a calibration function relating COD concentration to absorbance at 420 nm. Calibration using least-squares linear regression produces a straight line that does not necessarily pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (absorbance at 420 nm) versus the concentration of each standard. The instrument response is treated is the dependent variable (y) and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

Equation 1

$$y = ax + b$$

Where:

y = Instrument response (absorbance at 420 nm).

x = Concentration of the target analyte in the calibration standard.

a = Slope of the line.

b = The y-intercept of the line.

10.2.4 To calculate the concentration in an unknown sample, the regression equation is solved for concentration, resulting in the following equation, where x is now the concentration of the target analyte in the unknown sample extract:

Equation 2

$$x = \frac{y - b}{a}$$

10.2.5 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

10.2.6 **Calibration Acceptance Criteria**
The absolute value of the correlation coefficient for the linear equation must be 0.995 or greater. (Note that the correlation coefficient is always a negative value because of the inverse relationship between COD and chromate concentration.)

10.2.7 **Corrective Actions for Calibration Failures**
If the absolute value of the correlation coefficient is less than 0.995, the spectrophotometer conditions and calibration standards should be rechecked. Correct any problems found and recalibrate. Samples cannot be analyzed until the initial calibration is successful.

10.3 Sample Analysis

10.3.1 Turn on the spectrophotometer and allow it to warm up for 20 minutes.

10.3.2 Set the wavelength to 420 nm and zero using the Level 6 (200 mg/L) calibration standard.

NOTE: For clarification, any standard that is fully oxidized could be used to zero the spectrophotometer. The 200 mg/L standard is used for consistency and to fully challenge the oxidation step.

10.3.3 Wash each tube with water and dry using a Kimwipe before inserting it in the spectrophotometer. Any dirt or scratches on the tubes will affect results, so examine each tube carefully prior to analysis. Cover with the light shield while taking readings.

10.3.4 Measure the absorbance of each standard and sample. Record the absorbance to ± 0.001 units on the bench sheet.

10.3.5 Dilute and reanalyze any sample that has a result greater than 150 mg/L.

10.3.6 Turn off all equipment. Clean all glassware, apparatus, and the work area.

11.0 Calculations / Data Reduction

11.1 Concentration:

Enter the concentration and absorbance of each standard into a linear least squares regression program to generate the calibration function, as described in Section 10. The linear least squares regression equation (see Equation 1) is solved for concentration and results in the following equation that is used to calculate the COD concentration in the measured sample aliquots:

$$C = \frac{y - b}{a}$$

Equation 3

Where:

C = COD concentration in the measured sample (mg/L).

y = Instrument response for the sample (absorbance at 420 nm).

a = Slope of the line.

b =The y-intercept of the line.

11.2 Diluted Samples Concentration:

If the sample was diluted, multiply the calculated concentration by the appropriate dilution factor, as follows:

Equation 4

$$COD = C \times DF$$

Where:

C = COD concentration in the measured sample (mg/L).

DF = Dilution factor (DF is 1 if the sample was not diluted for measurement).

The reporting limit is 10 mg/L. Samples that have a COD concentration less than 10 mg/L are reported as not detected (ND). Samples that have a COD concentration greater than 150 mg/L must be diluted and reanalyzed.

Dilutions required for other reasons (e.g., high chloride, insufficient sample, or other interferences) must be explained in an NCM or other communication to the laboratory Project Manager to ensure that the analytical problem is explained in the final report case narrative.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy DV-QA-005P. An MDL verification is performed once a year to satisfy NELAC 2003 requirement. For DoD and AFCEE projects, an MDL verification is performed quarterly. MDLs are stored in the LIMS.

The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

An initial demonstration of capability for each method must be performed prior to analyzing samples.

12.2.1 The initial demonstration consists of the preparation and analysis of a QC check sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study.

12.2.2 Four aliquots of the QC check sample are analyzed with the same procedures used to analyze samples, including sample preparation.

12.2.3 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

The digestion reagent used in this procedure is a powerful oxidizing reagent containing mercury. This procedure uses much less sample and digestion reagent, approximately one-twentieth as much, than the older macro-COD methods in order to minimize the use of mercury.

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Used or expired COD tubes containing reagent – COD Vials - Waste Stream I

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Methods for the Chemical Analysis of Water and Waste (MCAWW); EPA, 1983.

15.2 Method 410.4, The Determination of Chemical Oxygen Demand by Semi-Automated Colorimetry, Revision 2.0, August 1993.

15.3 Standard Methods for the Examination of Water and Wastewater, 20th Edition; Clesceri, L.S.; Greenberg, A.E.; Eaton, A.D.; Editors; American Public Health Association, American Water Works Association, and Water Environment Federation, 1998.

16.0 Method Modifications:

Item	Method	Modification
1	410.4	The COD digestion tubes have been changed due to the instrument manufacturer's specifications. This change includes the use of premixed low level digestion tubes, which changes the sample volume added and the size of the digestion tube.
2	410.4	The EPA-approved Hach method 8000 is based on measurement of Cr+6 at 420 nm, rather than Cr+3 at 600 nm, which is used for EPA 410.4. A letter from EPA EMSL is on file, which verifies the acceptability of this approach.

17.0 Attachments

Attachment 1: Example COD Bench Sheet
Attachment 2: Example Data Review Checklist

18.0 Revision History

- Revision 6.3, dated 15 October 2010
  - Corrected LCS, MS and MSD acceptance criteria to 90-110% to meet method requirement.
-
- Revision 6.2, dated 02 March 2010
  - Annual Review
  - Added section 6.3

- Revision 6.1, dated 28 February 2009
  - Added Dilution tables in section 10.1
  - Added Hach method references

- Revision 6, dated 15 February 2008
  - Integration for TestAmerica and STL operations.
  - Updated formatting.
  - Added the reference concerning not to use ELGA water in section 7.2.
  - Added location of MDLs to section 12.
  - Updated SOP references to the new naming convention. (ex. DEN-WC-0018 is now DV-WC-0018).
  - Added updated bench sheet and checklist with new logos.

- Revision 5, dated 24 July 2007
  - The reference to Standard Methods Method 5220D has been removed.
  - The company name has been changed from STL to TestAmerica.

- Revision 4, dated 14 November 2006
  - Updated formatting to be consistent with Policy QA-001.
  - Incorporated the Safety Bulletin to meet STL Corporate requirements.
  - Incorporated Interim Changes.
  - Expanded Section 4 to include interferences cited in the source methods.
  - Revised Section 7 to match current practice.
  - Deleted recording the digestion block temperature at startup (Section 10.1.2). The temperature is recorded when samples are placed in the block and when they are removed.
  - Added instruction to examine tubes prior to analysis for scratches, as well as dirt (Section 10.3.3).
  - Added clarifications to instructions for performing the chloride screen to Sections 10.1.3 through 10.1.5.
  - Revised Section 9.1 to include reference to Policy QA-024.
  - Added calibration equations to Sections 10 and 12.

- Revision 3, dated 29 July 2002
  - The summary of the method in Section 2 was expanded.
  - References to relevant QA SOPs and policies were added throughout.
  - The reference to the Chemical Hygiene Plan was changed to the Corporate Safety

Manual.
  - The calibration curve was changed to include a standard at 10 mg/L, the reporting limit.
  - Details concerning preparation of calibration standards and spike solutions were updated in Section 7.
  - QC requirements in Section 9 were clarified, and increased MS/MSD requirements for South Carolina and North Carolina added.
  - Calibration information and controls were moved from Section 9 to 10. Section 10 now includes warnings against removing calibration points. Section 10 also specifies linear regression with absolute value of correlation coefficient > 0.995.
  - Section 11.3.11 was edited to remove provision for rapid cooling of COD tubes.
  - The calculation formula was added to 12.4.
  - The Method Performance Section 13 was expanded to include requirements for MDLs, IDOCs, and training.
  - The Pollution Prevention Section 14 was expanded.
  - The Waste Management Section 15 now identifies the COD waste stream.
- Revision 2, dated 12 September 2000
  - The company name was changed from Quanterra to STL.
  - The lower reporting limit is changed from 5 mg/L to 10 mg/L.
  - The second source standard is identified as an ICV, rather than an LCS.
  - A clarification is made in section 10 for zeroing the instrument.
  - In Section 11.4.1 the samples needing dilutions are changed from 0.000 absorbance to greater than 150 mg/L.

- Revision 1, dated 20 May 1999
  - The benchsheet was amended such that more details regarding the calibration curve would be recorded and Quantims lot numbers and work order numbers would be recorded.
  - The upper limit of the working range was changed from 200 mg/L to 150 mg/L.
  - The reporting limit was revised from 20 mg/L to 10 mg/L.

Attachment 1.

Example COD Bench Sheet

Analyst:	KBERTHA	Calibration Curve Information						Instrument Information	
Date:	09/29/06		Conc.(mg/L)	ABS.	Conc.(mg/L)		ABS.		
QC Lot:		STD1	10	0.572				Instrument:	Spec 301
QC Run:		Std. 2	20	0.526				Wavelength:	420
DATE OF CURVE=	7/11/2006	Std. 3	50	0.441				Parameter:	COD
SOP Information		Std. 4	100	0.285				Corr. Coef:	-0.99947
Number:	Den-WC-0018	Std. 5	150	0.137	MDL =	4		Slope:	-0.00299
Revision:	1.0	STD 6	200	0	RL =	20		Intercept:	0.3913

ICV/ Information: TV = 100 mg/L		Calibration, LCS/Matrix Spike			
Source	Ricca	std #	1973-06	COD Tube Source:	HACH
lot#	1412298 STD 0662-06 NA	made by	HACH lot A5[illegible]6	Lot & Exp:	A5[illegible]6 30-Nov-10
Concentration:	1000mg/L	Concentration:	1,000 mg/L	Made By:	FISHER 10,000 mg/L
Expiration Date:	10/31/2006	Expiration Date	7[illegible]9/2007	STD3203-0[illegible]	lot 66686 exp: 12/[illegible]/2006
True value:	ICV True value: 100 mg/L	LCS	100 mg/L	MS/MSD	50 mg/L

Int./Notes	LIMS ID	CI	Sample Amount (mL)	Sample ABS.	Color Blank ABS.	Concentration (mg/L-mg/kg)	Prep D.F.	Anal D.F.	Final Conc. (mg/L-mg/kg)	% Rec.
12:00										
	ICV		2	0.288		101.30[illegible]15	1	1	101.30	101%
	ICB		2	0.606		-4.90462	1	1	ND	
	DCS		2	0.279		104.30903	1	1	104.31	104%
	DCS		2	0.267		108.31687	1	1	108.32	108%
	MB		2	0.606		-4.90462	1	1	ND	
JE793	D6I280181-1	>3000	2	0.470		40.[illegible]1757	1	10	405.18	
JE796	2	>3000	2	0.290		100.63518	1	5	503.18	
JE797	3	>3000	2	0.385		[illegible].90644	1	5	344.53	
JE799	4	>3000	2	0.396		6[illegible].23259	1	5	326.16	
JE8AA	5	>3000	2	0.422		56.[illegible]1893	1	5	282.74	
JE8AD	6	>3000	2	0.[illegible]89		0.773[illegible]5	1	5	ND	
JE8AE	7	>3000	2	0.425		56.21495	1	5	281.07	
	CCV		2	0.268		107.98288	1	1	107.98	108%
	CCB		2	0.604		-[illegible].23664	1	1	ND	
JE8AH	8	>3000	2	0.[illegible]90		[illegible]7.20351	1	5	336.18	
JE8AK	9	<2000	2	0.606		-4.90462	1	1	ND	
JE71Q	D6I280148-2	<2000	2	0.595		-1.23076	1	1	ND	
MSD	2S		2	0.[illegible]29		50.87116	1	1	50.87	
MSD	2SD		2	0.[illegible]37		51.[illegible]3913	1	1	51.54	
JE711	3	<2000	2	0.59[illegible]		-0.562[illegible]	1	1	ND	
JE712	4	<2000	2	0.586		1.775[illegible]2	1	1	ND	
JE713	5	<2000	2	0.588		1.10[illegible]14	1	1	ND	
JE715	6	<2000	2	0.[illegible]00		[illegible].90070	1	1	ND	
JE717	7	<2000	2	0.58[illegible]		1.10714	1	1	ND	
	CCV		2	0.267		108.31687	1	1	108.32	108%
	CCB		2	0.602		-3.56867	1	1	ND	
JE72[illegible]	8	<2000	2	0.59[illegible]		-1.89874	1	1	ND	
JE722	9	<2000	2	0.589		0.77316	1	1	ND	
JE725	10	<2000	2	0.592		-0.22880	1	1	ND	
JE73F	12	<2000	2	0.[illegible]01		0.10518	1	1	ND	
	CCV		2	0.277		104.97700	1	1	104.98	105%
	CCB		2	0.602		-3.56867	1	1	ND	
			2			197.49132	1	1		
			2			197.49132	1	1		
			2			197.49132	1	1		
			2			197.49132	1	1		
			2			197.49132	1	1		
			2				1	1		

Attachment 2.

Example Data Review Checklist

TESTAMERICA Denver

Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Minimum of five standards in ICAL or as specified in method?				
2. Correlation coefficient ≥ 0.995 ?				
3. Second-source ICV analyzed, and recovery 90-110%?				
4. ICB analyzed immediately after the ICV & results < the RL?				
5. CCV analyzed after every ten samples & recovery ± 10% of true value?				
6. CCB analyzed after every CCV & results < RL?				
B. Sample Results				
1. All samples greater than highest calibration standard diluted and reanalyzed?				
2. Do associated RL / MDLs reflect dilutions or limited sample volume?				
3. All reported results bracketed by in control CCV results?				
4. Sample analyses done within holding time?				
5. Initial pH checks documented for all samples?				
6. Preparation benchsheet completed and included in package?				
7. Special client requirements met?				
8. Were data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures?				
9. Do the prep and analysis dates in QuanTIMS reflect the actual dates?				
10. Are all data being reported highlighted on the benchsheet?				
11. Raw data copies prepared and scanned?				
12. Manual integrations done properly?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples ≥ 20x blank have NCM?				
2. LCS run for batch and within QC limits ?				
3. MS run at required frequency and within limits?				
4. MSD or DU run at required frequency and RPD within 10%?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

7/6/07 Version
L:\QA\Edit\Forms\Wet Chemistry\Calib Curve Checklist

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Biological Oxygen Demand (BOD) and Carbonaceous Biochemical Oxygen Demand (CBOD)

[SM 5210B / EPA 405.1]

Approvals (Signature/Date):

Dave Elkin — 12/21/10 Date
Wet Chemistry Supervisor

Adam Alban — 30 Dec 10 Date
Health & Safety Manager / Coordinator

John Morris — 12/30/10 Date
Quality Assurance Manager

Robert C. Hanisch — 12/30/10 Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method is applicable to the determination of biochemical oxygen demand (BOD) by EPA Method 405.1 and Standard Method 5210 B, and carbonaceous biochemical oxygen demand (CBOD) by Standard Method 5210 B in waste water, effluents, polluted waters, and other aqueous samples.

1.1.2 This method is used to determine the relative oxygen requirements of wastewaters, effluents and polluted waters. The test allows calculation of the effect of organic waste discharges on the oxygen resources of the receiving water.

1.1.3 Under the conditions of this method, BOD or CBOD in the range of 2 to approximately 300 mg/L as dissolved oxygen (DO) can be determined. This can be extended to higher levels by dilution of the sample.

2.0 Summary of Method

2.1 The BOD test measures the molecular oxygen utilized during a specified incubation period for the biochemical degradation of organic material and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron. The CBOD test is identical to the BOD test, except that a nitrification inhibitor is added.

2.2 A series of dilutions is performed on a sample with a nutrient buffer solution. The diluted samples are inoculated with an active microbial population and incubated in the dark at 20 °C for 5 days. The bottles used are sealed to prevent absorption of oxygen during the test.

2.3 The BOD (or CBOD, if nitrification inhibitor is used) of the sample is calculated from dissolved oxygen readings taken before and after the incubation period.

3.0 Definitions

3.1 **Biochemical Oxygen Demand (BOD):** BOD is an empirical test in which standardized laboratory procedures are used to determine the relative oxygen requirements of waste waters, effluents, and polluted waters. It is widely used for measuring waste loadings to treatment plants and for evaluating the BOD removal efficiency of such treatment systems. The BOD test measures oxygen consumed by microbial life while assimilating and oxidizing organic matter in the sample under standardized conditions of dark incubation at 20°C for 5 days. Actual environmental conditions of temperature, biological population, water movement, sunlight, and oxygen concentration cannot be reproduced in the lab.

3.2 BOD is one of several laboratory tests that can be used to characterize the type and extent of organic matter in water samples. Water samples can contain a variety of organic compounds in various states of oxidation. Some of these compounds can be further oxidized by **biochemical oxygen demand (BOD), assimilable oxygen demand (AOD), and chemical oxygen demand (COD).**

Another related analytical value is total organic carbon (TOC), which, as the name implies, measures the total amount of organic carbon in a water sample.

3.3 **Carbonaceous Biochemical Oxygen Demand (CBOD):** The CBOD test is identical to a BOD test, except that a nitrification inhibitor is added. If an inhibitor is not used, the BOD test will measure the sum of the carbonaceous and nitrogenous demands(from oxidation of reduced forms of nitrogen in water samples, such as ammonia and organic nitrogen).

4.0 Interferences

4.1 Variations in environmental conditions, such as temperature, biological population, water movement, sunlight, and oxygen concentration can affect BOD results. Conditions for the test have therefore been standardized to minimize these variations. Exposure to light may result in photosynthetic production of oxygen.

4.2 Bacterial growth requires nutrients such as trace metals, nitrogen, and phosphorus. These are added to the dilution water.

4.3 The pH must be in the range 6.5 to 7.5 to be suitable for the growth of bacteria.

4.4 Residual chlorine will kill microorganisms seeded into the sample. Chlorine must be destroyed before starting the analysis.

4.5 High concentrations of ammonia will contribute to BOD. This can be prevented by using a nitrification inhibitor, which results in a measurement of carbonaceous biochemical oxygen demand (CBOD). Inhibition of nitrification is recommended for samples of secondary effluent, for samples seeded with secondary effluent, and for samples of polluted water. It is the responsibility of the client to understand the nature of the water sample and to request the appropriate analytical method.

4.6 Organic contamination of glassware and reagents will cause high BOD results.

4.7 Oil and volatile organics in samples may cause an erroneous dissolved oxygen reading.

4.8 Some samples may contain compounds which are toxic to seed organisms. This is indicated by increasing BOD results with increasing dilution of the sample. Such samples may require special study and treatment.

4.9 Some compounds are not easily degraded by the seed organisms and will result in low BOD results.

4.10 Any factor which alters the dissolved oxygen concentration in a sample between the two readings will affect the BOD result. Avoid agitation of the sample. Take the initial dissolved oxygen reading as soon as possible after dilution, then stopper and seal the bottle. Some compounds will be oxidized in as little time as 15 minutes after dilution.

4.11 Due to possible contamination contributions the use of soap is prohibited in the washing of the dilution water reservoirs.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Hydroxide (NaOH)	Corrosive Poison	2 mg/m3 (Ceiling)	Inhalation of dust or mist can cause a range of effects from mild irritation to serious damage of the upper respiratory tract. Symptoms may include sneezing, sore throat, or runny nose. Can cause irritation or severe burns and scarring in contact with skin. Causes irritation in contact with eyes and may cause burns that result in permanent impairment of vision, even blindness.
Sulfuric Acid (H2SO4)	Corrosive Water Reactive Poison Carcinogen	0.2 mg/m3 (TWA)	Inhalation damages mucous membranes and upper respiratory tract. Symptoms may include irritation of nose and throat and labored breathing. May cause lung edema. Can cause redness, pain, and severe burns in contact with skin. Prolonged skin contact can result in circulatory collapse with clammy skin, weak and rapid pulse, and shallow respirations. Can cause blurred vision, redness, pain, and severe tissue burns in contact with eyes. Chronic exposure to mists containing sulfuric acid is a cancer hazard.
Acetic Acid, Glacial	Corrosive Poison Flammable Liquid and Vapor	10 ppm (TWA)	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur. Can cause serious damage to skin, including redness, pain, and burns. Contact with eyes may cause severe damage followed by loss of sight.
1 - Always add acid to water to prevent violent reactions. 2 - Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Incubator controlled to 20 ± 1 °C. All light must be excluded. The incubator temperature is to be measured and recorded two times a day at least four hours apart. When the incubator temperature falls outside of 20 ± 1 °C, corrective action is taken as described in Section 10.3.15.1.
- A pH meter calibrated with a range covering 6.5 to 7.5. Narrow range pH paper covering the same range can be used.
- Dissolved oxygen meter. YSI Model 5100 with built-in barometric correction is currently in use, equivalent equipment can also be used. Consult manufacturer's instructions for proper use and storage of the probe. The probe is stored in a BOD bottle containing just enough water to cover the bottom.
- Magnetic stirrer, unless a self-stirring probe is used. Consult the manufacturer's instructions.
- Aquarium-type air pump, tubing, and bubbler.

NOTE: Please refer to the Master List of Documents and Software located on G:\QA\READ\Master List of Documents for the current software to be used for data processing.

6.2 Supplies

- BOD incubation bottles, 300 mL capacity, with ground glass stoppers and plastic caps. Disposable BOD bottles are purchased and are to be used one time only. If disposable bottles are not currently available, then glass BOD bottles will be used.
- Collapsible plastic bottles or carboys.
- Assorted laboratory glassware and apparatus such as pipettes, graduated cylinders, volumetric flasks, beakers, etc. All glassware must be scrupulously cleaned to remove traces of organic contaminants. For this reason, avoid using detergent to clean BOD bottles. Glass BOD bottles must be thoroughly cleaned with 10% HCl, rinsed with deionized water, and drained after cleaning.

7.0 Reagents and Standards

7.1 Reagent Water: Reagent water must be free of organics. Use Milli-Q water, distilled water, or other high purity water as available.

7.2 Chlorine Test Kit: Hach kit.

7.3 0.1N Sodium Hydroxide: Dissolve 0.4 g of sodium hydroxide in water, cool, and dilute to 100 mL.

7.4 **1.0N Sulfuric Acid:** Carefully add 2.8 mL of concentrated sulfuric acid to approximately 80 mL of deionized water, cool, and dilute to 100 mL with deionized water. This solution is usually purchased from Fisher.

7.5 **50% Glacial Acetic Acid:** Carefully add 10 mL of glacial acetic acid to 10 mL of deionized water.

7.6 **10% Potassium Iodide:** In a 100-mL volumetric flask, dissolve 10 g of potassium iodide (KI) in approximately 70mL of deionized water. Dilute to volume with deionized water.

7.7 **1% Starch Indicator Solution:** This reagent is available from commercial sources.

7.8 **Sodium Sulfite Solution:** Dissolve 0.16 g of sodium sulfite in deionized water and dilute to 100 mL with deionized water. This solution is unstable and must be prepared daily as needed. This solution may also be obtained from a commercial vendor.

7.9 **0.025 N Sodium Thiosulfate:** Dissolve 0.6205 g of sodium thiosulfate pentahydrate in deionized water and dilute to 100 mL with deionized water. This solution is unstable and must be prepared daily as needed. This solution may also be obtained from a commercial vendor.

7.10 **Dilution Water:** Add the contents of one HACH BOD Nutrient Buffer pillow to 3.0 liters of reagent water and aerate. It is recommended that dilution water older than 24 hours be used. Fresh dilution water may be used for analysis if necessary, but the dilution water blanks may be high due to small amounts of organic contamination in the reagents used. The dilution water is the first solution measured when setting up every BOD batch, and the quality check is explained in Section 10.3.3. The dilution water used in conjunction with this SOP is made daily.

7.11 **Seed (available commercially):** Add the contents of one Polyseed capsule to 500 mL of aerated dilution water and stir for one hour. Prepare fresh every day. Do NOT stir for longer than one hour. Always pre-test a new container of seed before use. This is done by adding the contents of one capsule to 500 mL of dilution water, and using the same aliquots per bottle, test the new seed along with the current seed as explained in Section 10.3.4. Increase or decrease the amount of dilution water if the new seed does not fall in the required range.

NOTE: Special projects may require the use of seed organisms adapted to the project waste stream.

7.12 **Nitrification Inhibitor (available commercially):** Dissolve 100 mg of 2-chloro-6-(trichloromethyl)pyridine in 100 mL of dilution water. Prepare fresh daily only if needed. Available commercially from HACH. Use two shots or 0.16 g per 300 mL bottle. This reagent is used for CBOD testing only.

7.13 **BOD LCS Solution (dextrose/glutamic acid):** Dry reagent grade dextrose and glutamic acid in a drying oven at 103 ± 5 oC. Cool in dessicator and store in a sealed container. In a 1-L volumetric flask, dissolve 150 mg of dextrose and 150

mg of glutamic acid in approximately 500 mL of deionized water and dilute to volume. This solution must be prepared fresh daily. Alternatively, a commercially-prepared dextrose/glutamic acid reagent may be purchased (packaged in ampules) from HACH or ERA. Dilute the reagents according to the manufacturer's instructions.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass or Plastic	1 liter	Cool, 4°C /None	48 Hours	40 CFR Part 136.3

NOTE: The laboratory follows the 48 hour hold time as determined in the Standard Methods 20th edition QC section and 40 CFR Part 136.3.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.2 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.3 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.4 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.5 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.6 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)	3 in 20 or fewer samples	198 ± 30.5 mg/L (85 to 115%)
Sample Duplicate[1]	1 in 20 or fewer samples	≤ 20% RPD
Dilution Water Blank	3 in 20 or fewer samples	< 0.2 mg/L of DO
Seed Control Samples	4 in 20 or fewer samples	The seed correction factor needs to be between 0.6 and 1.0 mg/L

[1]The sample selection for the sample duplicate is randomly selected, unless specifically requested by a client....predetermined by the extraction lab.

9.7 Instrument QC

9.7.1 Some regulatory programs require that the meter be calibrated versus the Winkler titration. See Attachment 7.

9.7.2 Check the probe calibration as indicated on the bench sheet. The first probe check is done at the start of the test, before any samples, including the blank and the LCS, have been read. Subsequent probe checks are done after every 9 readings and at the end of the run.

Acceptance Criteria: Probe calibration checks must be within 2% of the expected value. Allow sufficient time for stabilization

Corrective Action: Adjust calibration if necessary and repeat all readings following the last acceptable probe check. If the probe will not calibrate properly, it may be necessary to change the membrane. Consult the manufacturer's instructions.

9.7.3 The probe is usually calibrated in water-saturated air, but can also be water calibrated. The procedure for this depends on the model of dissolved oxygen meter being used. Attachment 1 and Attachment 2 give the instructions for probe calibration of the Orion Dissolved Oxygen Probe and YSI Model 5100 Probe, respectively.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any unauthorized deviations from this procedure must also be documents as a nonconformance, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 Precautions:

10.1.1.1 All additions of sample, reagents, dilution water, and seed must be made in such a way as to avoid entrapment of air.

10.1.1.2 Air bubbles must be excluded from the BOD bottles when making dissolved oxygen readings and again when placing stoppers on the bottles. Tapping on the side of the bottle can help in freeing any trapped air bubbles.

10.1.1.3 Dissolved oxygen readings must be taken IMMEDIATELY after making a dilution. Do not prepare several samples and then measure the dissolved oxygen. Dilute and measure each sample individually.

10.1.1.4 A water seal must be formed on top of the stoppers to prevent evaporation and entry of air. A plastic cap is used to prevent evaporation of the water seal.

10.1.1.5 Due to possible contamination contributions the use of soap is **prohibited** in the washing of the dilution water reservoirs. Reservoirs should only be cleaned with repeated rinses of DI water. A brush can be used to remove algae.

10.2 Calibration

10.2.1 Some regulatory programs require that the meter be calibrated versus the Winkler titration. See Attachment 7.

10.2.2 Check the probe calibration as indicated on the bench sheet. The first probe check is done at the start of the test, before any samples, including the blank and the LCS, have been read. Subsequent probe checks are done after every 9 readings and at the end of the run.

Acceptance Criteria: Probe calibration checks must be within 2% of the expected value. Allow sufficient time for stabilization.

Corrective Action: Adjust calibration if necessary and repeat all readings following the last acceptable probe check. If the probe will not calibrate properly, it may be necessary to change the membrane. Consult the manufacturer's instructions.

10.2.3 The probe is usually calibrated in water-saturated air, but can also be water calibrated. The procedure for this depends on the model of dissolved oxygen meter being used. Attachment 1 and Attachment 2 give the instructions for probe calibration of the Orion Dissolved Oxygen Probe and YSI Model 5100 Probe, respectively.

10.3 Sample Analysis

10.3.1 Measure the pH of each sample with a pH meter and record the value. If necessary, adjust the pH of an aliquot (approximately 500 mL) of each sample to between 6.5 and 7.5 by drop-wise addition of 0.1 N sodium

hydroxide or 1 N sulfuric acid, as appropriate. Do not use more than 1% of total sample volume of NaOH or H2SO4 to adjust pH.

10.3.2 Check for residual chlorine using the chlorine test kit.

10.3.2.1 Pour out approximately 10 mL of sample into a small container and add the chlorine test kit reagent.

10.3.2.2 If the solution turns pink, the sample contains residual chlorine and must be dechlorinated.

10.3.2.3 Remove residual chlorine by adding either sodium sulfite solution or sodium thiosulfate solution. The addition of sodium sulfite is done drop wise while stirring until the sample no longer tests positive for chlorine.

10.3.2.4 Recheck the pH, as sodium sulfite addition will increase the pH. Record the results of the test (positive or negative) on the bench sheet. See Attachment 5 Chlorine Neutralization Chart.

10.3.3 Dilution Water Check Samples (3 required).

10.3.3.1 Fill a BOD bottle with dilution water to just inside the neck.

10.3.3.2 Immediately measure and record the initial dissolved oxygen (DO) reading and the bottle number.

10.3.3.3 The initial DO reading of the dilution water should be between 6.5 and 9.0 mg/L depending on the elevation. It should not exceed 9.0 mg/L. If the dilution water is not within this range, allow the water to reach room temperature in a partially filled container and shake vigorously or aerate. Proper aeration is usually achieved with an aquarium air pump in 45 minutes to 1 hour.

10.3.3.4 Stopper the bottle, taking care to exclude all air bubbles. Be sure that there is a small amount of excess water on top of the stopper to form a water seal.

10.3.3.5 Cover the stopper with a plastic cap to prevent evaporation of the water seal and incubate with the samples.

10.3.4 Seed Controls

10.3.4.1 Be sure the seed is well mixed. Aliquot 15, 20, 25, and 30 mL of seed into 4 BOD bottles and fill to just inside the neck with dilution water.

10.3.4.2 Immediately measure and record the initial dissolved oxygen reading and the bottle number.

10.3.4.3 Stopper the bottle and form a water seal. Cover with a cap and incubate with the samples.

10.3.5 Method Blank (MB)

10.3.5.1 Aliquot 4.0 mL of seed into a BOD bottle and fill to just inside the neck with dilution water (approximately 300 mL of dilution water).

10.3.5.2 For a CBOD method blank, aliquot 4.0 mL of seed into a BOD bottle, add 0.16 g or two shots of nitrification inhibitor, and fill to just inside the neck with dilution water.

10.3.5.3 Immediately measure and record the initial dissolved oxygen reading and the bottle number.

10.3.5.4 Stopper the bottle and form a water seal. Cover with a cap and incubate with the samples.

10.3.6 Laboratory Control Sample (LCS)

10.3.6.1 Add 4.0 mL of well-mixed seed into each of 3 BOD bottles.

Note: If CBOD is required add 2 shots or 0.16 gram of the nitrification inhibitor to the three LCSs.

10.3.6.2 Pipette 6 mL of LCS (7.13)standard solution and dilute to just inside the neck with dilution water.

10.3.6.3 Immediately measure and record the initial dissolved oxygen reading and the bottle number.

10.3.6.4 Stopper the bottle and form a water seal. Cover with a cap and incubate with the samples.

10.3.6.5 The final concentration of the LCS should be 198 mg/L +/- 30 mg/L. Be careful not to contaminate the stock solution with seed or any other material.

10.3.7 For each sample, pipette 4.0 mL of seed into a BOD bottle. If nitrification inhibitor is required (CBOD), add 2 shots or 0.16 gram of the nitrification inhibitor and note the addition on the bench sheet and anomaly sheet.

10.3.8 Add the sample aliquot (see below), and dilute to just inside the neck with dilution water.

10.3.9 Immediately record the initial dissolved oxygen reading, the sample volume used and the bottle number.

10.3.10 The initial DO reading must be less than 9.0 mg/L and greater than 6.5 mg/L. If it is not, dilute the sample until a suitable reading is obtained. Do not aerate samples unless requested by client, as this could cause loss of volatiles.

10.3.11 Stopper the bottle and form a water seal. Cover with a plastic cap and incubate for 5 days.

10.3.12 If historical data are not available, use the following guidelines for determining sample dilutions:

- 0.0 to 1.0% for strong industrial wastes,
- to 5% for raw and settled wastewater,

- 5 to 25% for biologically treated effluent, and
- 25 to 100% for polluted river waters.

NOTE: If historical data is not available a minimum of five aliquots **must** be set.

10.3.13 A COD screen may also be performed to assist in determining the appropriate dilution for BOD analysis when historical data are variable or unavailable. The COD screen is performed using the reagents and general procedures presented in SOP DV-WC-0018. For screening purposes, it is not necessary to comply with all the QC requirements stipulated in the SOP, as the results will not be reported.

10.3.13.1 The approximate highest value of BOD that could be expected in the sample is estimated by multiplying the COD screening result by 0.6.

10.3.13.2 Using the table below, determine which dilution would be appropriate for the estimated highest BOD value so as to choose the most concentrated aliquot that will be analyzed.

10.3.13.3 Then choose several more dilute aliquots (4 to 6 depending on how much historical data are available for the sample) in a geometric progression so that the most likely BOD values are covered.

Aliquot (mL)	Approximate BOD Range (mg/L)
240	2 - 6
120	5 - 13
60	10 - 25
25	24 - 60
10	60 - 150
5	120 - 300

NOTE: Color and/or odor may be used as a guide in determining which dilutions to make. Raw wastes and highly polluted waters may require an initial dilution (see next paragraph).

10.3.13.4 Do not take aliquots less than 1 mL. If a sample requires larger dilutions, perform an initial 5X (or higher) dilution with dilution water, then take the required aliquots. The range will be extended by the initial dilution factor.

10.3.14 Check the probe calibration every 9 samples as indicated on the bench sheet and at the end of the batch.

10.3.15 Place BOD bottles into the incubator and incubate samples for for 5 days ± 2 hours. Label the first bottle in the batch with the date and approximate time that the batch is to be read back.

10.3.15.1 If during the 5-day incubation period, the temperature falls outside of the acceptable range, i.e., 20 ± 1 °C, record the temperature excursion on an NCM.

10.3.16 Final Dissolved Oxygen Readings

10.3.16.1 After 5 days (± 2 hours), remove the BOD bottles from the incubator.

10.3.16.2 Calibrate the probe and measure and record the final dissolved oxygen in each bottle. Check the probe calibration as indicated on the bench sheet(every 9 samples and at the end of the run)

10.3.17 Clean all glassware, apparatus, and the work area.

11.0 Calculations / Data Reduction

11.1 Dilution Water Samples:

$$DOU = DO1 - DO2$$

Where:

DOU = Dissolved oxygen uptake, mg/L
DO1 = Initial dissolved oxygen, mg/L
DO2 = Final dissolved oxygen, mg/L

11.2 Seed Correction: (depletion must be 40 – 70% to be acceptable)

$$SEED = DOUSEED \times F$$

Where:

SEED = Seed correction, mg/L (must be between 0.6 and 1.0 mg/L)
DOUSEED = DO1 - DO2

Where:

DO1 = Initial dissolved oxygen, mg/L and
DO2 = Final dissolved oxygen, mg/L (should read ≥ 1.0 mg/L)

$$F = \frac{\%\ \text{seed in each BOD sample}}{\%\ \text{seed in control bottle}}$$

NOTE: Typically 4 mL of seed is used, which makes the numerator above = 1.33%.

11.3 Samples and LCS

11.3.1 Reject all sample aliquots with DO2 < 1 mg/L.

11.3.2 Reject all sample aliquots with (DO1 - DO2) < 2 mg/L.

11.3.3 For all others, calculate BOD as follows:BOD = (DO1 - DO2 - SEED) x (300/V)

Where: BOD = BOD for sample aliquot, mg/L.
DO1 = Initial dissolved oxygen, mg/L.
DO2 = Final dissolved oxygen, mg/L.
SEED = Seed correction calculated above (Section 11.2), mg/L.
V = Sample aliquot volume, mL.

11.3.4 Calculate and report the average BOD for all sample aliquots which meet the two criteria above, unless there is evidence of toxicity or another anomaly. Consult the group leader or senior analyst in such a case.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy DV-QA-005P. An MDL verification is performed once a year to satisfy NELAC 2003 requirement. For DoD, AFCEE, and DOE projects, an MDL verification is performed quarterly. MDLs are stored in the LIMS.

12.2 Initial Demonstration of Capability

An initial demonstration of capability for each method must be performed prior to analyzing samples.

12.2.1 For the standard analyte list, the initial demonstration consists of the preparation and analysis of a QC check sample (e.g., LCS) containing all of the standard analytes for the method, as well as a method detection limit (MDL) study.

12.2.2 Four aliquots of the QC check sample are analyzed with the same procedures used to analyze samples, including sample preparation.

12.2.3 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria (e.g., LCS control limits). All four results must meet acceptance criteria before the method can be used to analyze samples.

12.2.4 For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is successful analysis of an extracted standard at the reporting limit and a single point calibration.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and

has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Treated Sample Waste – Neutral - Waste Stream W

14.2.2 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Method 405.1, "Biochemical Oxygen Demand", USEPA, Editorial Revision, 1974.

15.2 Method 5210B, "5-Day BOD Test", Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

15.3 Related SOPs

15.3.1 DV-WC-0018, Chemical Oxygen Demand

15.3.2 DV-WC-0006, Carbon In Water (TOC, TIC, DOC, and TC)

16.0 Method Modifications:

Item	Method	Modification
1	EPA 405.1: Section 3.1	The probe method is used for the dissolved oxygen measurements. It is NOT calibrated versus the Winkler method unless otherwise stated on the bench sheet.
2	SM 5210B: Section 4E.2	The reference method calls for residual chlorine to be determined by titration on one portion of the sample and a second portion treated for chlorine removal. This is not done since there is usually not enough sample. Instead, an aliquot of the sample is treated until it gives a negative test for chlorine.
3	SM 5210B: Section 4A	A pre-packaged nutrient buffer is used in place of the four individual solutions specified in the method. The final dilution water has the same composition.
4	SM 5210B: Section 4D.1	Pre-packaged lyophilized seed is used since it is not always possible to obtain seed from the waste treatment plant on evenings and weekends. This is an acceptable option in Method 5210B. The proportion of seed used is in accordance with the manufacturer's instructions.
5	SM 5210B: Secttion 6B	Due to the altitude of the laboratory facility, the acceptable initial DO level has been lowered to 6.5 from the recommended 7.0.

17.0 Attachments

Attachment 1: Instructions for Orion Dissolved Oxygen Probe

Attachment 2: Instruction for YSI Model 5100 Dissolved Oxygen Meter

Attachment 3: Example BOD Cover Sheet (i.e., data review checklist)

Attachment 4: Flow Chart

Attachment 5: Pre-treatment for the Presence of Chlorine

Attachment 6: Winkler DO Method and Sodium Thiosulfate Standardization Procedure

Attachment 7: Temperature – Dissolved Oxygen Calibration Values Table (5340ft altitude)

Attachment 8: Calibration Values for Various Atmospheric Pressures and Altitudes

Attachment 9: YSI Model 5100 Barometer Calibration Instructions

18.0 Revision History

- Revision 8 dated 30 December 2010
  - Added Attachments 7, 8 and 9.
  - Deleted Attachment 3

Revision 7 dated 02 October 2009

  - Added note to section 6.1 – Master List of Documents
  - Deleted the sentence concerning the washing of glass bottles in section 6.2.
  - Modified sections 10.3.3.3 and 10.3.10 to have the minimum DO reading 6.5.
  - Added Method Modification #5 to section 16.0.
  - Updated Attachment 4 to current checklist.
- Revision 6.1 dated 27 May 2009
  - Updated minor grammatical and formatting errors.
  - Added note concerning hold time to section 8.
  - Updated section 10.3.2 to match current practices.
  - Added note to section 10.3.12 concerning the use of 5 aliquots for samples without historical data.
  - Updated Attachment 4.

- Revision 6 dated 27 May 2008
  - Revised formatting throughout the SOP to be consistent with the new corporate guidance.
  - Added section 4.11 –prohibition of soap.
  - Additional information added to the section 5.
  - Added section 10.1.15 – prohibition of soap.
  - Added note in section 10.3.6 concerning nitrification inhibitor added to LCSs.

- Revision 5 dated 30 November 2007
  - Integration for TestAmerica and STL operations.
  - Revised formatting throughout the SOP to be consistent with the new corporate guidance.
  - Changed 10.2.2 to reflect check done at end of run.

- Revision 4 dated 28 June 2006
  - Revised formatting throughout the SOP to be consistent with the guidance in Policy QA-001.
  - Section 5, Safety, was revised to meet current STL requirements.
  - Added pH meter to Section 6 and deleted pH paper from Section 7.
  - Incorporated Interim Change to add the chlorine test kit to Section 7.
  - Revised Section 13, Pollution Prevention, to meet current STL requirements.
  - Added Section 15, Waste Management, to meet current STL requirements.

- Revision 3 dated 16 August 2002
  - References to Quanterra were changed to STL.
  - Reference to the Chemical Hygiene Plan was changed to the Corporate Safety Manual.
  - Reagents 7.6, 7.7, 7.8 added for the residual chlorine removal
  - Section 6.3 was changed to require two temperature checks daily for the incubator.

- Section 7.10 was changed so that the glucose/glutamic acid is prepared fresh daily.
- Sections 7.6, 7.7 and 7.8 were added as the reagents for residual chlorine removal procedure.
- Section 11.4.3 was updated to add titration to the residual chlorine removal procedure.
- Sections 11.5.1.3 and 11.5.4 were changed so that the initial DO requirement is 7.0 to 9.0 mg/L.
- Waste management section updated.

Attachment 1.

Instructions for Orion Dissolved Oxygen Probe

NOTE: The overflow funnel must be substantially modified in order to work with 60 mL BOD bottles. Be sure that it forms a tight fit in the neck of the bottle.

NOTE: Do not allow the membrane to become scratched or damaged in any way, and do not touch it with your fingers.

1. Calibration

- Connect the probe to the meter. Turn the meter to pH mode and the probe to OFF. Use the meter calibration knob to adjust the reading to 7.00.
- Turn the probe switch to BATT. The reading should be greater than 13 or off-scale. If not, the batteries need to be replaced.
- Turn the probe to ZERO. Use the calibration knob on the left to adjust the reading to 0.00.
- Insert the probe into a BOD bottle containing enough water to cover the bottom of the bottle. **THE PROBE TIP MUST NOT BE IMMERSED IN WATER OR HAVE WATER DROPLETS CLINGING TO THE MEMBRANE.** Gently touch the corner of a Kim-wipe to any water drops to very carefully dry the membrane if necessary. This bottle is also used for storing the probe.
- Turn the probe to AIR, allow to stabilize and use the calibration knob on the right to adjust the reading to the proper calibration value. This value depends on altitude and at TestAmerica Denver, it is 6.28. The value can be obtained from the table in the manufacturer's instruction book. Record this value on the bench sheet.
- Turn the probe switch to H2O for measurement of samples.

2. Measurement of Dissolved Oxygen

- Be sure that the probe has been set to H2O.
- Place the sample to be measured on a magnetic stirrer. Be sure that the stirrer is rotating.
- Wait for the reading to stabilize, then record it.
- Rinse the probe tip with a stream of deionized water or dilution water between measurements.
- Store the probe in a BOD bottle with a small amount of water in the bottom and turn it off when not in use. Do not store the probe with the tip completely immersed in water.

Attachment 1.

Instructions for Orion Dissolved Oxygen Probe (Continued)

3. **Calibration Check**

- Carefully blot and dry off the tip of the probe by touching the corner of a Kim-wipe to any water droplets. Do not damage or touch the membrane with your fingers.
- Place the probe in a BOD bottle containing a small amount of water in the bottom. Do not immerse the membrane.
- Switch the probe to the AIR position and observe the reading.
- When the reading stabilizes, it should be between 6.15 and 6.41.
- Return the probe to H2O before continuing with any more dissolved oxygen measurements.etc....

Attachment 2.

Instruction for YSI Model 5100 Dissolved Oxygen Meter

NOTE: Do not allow the probe to become scratched or damaged in any way, and don't touch it with your fingers.

1. **Calibration**

- Place the probe in a BOD bottle with a small amount of water in the bottom.
- Do not immerse the membrane.
- Turn the function switch to TEMP oC. A tone will sound followed several seconds later by a second tone.
- Allow the meter to warm up for 15 minutes. The warm-up time may be eliminated if the probe is not turned off after use.
- Record the temperature of the probe on the bench sheet.
- Determine the calibration value for the probe temperature from the table below (these values have already been corrected for TestAmerica-Denver's altitude; consult the manufacturer's instruction manual for corrections at other altitudes/pressures). Record this value on the bench sheet.
- Set the switch to mg/L CAL and use the keypads beneath the display to set the calibration value obtained in step 1.5.
- Turn the switch to mg/L. CAL will appear in the display, followed in a few seconds by a tone. The reading will then be displayed; it should match the calibration value.
- Observe the reading for 2 to 3 minutes. If it drifts by more than 0.02 units, insufficient warm-up was allowed. Repeat steps 1.4 through 1.7, if necessary. Otherwise, the probe is now calibrated and ready for use.

2. **Measurement of Dissolved Oxygen**

- Place the probe into the sample to be measured and turn on the stirrer motor.
- Record the reading when stable.
- Turn off the stirrer and remove the probe from the bottle.
- Rinse the probe with a stream of water after each measurement.
- Store the probe in a BOD bottle containing a small amount of water. Do not immerse the membrane.

Attachment 2.

Instruction for YSI Model 50 Dissolved Oxygen Meter (continued)

3. **Calibration Check**

- Place the probe in a BOD bottle with a small amount of water in the bottom.
- Record the reading when stable.
- Switch back to TEMP oC and record the temperature.
- Look up the calibration value for this temperature from the table.
- The reading should match the calibration value within 2%.
- Switch back to mg/L and continue with readings.

YSI Model 50 Dissolved Oxygen Meter
Calibration Values (for TestAmerica Denver altitude)

Temperature (°C)	Calibration Value		Temperature (°C)	Calibration Value
15	8.28		23	7.05
16	8.11		24	6.92
17	7.94		25	6.79
18	7.78		26	6.70
19	7.62		27	6.32
20	7.47		28	6.43
21	7.33		29	6.32
22	7.18		30	6.21

Attachment 3.

Example BOD Data Review Checklist

TestAmerica Denver

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

BOD Data Review Checklist

Methods: EPA 405.1, Standard Methods 5210 B

SOP # DV-WC-0019

Run Date:_____ **Analyst:**_____ **Instrument:**_____

Log-in / Sample Numbers	Batch	Method	Special Inst

A. Materials and Instrument Checks	Yes	No	N/A	2nd Level
1. Seed water stirred/aerated for at least one hour?				
2. Incubator temperature in control (20 ± 1°C) for 5-day incubation?				
3. Probe checks done at prescribed intervals as shown on the benchsheet?				
4. If CBOD in batch, is there a Blank, Duplicate for CBOD in the batch?				
B. Method Required QC				
1. Initial D.O. readings for samples, blanks and GGA standard between 7.0 and 9.0 mg/L?				
2. Seed control depletion is at least 2.0 mg/L (40 – 70%)?				
3. Seed correction value between 0.6 and 1.0 mg/L?				
4. Glucose/Glutamic acid (LCS) within control limits?				
C. Sample Results				
1. 48 hour holding time met for all samples?				
2. Was sample pH between 6.5 and 7.5 (adjustment made if necessary)?				
3. Were samples checked for residual chlorine (removed if necessary)?				
4. Are all sample dilutions with depletion <2.0 mg/L, or final D.O. <1.0 mg/L rejected?				
5. Do reporting limits reflect dilutions and/or limited sample volume?				
6. Were there any signs of toxicity in the samples?				
7. Were there any air bubbles in the BOD bottles?				
8. Were special client requirements met?				
9. STD/True Value information is updated and included?				

Analyst: _____ **Date:** _____

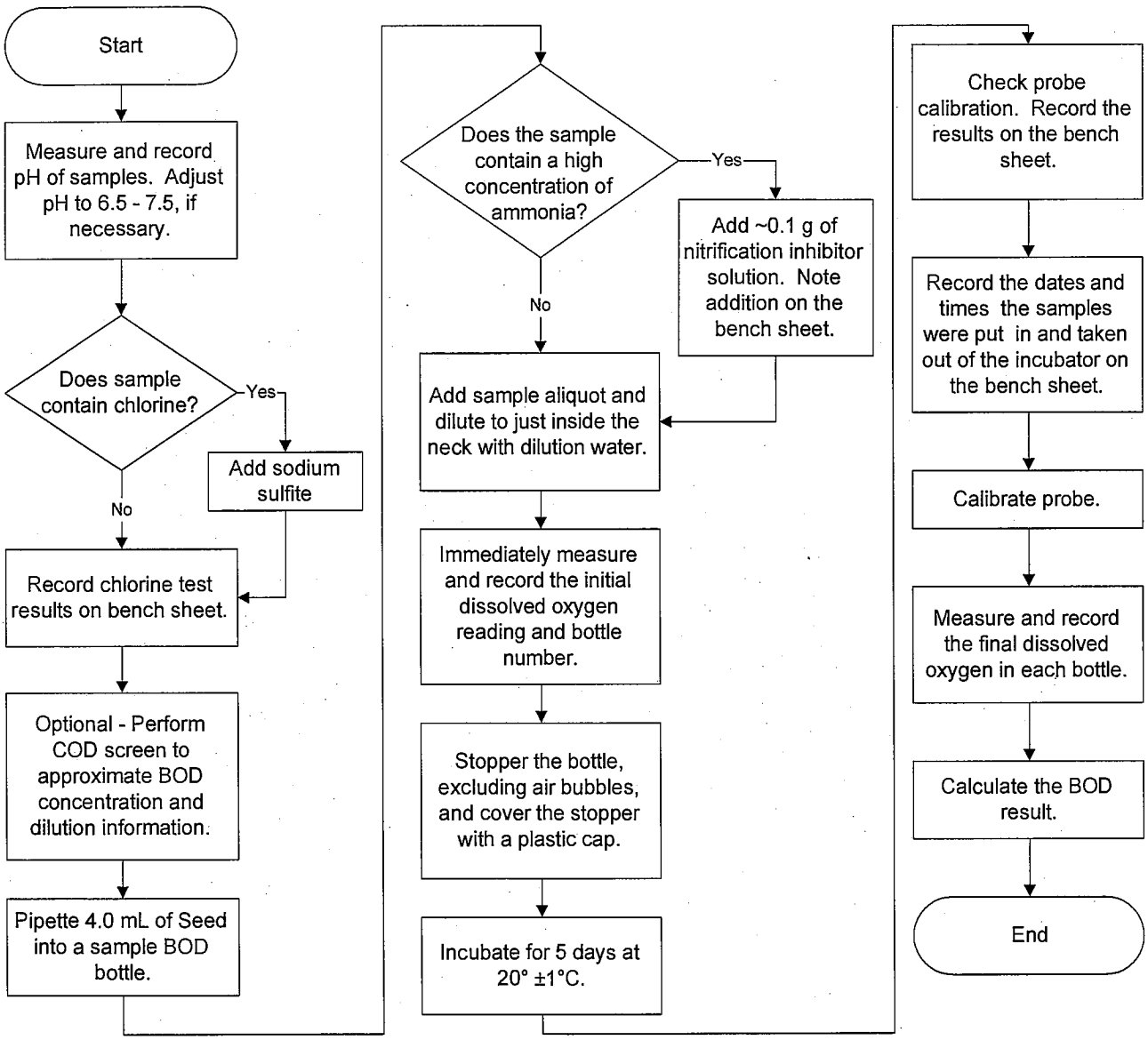
_____

_____

2nd Level Reviewer : _____ **Date:** _____

Attachment 4.

Flow Chart



Attachment 5.

Chlorine Neutralization Chart[1]

Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)
0.05	0.10	2.10	4.20	4.15	8.40	6.20	12.5
0.10	0.20	2.15	4.30	4.20	8.50	6.25	12.6
0.15	0.30	2.20	4.40	4.25	8.60	6.30	12.7
0.20	0.40	2.25	4.50	4.30	8.70	6.35	12.8
0.25	0.50	2.30	4.60	4.35	8.80	6.40	12.9
0.30	0.60	2.35	4.70	4.40	**8.90**	**6.45**	13.1
0.35	0.70	2.40	4.80	4.45	9.00	6.50	13.2
0.40	0.80	2.45	4.90	4.50	9.10	6.55	13.3
0.45	0.90	2.50	5.00	4.55	9.20	6.60	13.4
0.50	1.00	2.55	5.10	4.60	9.30	6.65	13.5
0.55	1.10	2.60	5.20	4.65	9.40	6.70	13.6
0.60	1.20	2.65	5.30	4.70	9.50	6.75	13.7
0.65	1.30	2.70	5.40	4.75	9.60	6.80	13.8
0.70	1.40	2.75	5.50	4.80	9.70	6.85	13.9
0.75	1.50	2.80	5.60	4.85	9.80	6.90	14.0
0.80	1.60	2.85	5.70	4.90	9.90	6.95	14.1
0.85	1.70	2.90	5.80	4.95	10.0	7.00	14.2
0.90	1.80	2.95	5.90	5.00	10.1	7.05	14.3
0.95	1.90	3.00	6.00	5.05	10.2	7.10	14.4
1.00	2.00	3.05	6.10	5.10	10.3	7.15	14.5
1.05	2.10	3.10	6.20	5.15	10.4	7.20	14.6
1.10	2.20	3.15	6.30	5.20	10.5	7.25	14.7
1.15	2.30	3.20	6.40	5.25	10.6	7.30	14.8
1.20	2.40	**3.25**	**6.60**	5.30	10.7	7.35	14.9
1.25	2.50	3.30	6.70	5.35	10.8	7.40	15.0
1.30	2.60	3.35	6.80	5.40	10.9	7.45	15.1
1.35	2.70	3.40	6.90	5.45	11.0	7.50	15.2
1.40	2.80	3.45	7.00	5.50	11.1	7.55	15.3
1.45	2.90	3.50	7.10	5.55	11.2	7.60	15.4
1.50	3.00	3.55	7.20	5.60	11.3	7.65	15.5
1.55	3.10	3.60	7.30	5.65	11.4	7.70	15.6
1.60	3.20	3.65	7.40	5.70	11.5	7.75	15.7
1.65	3.30	3.70	7.50	5.75	11.6	7.80	15.8
1.70	3.40	3.75	7.60	5.80	11.7	7.85	15.9
1.75	3.50	3.80	7.70	5.85	11.8	7.90	16.0

Attachment 5.

Chlorine Neutralization Chart[1] (continued)

Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)	Chlorine (mg/L)	Na_2SO_3 (mL of 0.025N to be added)
1.80	3.60	3.85	7.80	5.90	11.9	7.95	16.1
1.85	3.70	3.90	7.90	5.95	12.0	8.00	16.2
1.90	3.80	3.95	8.00	6.00	12.1	8.05	16.3
1.95	3.90	4.00	8.10	6.05	12.2	8.10	16.4
2.00	4.00	4.05	8.20	6.10	12.3	8.15	16.5
2.05	4.10	4.10	8.30	6.15	12.4	8.20	16.6
8.25	16.7	8.55	17.3	8.85	17.9	9.15	18.5
8.30	16.8	8.60	17.4	8.90	18.0	9.20	18.6
8.35	16.9	8.65	17.5	8.95	18.1	9.25	18.7
8.40	17.0	8.70	17.6	9.00	18.2	9.30	18.8
8.45	17.1	8.75	17.7	9.05	18.3	9.35	18.9
8.50	17.2	8.80	17.8	9.10	18.4	9.40	19.0

[1] The concentration of residual chlorine present must first be determined, refer to section 10.3.2. This chart shows the amount of 0.025 *N* sodium sulfite to be added to a 2-liter sample aliquot with the applicable concentration of residual chlorine. The amount of sodium sulfite that is added to the sample aliquot must be adjusted, unless a 2-liter aliquot is used. To determine the amount of sodium sulfite to be added to the sample aliquot, divide 2-liters by the volume of sample (in liters) being adjusted, then divide that factor into the amount of sodium sulfite to be added listed in the chart.

It is important that this chart is followed as closely as possible to avoid over de-chlorination. Excess amounts of sodium sulfite can add a positive bias to the final BOD result. Also, incomplete de-chlorination can lead to low BOD results since chlorine can kill seed organisms thus reducing the amount of oxygen depletion of the sample during incubation.

Do not add more than 1% of total volume of 0.025*N* Na_2SO_3. If a greater volume of Na_2SO_3 is required to de-chlorinate the sample, use a higher concentration of Na_2SO_3.

Attachment 6.

Winkler DO Method and Sodium Thiosulfate Standardization Procedure

Winkler DO Method

This method is used to measure the DO content of the dilution water.

1. To the BOD bottle, add 2 mL of manganous sulfate solution by holding the tip of the pipet just above the surface of the water.
2. Add 2 mL of alkaline-iodine-azide reagent by placing the tip of the pipet just under the surface of the water.
3. **IMMEDIATELY** re-stopper the bottle so that no air bubbles are trapped inside.
4. Mix gently by inverting the bottle fifteen times to form a manganese hydroxide flock that is brown and cloudy.

NOTE: If no DO is present in the bottle, the flock will appear as a white, cloudy substance. **REGARDLESS OF WHAT COLOR APPEARS, CONTINUE TO THE ACID ADDITION IN STEP #5 BELOW.**

5. Allow the flock to settle until a clear supernatant appears in at least half of the bottle.
6. Re-mix as in step #4 above, and allow to settle again.
7. Remove the stopper and add 2 mL of concentrated sulfuric acid by holding the tip of the pipet just **above** the surface of the water.
8. Re-stopper the bottle so no air bubbles are trapped inside.
9. Mix gently by inverting the bottle fifteen times.

NOTE: If this produces a clear, colorless solution in the bottle, **the DO must be reported as <1 mg/L.**

10. After the acid is added, do not delay more than 45 minutes to perform the titration.
11. If color is present in the bottle, transfer the entire contents of the bottle to a 500 mL Erlenmeyer flask.
12. While stirring, titrate using a buret filled with 0.037*N* sodium thiosulfate standard titrant until a pale, straw color remains.
13. Continue stirring, and add 2 mL of starch indicator solution, which will turn the water, a blue color. Continue stirring and titrating slowly until a clear end point is achieved.

NOTE: DISREGARD ANY RETURN OF THE BLUE COLOR.

14. Record the volume of the titrant used.
15. If the normality of the sodium thiosulfate standard titrant is within the specified range as determined in the Normality Standardization Procedure, the 1 mL of titrant is equal to 1 mg/L of DO.
16. If not, apply the factor to determine the DO.

Attachment 6.

Winkler DO Method and Sodium Thiosulfate Standardization Procedure (continued)

Sodium Thiosulfate Standardization Procedure

This method is used to determine the normality of the sodium thiosulfate titrant used in the Winkler DO procedure.

1. Add 150 mL of distilled water to a 350 mL Erlenmeyer flask.
2. Add approximately 2 grams of KI crystals and mix until dissolved.
3. Add 10 mL of 10% sulfuric acid solution.
4. Add 20 mL of 0.0375*N* potassium bi-iodate standard and mix.
5. Place in the dark for 5 minutes.
6. Remove from the dark, dilute to 300 mL with distilled water, and mix.
7. While stirring the standard, fill a buret with the 0.0375*N* sodium thiosulfate standard titrant and titrate to a pale straw color.
8. Continue stirring, and add 2 mL of starch indicator solution.
9. Continue to titrate until a clear end point is reached and disregard the return of any blue color.
10. Record the amount of titrant used and calculate the normality of the sodium thiosulfate standard titrant using the following formula:

$$\textbf{Volume}_1 \textbf{ x Normality}_1 = \textbf{Volume}_2 \textbf{ x Normality}_2$$

If the normalities are not equal, a 1-to-1 relationship is not present and 1 mL of titrant will not be equal to 1 mg of DO in the sample. A correction factor must be determined by dividing the calculated sodium thiosulfate normality by the known bi-iodate normality. Multiply this factor by the volume of titrant used in each Winkler DO to determine the amount of DO in each sample.

The following is an example calculation:

- For this example, the normality of potassium bi-iodate is 0.0375*N*, 20 mL of potassium bi-iodate was used, and 18.6 mL of sodium thiosulfate titrant was used.

$$V_1N_1 = V_2N_2$$
$$20.0 \text{ mL} \times 0.0375 \text{ mequiv/mL} = 18.6 \text{ mL} \times N_2$$
$$N_2 = 0.0403 \text{ mequiv/mL}^*$$

***NOTE:** THAT THE FINAL NORMALITY WAS ROUNDED OFF TO THE SAME NUMBER OF DECIMAL PLACES AND SIGNIFICANT FIGURES AS THE KNOWN NORMALITY OF THE POTASSIUM BI-IODATE.

Attachment 6.

Winkler DO Method and Sodium Thiosulfate Standardization Procedure (continued)

- Next, divide the sodium thiosulfate normality by the bi-iodate normality to determine the factor as follows: 0.0403/0.0375 = 1.07.

- This factor (1.07) will be multiplied by all Winkler DO titrations to determine the DO in each sample that is titrated.

11. Complete the normality procedure on a duplicate sample, which should agree within ± 0.05 mL of the first test.

12. Keep a written record of this procedure.

Attachment 7.

Temperature – Dissolved Oxygen Calibration Values Table (5340ft altitude)

YSI Dissolved Oxygen Meter Calibration Values (5340 ft. altitude)			
Temperature °C	Setting mg/L O_2	Temperature °C	Setting mg/L O_2
15.0	8.28	21.0	7.33
16.0	8.11	21.2	7.30
17.0	7.94	21.4	7.27
18.0	7.78	21.6	7.24
18.2	7.75	21.8	7.21
18.4	7.72	22.0	7.18
18.6	7.68	22.2	7.15
18.8	7.65	22.4	7.13
19.0	7.62	22.6	7.10
19.2	7.59	22.8	7.08
19.4	7.56	23.0	7.05
19.6	7.53	24.0	6.92
19.8	7.50	25.0	6.79
20.0	7.47	26.0	6.70
20.2	7.44	27.0	6.55
20.4	7.41	28.0	6.43
20.6	7.39	29.0	6.32
20.8	7.36	30.0	6.21

Attachment 8.

Calibration Values for Various Atmospheric Pressures and Altitudes

Calibration Values for Various Atmospheric Pressures and Altitudes					
Pressure			Altitude		Calibration
Inches Hg	mm Hg	Millibars	Feet	Meters	Value %
30.23	768	1023	-276	-84	101
29.92	760	1013	0	0	100
29.61	752	1003	578	85	99
29.33	745	993	558	170	98
29.02	737	983	841	256	97
28.74	730	973	1126	343	96
28.43	722	963	1413	431	95
28.11	714	952	1703	519	94
27.83	707	942	1995	608	93
27.52	699	932	2290	698	92
27.24	692	922	2587	789	91
26.93	684	912	2887	880	90
26.61	676	902	3190	972	89
26.34	669	892	3496	1066	88
26.02	661	882	3804	1160	87
25.75	654	871	4115	1254	86
25.43	646	861	4430	1350	85
25.12	638	851	4747	1447	84
24.84	631	841	5067	1544	83
24.53	623	831	5391	1643	82
24.25	616	821	5717	1743	81
23.94	608	811	6047	1843	80
23.62	600	800	6381	1945	79
23.35	593	790	6717	2047	78
23.03	585	780	7058	2151	77
22.76	578	770	7401	2256	76
22.44	570	760	7749	2362	75
22.13	562	750	8100	2469	74
21.85	555	740	8455	2577	73
21.54	547	730	8815	2687	72
21.26	540	719	9178	2797	71
20.94	532	709	9545	2909	70
20.63	524	699	9917	3023	69
20.35	517	689	10293	3137	68

Attachment 9.

YSI Model 5100 Barometer Calibration Instructions

The YSI Model 5100 has an internal barometer for pressure compensation during AUTO Dissolved Oxygen Calibration. This barometer only needs to be calibrated when it is no longer reading the correct barometric pressure. If the 5100 is kept at a fairly constant ambient temperature (±10°C), the barometer calibration should be accurate for approximately 30 days. As a result the barometer calibration **MUST** be performed monthly

The Model 5000 does not contain a barometer; therefore, the current barometric pressure must be entered before an AUTO Cal is performed. The pressure value displayed is the setting that was entered and stored during the previous calibrations.

From the calibration menu press the [DO CAL] soft-key, then press [NEXT] soft-key until the barometric pressure is flashing and “Barometer” appears in the top right corner of the display.

Using the [UP], [DOWN], and [DIGIT] soft-keys, enter the true local barometric pressure. This corresponds to a reading from a mercury barometer. Do **NOT** use the pressure reported by the weather bureau. Weather bureaus correct pressures to sea level.

NOTE: You may estimate the standard pressure at your altitude by using Attachment 9.

Press [ENTER} to confirm. The message “PRESSURE CALIBRATION SAVED” will be displayed, on the model 5100. The model 5000 will display “PRESSURE SETTING SAVED”, since it does not contain an internal barometer.

NOTE: If you wish to abort before pressing [ENTER}, you may press [MODE] to return to the calibrate menu without saving the new value for barometric pressure. You may also press [NEXT] to select a different parameter (any change made will not be saved).

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Title: Anions by Ion Chromatography

[EPA 300.0, SW 9056 and 9056A]

Approvals (Signature/Date):

Dave Elkin 12/21/10 Date Wet Chemistry Supervisor	Adam Alban 21 Dec 10 Date Health & Safety Manager / Coordinator
John Morris 12/21/10 Date Quality Assurance Manager	Robert C. Hanisch 12/21/10 Date Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This procedure describes the determination of the anions fluoride, chloride, nitrite, bromide, nitrate, ortho-phosphate, and sulfate in water samples by ion chromatography, based on EPA Method 300.0 and SW-846 Methods 9056 and 9056A.

1.1.2 This procedure can also be applied to leachates from soil samples. The soil leaching procedure is described in SOP DV-WC-0036.

1.1.3 The anions included in this procedure and their routine working ranges for interference-free samples are as follows:

Analyte	CAS Number	Working Range (mg/L)	Working Range (mg/kg)
Fluoride	66-30-0	1.0 – 10	10 – 100
Chloride	1-00-3	3.0 – 50	30 – 500
Nitrite as N	15-90-0	0.5 – 10	5 – 100
Bromide	28-20-0	0.2 – 10	2 – 100
Nitrate as N	25-90-0	0.5 – 10	5 – 100
Phosphate as P	226750-80-0	0.5 – 10	5 – 100
Sulfate	3-03-5	5.0 – 50	50 – 500

Note: The working range can be extended by dilution of the sample.

1.1.4 The reporting limits for the following analytes are based on a 25 µL injection volume:

Analyte	Water RL (mg/L)	Soil RL (mg/kg)
Fluoride	0.5	5
Chloride	3.0	30
Nitrite	0.5	5
Bromide	0.2	2
Nitrate	0.5	5
Phosphate	0.5	5
Sulfate	5.0	50

NOTE 1: Report nitrite (NO_2^-) as N, nitrate (NO_3^-) as N, and phosphate (PO_4^{-3}) as P.

NOTE 2: Depending client or project requirements, reporting limits may be higher than those shown above.

NOTE 3: Reporting limits for soils are based on the DI Leach procedure using a soil to water ratio of 1:10. Client-specific soil to water ratios may differ.

2.0 Summary of Method

2.1 Aqueous samples are measured directly, unless visible particulate is present or the measured concentration exceeds the calibration. Samples with particulate must be filtered before they are injected into the ion chromatograph. High concentration samples must be diluted for analysis. Soil samples are leached using deionized water in accordance with SOP DV-WC-0036, and the water leach is analyzed.

2.2 A small volume of sample is introduced into the ion chromatograph to flush and fill a constant volume loop. The sample is injected into a stream of carbonate-bicarbonate or hydroxide eluent.

2.3 The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a guard column and a separator column, are packed with low-capacity, strongly basic anion exchange resin. Ions are separated based on their affinity for the exchange sites of the resin. The last column is a suppressor column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids.

2.4 The separated anions in their acid forms are measured using an electrical conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

3.0 Definitions

3.1 This procedure includes the drinking water QC terminology from Method 300.0 and the solid waste terminology from SW-846 Methods 9056 and 9056A. Where there are two terms for the same concept, the cross reference is explained below. The frequency and evaluation of these QC samples are discussed in Sections 9 and 10.

3.2 **Calibration Blank (CB)** - A volume of reagent water fortified with the same matrix as the calibration standards, but without the addition of any of the analytes of interest.

3.3 **Laboratory Reagent Blank (LRB, also referred to as a Method Blank)** - For water samples, which do not require any preparation steps, the calibration blank and the method blank are the same thing. When soils are being analyzed, the method blank consists of the same reagents and preparation steps as applied to samples.

3.4 **Field Duplicates (FD)** - Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures. The laboratory project managers are responsible for informing clients of the relevant regulatory requirements for field duplicates.

3.5 **Laboratory Fortified Blank (LFB, also referred to as a Laboratory Control Sample, LCS)** - An aliquot of reagent water or other blank matrix to which known quantities of method analytes are added in the laboratory. The LCS is analyzed exactly like a sample and its purpose is to determine whether the methodology is

in control and whether the laboratory is capable of measurements the meet data quality objectives for accuracy and precision.

3.6 **Laboratory Fortified Sample Matrix (LFM, also referred to as a Matrix Spike, MS)** - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix affects the accuracy of the analytical result. The background concentrations of the analyte in the sample matrix must be determined if method analytes or other interference is present in the laboratory environment, the reagent, or the apparatus.

3.7 **Instrument Performance Check Solution (IPC, also referred to as Continuing Calibration Verification Standards, CCV)** - The CCV serves to monitor instrument drift from the beginning to the end of a given analytical sequence.

3.8 **Linear Concentration Range (LCR)** - The concentration range over which the instrument response is linear.

3.9 **Quality Control Sample (QCS)** - The QCS provides an independent verification of the accuracy of calibration standards and instrument performance. For the purposes of this SOP, the second-source ICV provides this verification.

3.10 Other quality control terms (e.g., MDL and PE sample) are defined in the Glossary Section of the Quality Assurance Manual.

4.0 Interferences

4.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

4.2 The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can usually be eliminated by the addition of the equivalent of 1 mL of concentrated eluent to 100 mL of each standard and sample. However, for routine samples, this problem is not severe enough to require this procedure.

4.3 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in the ion chromatograms.

4.4 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate (with carbonate/bicarbonate eluent) and other low molecular weight organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.5 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Nitrite	Oxidizer Toxic	None established	**Danger - strong oxidizer. Contact with other material may cause fire.** Toxic by inhalation; causes irritation to the respiratory tract and systemic poisoning, including intense cyanosis, nausea, dizziness, vomiting, collapse, spasms of abdominal pain, rapid heart beat, irregular breathing, coma, convulsions, and death due to circulatory collapse. Causes irritation, redness, and pain in contact with skin. May be absorbed through the skin causing systemic poisoning. Causes irritation, redness, and pain in contact with eyes.
Sodium Nitrate	Oxidizer	None established	**Danger - strong oxidizer. Contact with other material may cause fire.** Inhalation of dust irritates respiratory tract; symptoms may include coughing and shortness of breath. May cause irritation, redness, itching, and pain in contact with skin and eyes.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Fluoride	Poison	2.5 mg/m3 (TWA as F)	**Highly Toxic!** Inhaled or swallowed, this compound can cause fluoride poisoning. Early symptoms include nausea, vomiting, diarrhea, and weakness. Later effects include central nervous system effects, cardiovascular effects, and death. Causes severe irritation to the respiratory tract, symptoms may include coughing, sore throat, and labored breathing. Causes irritation in contact with skin, with redness and pain. Solutions are corrosive. Eye irritant! May cause irritation and serious eye damage. Effects may not appear immediately.
1 – Always add water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- **Balance:** Analytical, capable of accurately weighing to the nearest 0.0001 g. Checked for accuracy each day of use in accordance with DV-QA-0014.
- Conductivity meter
- **Ion Chromatograph:** Analytical system complete with ion chromatograph and all required accessories including analytical columns, compressed gases, and detectors.
- **Anion guard column:** A protector of the separator column. If omitted from the system, the retention times will be shorter. Usually packed with a substrate that is the same as on the separator column. Dionex AG14, 4 x 50 mm, Dionex AG17-C, or equivalent.
- **Anion separator column:** The separation shown in Figure 1 was generated using a Dionex AS14 column. An alternate column, e.g., Dionex AS17-C, may be used if comparable resolution of the peaks is obtained, and all QC requirements can be met.
- **Anion suppressor device:** Dionex Anion Self-Regenerating Suppressor, or equivalent.
- **Detector:** Conductivity cell, approximately 1.25 µL internal volume, Dionex, or equivalent.
- Dionex ion chromatography software.
- Interfaced computer with printer.
- **Computer Software and Hardware:** Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

6.2 Supplies

- Volumetric Flasks (Class A): various sizes
- Eppendorf Pipettes, varying volumes
- Autosampler vials and caps
- 0.2 micron nylon syringe filters, and other miscellaneous filtration materials
- Disposable plastic beakers
- Other miscellaneous laboratory supplies

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all materials listed below must be reagent grade, unless listed otherwise.

7.1 Reagent Water

Deionized water, Milli-Q grade, ≥ 18 Megohm-cm.

7.2 Eluent for New Generation ICs with column AS17-C

New chromatographs use the Dionex RFIC™ systems, which employs an eluent generator. The eluent generators are purchased from Dionex. The eluent generators use electrolysis to generate a potassium hydroxide eluent, the concentration of which is controlled by the instrument software.

7.3 Eluent Concentrate:

The eluent concentrate for IC 3 (column AS14) is prepared at concentrations of the eluent species that are 100 times greater than used in the eluent solution. Dissolve 37.12 g of sodium carbonate (Na_2CO_3) and 8.4 g of sodium bicarbonate ($NaHCO_3$) in reagent water and dilute to 1 liter. This solution may be purchased commercially.

NOTE: A small amount of eluent concentrate, i.e., 50 µL in 5 mL of sample, is added to each sample, QC sample, and standard injected to eliminate the water dip. The eluent concentrate is not added to samples and standards analyzed on newer generation ion chromatographs that employ an eluent generator.

7.4 Eluent Solution:

Dilute 20 mL of the eluent concentrate (7.3) in reagent water to 2 liters.

NOTE: The bicarbonate/carbonate eluent solution is used in older generation ion chromatographs. New chromatographs use the Dionex RFIC™ systems, which employs an eluent generator. The system uses electrolysis to generate a potassium hydroxide eluent, the concentration of which is controlled by the instrument software.

7.5 Stock Solutions (1,000 mg/L)

All stock solutions are purchased from commercial sources.

WARNING! Sodium fluoride is highly toxic. Exercise extreme caution when working with sodium fluoride. See Section 5.2.

7.5.1 Fluoride Stock Solution (1.00 mg of F^- in 1.00 mL of solution)

In a 1-liter volumetric flask, dissolve 2.21 g of sodium fluoride (NaF) in reagent water, and dilute to volume with reagent water. Store in chemical-resistant glass or polyethylene.

7.5.2 Chloride Stock Solution (1.00 mg of Cl^- in 1.00 mL of solution)
Dry sodium chloride (NaCl) for 1 hour at 600 °C, and cool in a desiccator. In a 1-liter volumetric flask, dissolve 1.6484 g (weigh to the nearest mg), of the dry salt in reagent water and dilute to volume with reagent water.

7.5.3 Nitrite Stock Solution (1.00 mg of NO_2^- as N in 1.00 mL of solution)
Place approximately 10.0 g of sodium nitrite ($NaNO_2$) in a 125 mL beaker and dry to a constant weight (about 24 hours) in a desiccator. In a 1-liter volumetric flask, dissolve 4.9257 g (weigh to the nearest mg) of the dried salt in reagent water and dilute to volume with reagent water. Store in a sterilized glass bottle. Refrigerate and prepare monthly.

- Nitrite is easily oxidized, especially in the presence of moisture, and only fresh reagents are to be used each day.
- Prepare sterile bottles for storing nitrite solutions by heating for 1 hour at 170 °C in an air oven.

7.5.4 Bromide Stock Solution (1.00 mg Br^- in 1.00 mL of solution)
Dry approximately 5.0 g of sodium bromide (NaBr) for 6 hours at 150 °C, and cool in a desiccator. In a 1-liter volumetric flask, dissolve 1.2877 g (weigh to the nearest mg) of the dried salt in reagent water and dilute to volume with reagent water.

7.5.5 Nitrate Stock Solution (1.00 mg NO_3^- as N in 1.00 mL of solution)
Dry approximately 10.00 g of sodium nitrate (NaNO3) at 105 °C for 24 hours. In a 1-liter volumetric flask, dissolve 6.0679 g (weigh to the nearest mg) of the dried salt in reagent water and dilute to volume with reagent water.

7.5.6 Phosphate Stock Solution (1.00 mg PO_4^{-3} as P in 1.00 mL of solution)
Dry approximately 10.0 g of potassium dihydrogen phosphate (KH2PO4) for 1 hour at 105 °C and cool in a desiccator. In a 1-liter volumetric flask, dissolve 4.3937 g (weigh to the nearest mg) of the dry salt in reagent water and dilute to volume with reagent water.

7.5.7 Sulfate Stock Solution (1.00 mg SO_4^{-2} in 1.00 mL of solution)
Dry approximately 5.00 g of potassium sulfate (K2SO4) at 105 °C for 1 hour and cool in a desiccator. In a 1-liter volume flask, dissolve 1.8141 g (weigh to the nearest mg) of the dried salt in reagent water and dilute to volume with reagent water.

NOTE: The stability of the stock standards is at least 1 month when stored at 4 °C. Dilute working standards should be prepared weekly, except those that contain nitrite and phosphate should be prepared fresh daily.

7.6 Second Source Stock Solutions (1,000 mg/L)
Stock solutions for each anion of interest are purchased at the same concentration but from a vendor other than the one that supplied the Stock Solutions described in Section 7.5. The second source stock solutions are used to prepare the ICV Intermediate Stock Solution (Section 7.8).

7.7 Calibration (CAL) Intermediate Stock Solution

Prepare the intermediate stock solution by combining the prescribed aliquots of each of the stock solutions described in Section 7.5(see table below) in a 100 mL volumetric flask. Dilute to volume with reagent water. Include all anions of interest, but omit all others.

Analyte	mL of Stock	Final Volume (mL)	Final Concentration (mg/L)
Fluoride	5.0	100	50
Chloride	25.0	100	250
Nitrite as N	5.0	100	50
Bromide	5.0	100	50
Nitrate as N	5.0	100	50
Phosphate as P	5.0	100	50
Sulfate	25.0	100	250

NOTE: The aliquots and final concentrations shown above are only recommended and assume a 1000 mg/L stock solution. Other aliquots and final concentrations may be used.

7.8 ICV Intermediate Stock Solution

Prepare the intermediate ICV stock solution by combining the prescribed aliquots of the second source (ICV) stock solutions (Section 7.6) in a 10 mL volumetric flask. Dilute to volume with reagent water. Include all anions of interest, but omit all others. The following table prescribes aliquot sizes for the individual anion stock solutions.

Analyte	mL of Stock	Final Volume (mL)	Final Concentration (mg/L)
Fluoride	0.5	10	50
Chloride	2.5	10	250
Nitrite as N	0.5	10	50
Bromide	0.5	10	50
Nitrate as N	0.5	10	50
Phosphate as P	0.5	10	50
Sulfate	2.5	10	250

NOTE: The aliquots and final concentrations shown above are only recommended and assume a 1000 mg/L stock solution. Other aliquots and final concentrations may be used.

7.9 Working Standards

Prepare working standards by pipetting 10mL aliquots of the CAL Intermediate Stock Solution (Section 7.7) into 100 mL volumetric flasks. Dilute to volume with reagent water. Refer to the following tables for the concentration of each analyte in the calibration standard.

7.9.1 Calibration Standard #1

Dilute 10 uL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	0.1
Chloride	0.5
Nitrite as N	0.1
Bromide	0.1
Nitrate as N	0.1
Phosphate as P	0.1
Sulfate	0.5

7.9.2 Calibration Standard #2

Dilute 50 uL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	0.5
Chloride	2.5
Nitrite as N	0.5
Bromide	0.5
Nitrate as N	0.5
Phosphate as P	0.5
Sulfate	2.5

7.9.3 Calibration Standard #3

Dilute 100 uL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	1.0
Chloride	5.0
Nitrite as N	1.0
Bromide	1.0
Nitrate as N	1.0
Phosphate as P	1.0
Sulfate	5.0

7.9.4 Calibration Standard #4

Dilute 0.4 mL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	4.0
Chloride	20.0
Nitrite as N	4.0
Bromide	4.0
Nitrate as N	4.0
Phosphate as P	4.0
Sulfate	20.0

7.9.5 Calibration Standard #5

Dilute 0.8 mL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	8.0
Chloride	40.0
Nitrite as N	8.0
Bromide	40.0
Nitrate as N	8.0
Phosphate as P	8.0
Sulfate	40.0

7.9.6 Calibration Standard #6

Dilute 1 mL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	10.0
Chloride	50.0
Nitrite as N	10.0
Bromide	10.0
Nitrate as N	10.0
Phosphate as P	10.0
Sulfate	50.0

NOTE: The aliquots and final concentrations shown above are recommended for calibration. Other aliquots and final concentrations may be used.

7.9.7 Lower Limit of Quantitation (LLOQ)
Dilute 20 uL of the CAL Intermediate Stock Solution (Section 7.7) to 5 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	0.2
Chloride	1.0
Nitrite as N	0.2
Bromide	0.2
Nitrate as N	0.2
Phosphate as P	0.2
Sulfate	1.0

7.10 Initial Calibration Verification (ICV)
Dilute 1.0 mL of the second source ICV Intermediate Stock Solution (Section 7.8) to 25 mL using reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	2.0
Chloride	10.0
Nitrite as N	2.0
Bromide	2.0
Nitrate as N	2.0
Phosphate as P	2.0
Sulfate	10.0

7.11 Continuing Calibration Verification (CCV) and Laboratory Control Sample (LCS) Solution
Prepare the CCV and LCS spike solution by diluting 10.0 mL of the ICAL Intermediate Stock Solution (Section 7.7) to 100 mL with reagent water.

Analyte	Final Concentration (mg/L)
Fluoride	5.0
Chloride	25.0
Nitrite as N	5.0
Bromide	5.0
Nitrate as N	5.0
Phosphate as P	5.0
Sulfate	25.0

NOTE: The aliquots and final concentrations shown above are recommended. Other aliquots and final concentrations may be used.

7.12 Stock Spiking Solutions (5000 mg/L chloride, sulfate and 1000 mg/L fluoride, bromide, nitrate, phosphate/1000 mg/L nitrite)

For the spiking solution, solids are dried and desiccated as listed in Section 7.4 for each individual component of the solution. Two solutions are made as summarized in the following two tables. Alternatively, a commercially prepared solution may be used.

Stock Spiking Solution #1

Analyte	Mass of Solid (g)	Final Volume (mL)	Anion Conc. (mg/L)
Sodium Fluoride	2.21	1000	1000
Sodium Chloride	8.2424	1000	5000
Sodium Bromide	1.2876	1000	1000
Sodium Nitrate	6.068	1000	1000
Potassium Dihydrogen Phosphate	4.3936	1000	1000
Potassium Sulfate	9.0704	1000	5000

Stock Spiking Solution #2

Analyte	Mass of Solid (g)	Final Volume (mL)	Anion Conc. (mg/L)
Sodium Nitrite	4.9256	1000	1000

7.13 Spikes for the Matrix Spike and Matrix Spike Duplicate (MS/MSD)

A working solution consisting of 10 mL of Stock Spiking Solution #1 and 10 mL of Stock Spiking Solution #2 is used. The MS is prepared by adding 50 µL of this working solution to a 5 mL aliquot of the selected sample. The MSD is prepared by adding 50 µL of this working solution to a second 5 mL aliquot of the same sample. This will result in the following anion concentrations ("true values") in the spiked sample: 25 mg/L for chloride and sulfate, and 5 mg/L for fluoride, nitrite, bromide, nitrate, and phosphate.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Analyte	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Fluoride	HDPE	50 mLs	Cool 4 ± 2°C	28 days	40 CFR Part 136.3
Chloride	HDPE	50 mLs	Cool 4 ± 2°C	28 days	40 CFR Part 136.3
Nitrite as N	HDPE	50 mLs	Cool 4 ± 2°C	48 hours	40 CFR Part 136.3
Bromide	HDPE	50 mLs	Cool 4 ± 2°C	28 days	40 CFR Part 136.3
Nitrate as N	HDPE	50 mLs	Cool 4 ± 2°C	48 hours	40 CFR Part 136.3
Phosphate as P	HDPE	50 mLs	Cool 4 ± 2°C	48 hours	40 CFR Part 136.3
Sulfate	HDPE	50 mLs	Cool 4 ± 2°C	28 days	40 CFR Part 136.3

NOTE: Soil leachates follow the same preservation and holding times as the water samples, starting from the time of extraction.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. The process of establishing control limits, and the use of control charts are described more completely in DV-QA-003P, Quality Assurance Program. Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents.

9.3 Attachment 1 reconciles the various QC requirements specified in the reference methods with the QC requirements specified in this SOP.

9.4 Before analyzing samples, the laboratory must establish a method detection limit (MDL) as described in Section 12.1 and the linear concentration range (LCR) as described in Section 9.7.6. In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument they will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12 for more details.

9.5 Batch Definition
Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See Policy DV-QA-003P for further details.

9.6 **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.6.1 Method Blank (same as Laboratory Reagent Blank, LRB)
A method blank (MB) is required with every batch of 20 or less samples. The MB is deionized water taken through the procedure as if it were a sample.

Acceptance Criteria: The MB must not contain anions of interest above the reporting limit or above one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL).

Corrective Action: If the method blank exceeds allowable levels, laboratory contamination is suspected and corrective action must be taken before continuing. All samples associated with the failed blank must be reanalyzed

9.6.2 Laboratory Control Sample (same as Laboratory Fortified Blank, LFB)

One Laboratory Control Sample (LCS) is required with each analytical batch. Depending on client or project requirements, an LCS duplicate may also be analyzed. The LCS and LCSD are prepared as described in Section 7.11. An LCS that is determined to be within acceptance criteria effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch.

Acceptance Criteria: For Method 300.0, the LCS recovery for each analyte of interest must be within 90-110%. For Method 9056, the LCS recovery for each analyte of interest must be within statistical control limits, not to exceed 85-115%. For Method 9056A, the LCS recovery for each analyte of interest must be within statistical control limits, not to exceed 80-120%.The absolute value of the relative percent difference (RPD) between the LCS and LCSD must be ≤10%. Statistical control limits are set at ± 3 standard deviations around the historical mean. The process of establishing control limits is described in more detail in Policy DV-QA-003P. Control limits are maintained in the LIMS.

Corrective Action: If the LCS recovery falls outside of the established control limits, and/or when the RPD for the LCS/LCSD exceeds the RPD limit, then check instrument conditions and the standards being used for problems. Correct any problems before continuing. Reanalyze all samples associated with the failed LCS.

9.6.3 Matrix Spike / Matrix Spike Duplicate (MS/MSD, same as Laboratory Fortified Matrix)

For Method 9056, one MS/MSD pair is required with each analytical batch of 20 or fewer samples. For Method 300.0, one MS is required for every 10 routine samples. Also note that some programs (e.g., North Carolina and South Carolina) require an MS/MSD pair for every 10 samples. The MS and MSD are prepared as described in Section 7.13.

Acceptance Criteria: The recovery of each anion of interest must be within the established statistical control limits. Statistical control limits are set at ± 3 standard deviations around the historical mean, and must be within 80-120%. The relative percent difference (RPD) between the MS and MSD must be less than 20%, or less than the established control limit, depending on project requirements. The process of establishing control limits is described in more detail in Policy DV-QA-003P. Control limits are maintained in the LIMS.

Corrective Action: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS

and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly;
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses); and
- Document the failure in an NCM and note it on the final report.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and document the failure in an NCM.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.7 **Instrument QC** - The following quality control samples are prepared with each analytical instrument run.

9.7.1 Initial Calibration Verification (ICV)

The second-source ICV, as described in Section 7.10, is analyzed immediately following the ICAL.

Acceptance Criteria: The ICV recovery for each anion must be 90-110%. The retention time for each analyte in the ICV must be within ± 10% of the established retention time for that analyte.

Corrective Action: If the recovery and/or retention time is outside of the acceptance limits, repeat the test. If the test fails on the second attempt, then the problem must be investigated and the instrument recalibrated for the failed analyte(s).

9.7.2 Initial Calibration Blank (ICB)

An ICB is analyzed following the ICV.

Acceptance Criteria: The result must be less than the reporting limit.

Corrective Action: If the blank is above the acceptance limit, check for carryover or the need for instrument

maintenance. The instrument must be recalibrated.

9.7.3 Continuing Calibration Verification (CCV, same as IPC in Method 300.0)

A CCV is required after every 10 or fewer samples and after the last sample.

Acceptance Criteria: The CCV recovery must be 90-110%. The retention time for each analyte in the CCV must be within ± 10% of the established retention time for that analyte.

Corrective Action: If the recovery and/or retention time is outside of the acceptance limits, the instrument must be recalibrated, and all samples analyzed since the last successful CCV must be reanalyzed.

9.7.4 Continuing Calibration Blank (CCB)

A CCB is analyzed after each CCV.

Acceptance Criteria: The result must be less than the reporting limit.

Corrective Action: If the blank is above the acceptance limit, check for carryover or the need for instrument maintenance. The instrument must be recalibrated, and all samples analyzed since the last successful CCB must be reanalyzed.

9.7.5 Lower Limit of Quantitation (LLOQ)

A LLOQ is required for method 9056A to verify the data reporting limit for each analyte.

Acceptance Criteria: The LLOQ recovery must be ± 50% of the true value.

Corrective Action: If the recovery is outside of the acceptance limits, the instrument must be recalibrated.

9.7.6 Linear Concentration Range (LCR)

9.7.6.1 The LCR must be determined initially and verified every 6 months or whenever a significant change in instrument response is observed or a significant change (eg. Change to column type, eluent or instrument pressures) in instrument configuration is made.

9.7.6.2 The initial demonstration of linearity must use a sufficient number of standards to ensure that the resulting curve is linear.

9.7.6.3 The semi-annual verification of linearity must use a minimum of a blank and three standards. If the recovery for any analyte falls outside of 90-110%, linearity must be reestablished.

9.7.6.4 Linearity study data are maintained by the Wet Chemistry group leader.

9.7.7 Retention Time Study

The width of the retention time window used to make identifications should be based on measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms. See Section 10.3.4 for detailed instructions on performing the retention time study.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 Screen the conductivity of samples prior to analysis to determine whether the samples require dilution using a conductivity meter. Record the conductivity on a sample observation benchsheet if the conductivity is > 3000 µmhos/cm.

10.1.2 If the conductivity is > 3000 µmhos/cm, dilute as necessary with reagent water, based on the following calculation, rounding up to the smallest round

$$\text{dilution factor} = \frac{conductivity}{3000}$$

dilution that will bring the conductivity of the dluted sample to under 3000.

NOTE: If a sample requires filtration, prior to being loaded on the instrument, the method blank **must** also be filtered.

10.2 Calibration

10.2.1 An initial calibration is performed every three months, or as needed, based on instrument performance and maintenance.

10.2.2 Calibrate the instrument at six levels. See Section 7.9 for preparation of calibration standards.

NOTE: It is generally NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason is clearly documented, for example a cracked tube, and a minimum of five levels remain.

10.2.3 Construct a calibration curve using a weighted linear regression. Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

Acceptance Criteria: The absolute value of the correlation coefficient must be 0.995 or greater.

Corrective Action: If the correlation coefficient is less than the acceptance limit, recheck instrument conditions and calibration standards. Samples cannot be analyzed until the initial calibration is successful.

10.2.4 Attachment 2 summarizes the recommended operating conditions for the ion chromatograph.

10.2.5 Attachment 3 is an example chromatogram. Included in this chromatogram are example retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the data quality objectives can be met.

10.2.6 Check system calibration daily by analyzing an ICV and ICB (see Sections 9.7.1 and 9.7.2) and, if required, recalibrate.

10.3 Sample Analysis

10.3.1 When using the older generation ion chromatographs that do not employ eluent generation, add 50 µL of eluent concentrate (Section 7.3) to 5 mL of each standard, sample, and QC sample prior to injecting.

10.3.2 Load samples into the autosampler according to the schedule. The instrument will flush and load the sample loop for injection. See Attachment 2 for sample loop specifications. The instrument software detects and integrates peaks in the resulting chromatograph.

10.3.3 Following is a typical analytical sequence:

ICAL
ICV and ICB
LCS and LCSD (if LCSD required)
Method Blank
10 injections
CCV and CCB
10 injections
CCV and CCB
10 injections
CCV and CCB

10.3.4 Retention Times and Anion Identification

10.3.4.1 The width of the retention time window used to make identifications is based on measurements of actual retention time variations of standards over 72 hours. (This should be done prior to analysis of samples since the retention time window must be entered into the software before starting the run). The retention time windows should be re-evaluated and a retention time study run when significant changes (eg. Change to column type, eluent or instrument pressures) are made to the instrument.

10.3.4.2 Record the retention time of each calibration standard on at least 3 different days. Calculate the standard deviation for each analyte. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.3.4.3 The calibration curve is verified each working day or whenever the eluent is changed by analyzing an ICV and ICB, and after every 10 injections by analyzing a CCV and CCB. If the retention time for any analyte varies from the expected values (±5% for phosphate and sulfate and ± 10% for fluoride, chloride, nitrite, bromide and nitrate) a new calibration curve must be prepared.

10.3.4.4 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze. The addition of one to two times the sample concentration normally provides the best peak height for analyte identification.

NOTE: Concentration can affect retention time and cause peak migration. Late eluting species, e.g., nitrate and sulfate, exhibit the greatest amount of change, although all anions are affected to some degree. In some cases, this peak migration may produce poor resolution or misidentification. If a peak has shifted outside of its retention time window (as confirmed by a CCV or Matrix Spike), change the window in the software and reprocess the chromatogram. Document the reason for reprocessing the chromatogram along with the date and initials.

10.3.4.5 Should more complete resolution be needed between peaks, the eluent can be diluted. This will spread out the run but will also cause the later eluting anions to be retained longer. The analyst must determine to what extent the eluent is diluted. This dilution should not be considered a deviation from the method.

10.3.5 If the response for the peak exceeds the working range of the system, dilute the sample with reagent water and reanalyze.

11.0 Calculations / Data Reduction

11.1 Using the computer and the Dionex software package, prepare a linear calibration curve for each analyte by plotting instrument response against standard concentration. The software calculates a calibration function in the following form:

$$Y_i = SX_i + I$$

Where:

Y_i = Instrument response (peak area) for specific anion in the i^{th} calibration standard.
X_i = Concentration of anion in the i^{th} calibration standard, mg/L.
S = Slope of calibration curve determined by linear regression analysis.
I = Intercept of calibration curve determined by linear regression analysis.

11.2 The anion concentration in the injected sample is calculated by solving the calibration function for Xi as follows:

$$X_i = \frac{Y_i - I}{S}$$

Where:

X_i = Calculated concentration of the i^{th} sample at the instrument, mg/L.
Y_i = Instrument response for the i^{th} sample (peak area).
I = Intercept of the calibration curve.
S = Slope of the calibration curve.

11.3 If the sample was diluted, the final anion concentration in the original sample is calculated as follows:

$$X_f = X_i \times DF$$

Where:

X_f = Anion concentration in original sample, mg/L.
X_i = Calculated concentration of sample at the instrument, mg/L.
DF = Dilution factor.

11.4 For soil leachates, the concentration in the original soil sample is calculated as follows:

$$X_s = X_i \times DF \times \frac{V}{M_s}$$

Where:

X_s = Anion concentration in original soil sample, mg/kg.
X_i = Calculated concentration of sample at instrument, mg/L.
DF = Dilution factor, if applicable.
V = Volume of leachate, L.
M_s = Mass of original soil sample, kg.

11.5 Report only those values that are less than the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

11.6 Use the appropriate data qualifier (i.e., "flag") to indicate when a sample requires dilution due to high conductivity. In most cases, a "G" flag denotes dilution due to matrix effects, and a "Q" indicates dilution to bring the sample into the calibration range. Special flagging conventions may apply depending on client or project requirements.

11.7 All results are subject to two levels of review, which are documented on the form shown in Attachment 4.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method Detection Limit Study an initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Prepare and analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P) for each analyte.
- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy # DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

NOTE: EPA method 300.0 requires an MDL study to be performed every 6 months.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of capability (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

- Initially the analyst must perform an MDL study (see section 12.1).
- Four aliquots of the QC check sample (independent source from the calibration) are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration standard.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

- If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive ICVs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

The use of ion chromatography eliminates the need to use a variety of hazardous reagents required by the other approved methods for the determination of the same anions.

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

- IC process waste from older generation ion chromatographs – aqueous carbonate/bicarbonate eluent waste: Non-hazardous
- IC process waste from new generation ion chromatographs with hydroxide eluent generating system: Excess Sample – Aqueous – Waste Stream W

- Expired aqueous reagents/standards – Contact Waste Coordinator
- Expired solid chemicals – Contact Waste Coordinator

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA Publication SW846, 3rd Edition, Final Update IIIB (December 1996), Method 9056, "Determination of Inorganic Anions by Ion Chromatography", Revision 0, September 1994.

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA Publication SW846, 4th Edition, Final Update IV, Method 9056A, "Determination of Inorganic Anions by Ion Chromatography", Revision 1, February 2007.

15.3 Method 300.0, "Determination of Inorganic Anions by Ion Chromatography", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, Revision 2.1, August 1993.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 300.0	Method 300.0 includes a requirement for a second-source quality control standard (QCS), which is to be run quarterly and be within ±10% of the expected value. This SOP establishes calibration accuracy every day of operation using a second-source initial calibration verification (ICV) standard.
2	EPA 300.0	Method 300.0 specifies that target analytes must be less than the MDL in the Laboratory Reagent Blank. TestAmerica Denver QA policy (Policy DV-QA-003P) defines the acceptance limit for the method blank as the laboratory reporting limit (RL) and not the MDL. If specified in client or project requirements, the method blank acceptance limit may be set at the MDL.
3	EPA 300.0, SW 9056 and SW 9056A	Methods 9056 and 9056A specifies bomb combustion for solid waste samples. Method 300.0 specifies water leaching for solid samples. This SOP specifies water leaching and references SOP DV-WC-0036 for the deionized water leach procedure. In this respect, this SOP complies with Method 300.0, but deviates from Method 9056A.

17.0 Attachments

Attachment 1: Quality Control Summary
Attachment 2: Suggested Standard Instrument Operating Parameters
Attachment 3: Example Ion Chromatogram
Attachment 4: Example Data Review Checklist

18.0 Revision History

- Revision 7.2, dated 23 December 2010
  - Annual Technical Review

- Revision 7.1, dated 04 December 2009
  - Added bullet in section 6.1 concerning the Master List of Documents.
  - Added note on filtration to section 10.1.
  - Updated Attachment 4

- Revision 7, dated 19 June 2009
  - Added Lower limit of quantitation information.
  - Updated reagent and standard preparation information.
  - Made minor formatting and grammatical corrections.
  - Added 6 month MDL requirement note to section 12.1
  - Updated Attachment 4.

Revision 6, dated 22 February 2008

- Update the recipes for the eluent concentrate and eluent solution to current practices.
- Added the information concerning the eluent generators and need to purchase them from Dionex.

- Revision 5, dated 22 February 2008
  - Integration for TestAmerica and STL operations.
  - Updated formatting.
  - Moved the reporting limit table to Section 1.
  - Updated attachments with the TestAmerica logo.

- Revision 4, dated 24 July 2007
  - The method Reference has been changed for SW846 from Method 9056 to method 9056A.
  - The company name has been changed from STL to TestAmerica.

- Revision 3, dated 28 February 2006
  - Revised Sections 5, 14, and 15 (Safety, Pollution Prevention, and Waste Management, respectively) to comply with STL Corporate requirements.
  - Updated formatting to comply with current STL Denver guidance (Policy QA-001).
  - Updated the reporting limit table in Section 1.1.4 to reflect current reporting limits.
  - Expanded discussion of the matrix spike quality control criteria (Section 9.6.3) to be consistent with Policy QA-003, Quality Control Program.
  - Removed details concerning Method Performance, and included references to the applicable STL Denver SOPs.
  - Expanded Attachment 1, Quality Control Summary, to include references to the source methods.

- Revision 2, dated 6 September 2002
  - Calibration standards updated and includes addition of eluent concentrate to

working standards.
- Eluent concentrate recipe added.
- Changed reporting limit tables to reflect current reporting limits.
- Added requirement for MS/SD pair per 10 samples for certain clients or agencies.
- The spike concentration was increased to avoid overly diluting the unspiked sample when matrix spikes are prepared.
- The Method Performance Section 13 was expanded.
- A Data Review Checklist was added to the end of the SOP.

- Revision 1, dated 9 June 1999
  - The size of the injection loop for low level analysis is changed from 300 uL to 25 uL, which is the manufacturer's recommendation for the latest equipment.

Attachment 1.

Quality Control Summary

QC Samples	Frequency	Acceptance Criteria	Corrective Action	Reference Method Equivalent
Initial Calibration Verification (ICV)	Immediately following the initial calibration, and at the start of each day prior to sample analysis, or whenever the eluent is changed.	90 - 110% of true value RT must be ± 10% of established RT	Repeat once, and recalibrate and reanalyze if it fails a second time.	QC Reference Sample (9056A) IPC and QCS (300.0)
Initial Calibration Blank (ICB)	After the ICV and prior to sample analysis.	≤ the Reporting Limit	Re-prepare and reanalyze	Calibration Blank (300.0)
Laboratory Control Sample (LCS)	1 per QC batch	Within laboratory historical limits	Recalibrate and reanalyze all samples associated with unacceptable LCS	LFB (300.0)
Matrix Spike Sample (MS/MSD)	1 MS/MSD pair per QC batch for 9056. 1 MS/MSD pair per 10 samples for 300.0.	%R within laboratory historical limits and RPD ≤ laboratory historical limits.	If LCS and CCVs are in control, then document in an NCM, unless project requires reanalysis.	Matrix Spike (9056A) LFM (300.0)
Continuing Calibration Verification (CCV)	Between each group of 10 injections and at the end of the analytical sequence.	90 - 110% of true value	Repeat. If repeat fails, recalibrate and reanalyze all samples since the last acceptable CCV.	Mid-range Calibration Standard (9056A) IPC (300.0)
Continuing Calibration Blank (CCB)	Between each group of 10 injections and at the end of the analytical sequence	< the Reporting Limit	Repeat. If repeat fails, recalibrate and reanalyze all samples since the last acceptable CCB.	Calibration Blank (300.0)
Method Blank (MB)	1 per QC batch	≤ the Reporting Limit	See Section 9.6.1 or Policy QA-003.	LRB (300.0)

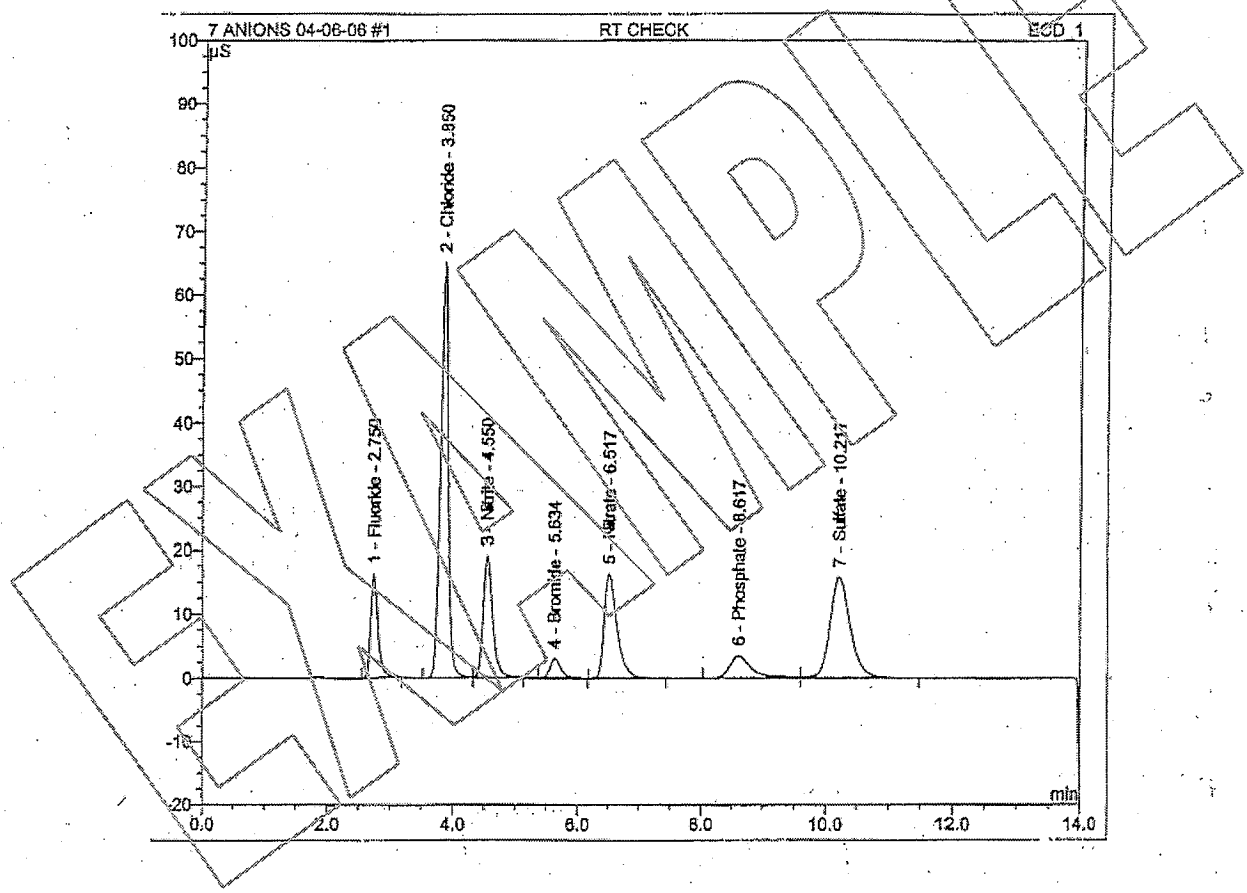
Attachment 2.

Suggested Standard Instrument Operating Procedures

Instrument Conditions	
Anion Guard Column	Dionex AG 4A or AG17-C
Anion Separator Column	Dionex AS4A or AS17
Supressor Device	SRS Ultra II Self-Regenerating Suppressor
Pump Rate	1.2 mL/min or 1.0 mL (depending on specific instrument used)
Sample Loop	10 µL
Eluent	Older Generation Instruments: 1.0 mM sodium bicarbonate, 3.5 mM sodium carbonate Dionex RFIC™ Systems: Potassium hydroxide, generated by electrolysis of water, in the range of 10 to 40 mM
Detector Output	Baseline conductivity should be between 15 - 16 µS prior to sample analysis.

Attachment 3.

Example Ion Chromatogram



Attachment 4.

Example Data Review Checklist

TESTAMERICA Denver

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Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Lot / Sample Numbers	Matrix	Batch	Method	QC	Special Input

	Yes	No	N/A	2nd Level
A. Calibration/Instrument Run QC				
1. Minimum of five standards in ICAL or as specified in method?				
2. Correlation coefficient ≥ 0.995 ?				
3. Second-source ICV analyzed, and recovery 90-110%?				
4. ICB analyzed immediately after the ICV & results < the RL?				
5. CCV analyzed after every ten samples & recovery ± 10% of true value?				
6. CCB analyzed after every CCV & results < RL?				
7. Absolute value of the intercept is < ± ½ the RL?				
B. Sample Results				
1. All samples greater than highest calibration standard diluted and reanalyzed?				
2. Do associated RLs/MDLs reflect dilutions or limited sample volume?				
3. All reported results bracketed by in control CCV results?				
4. Sample analyses done within holding time?				
5. Initial pH check documented for all samples?				
6. Preparation benchsheet completed and included in package?				
7. Client requirements reviewed and met?				
8. Were data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures and correct units?				
9. Do the prep and analysis dates in QuanTIMS reflect the actual dates? Lot Dates report checked?				
10. Are all data being reported highlighted on the benchsheet?				
11. Raw data copies prepared and scanned?				
12. Manual integrations done properly and initialed and dated?				
13. STD/True Value sheet is updated and included?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10x blank have NCM?				
2. Method blank < ½ RL or NCM provided?				
3. LCS/LCSD run for batch and within QC limits?				
4. MS/MSD run at required frequency and within limits or NCM written?				
5. DUP run at required frequency and RPD within 20% or NCM written?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

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Electronic Copy Only

Title: Alkalinity by Automated Titration [EPA 310.1, SW9040B, SM2320B, SM 4500-H+B]

Approvals (Signature/Date):

Dave Elkin — Date 11/2/10
Wet Chemistry Supervisor

Adam Alban — Date 04 Nov 10
Health & Safety Manager / Coordinator

John P. Morris — Date 11/2/10
Quality Assurance Manager

Robert C. Hanisch — Date 11/4/10
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This procedure is to be used for the determination of alkalinity. The different forms of alkalinity (total, bicarbonate, carbonate, and hydroxide) can also be calculated.

Analyte	CAS Number
Alkalinity	N/A

1.1.2 This method is applicable to all ranges likely to be encountered. As a practical matter, samples with an alkalinity greater than about 1200 mg/L as calcium carbonate require a reduced volume or stronger titrant in order to keep the titrant volume to a reasonable amount.

2.0 Summary of Method

2.1 Samples are analyzed by each method simultaneously on an automated titrator as follows:

2.1.1 The pH is determined electrometrically with a glass electrode in combination with a reference electrode. The special glass used in the electrode develops a voltage across it that depends on the pH of the solution being analyzed. The voltage is measured and converted to pH by calibration against buffers of known pH.

2.1.2 Alkalinity is determined by titration of the sample with a standardized acid to specified endpoints (pH 8.3 and 4.5). Alkalinity is calculated from the volume of acid required to reach the endpoints and is traditionally reported as calcium carbonate. Samples for alkalinity should not be altered (i.e., filtered or diluted).

3.0 Definitions

3.1 **pH** - At a given temperature, the intensity of the acidic or basic character of a solution is indicated by pH or hydrogen ion activity. Because of the ionic interactions in all but very dilute solutions, it is necessary to use the "activity" of the hydrogen ion and not its molar concentration. The approximate equivalent to molarity can be presumed only in very dilute solutions. A logarithmic scale is used for pH in order to express a wide range of hydrogen ion activities. Neutral pH is 7.0 at 25 °C, while acidic pH's are <7 and basic pHs are >7.

3.2 **Alkalinity** – A measure of the acid-neutralizing capacity of water.

4.0 Interferences

4.1 The pH response of most glass electrodes is imperfect at both ends of the scale. The indicated pH value of highly alkaline solutions, as measured with the glass electrode, will be too low. The indicated pH value of salts and strong acids having a pH less than 1, will often be higher than the true pH value. Interferences can be minimized by the selection of

the proper electrodes for these conditions. For example, sodium may interfere at pH > 10, and is controlled by using a "low sodium error" electrode.

4.2 The pH electrode may exhibit slow or noisy response with high purity waters due to the lack of ionic strength.

4.3 Coatings of oil and particulate matter may impair electrode response.

NOTE: If the electrode becomes coated with oil, immerse in a mild detergent solution, rinse well with deionized water, and recalibrate. If this fails, try rinsing in 10% HCl.

4.4 Temperature variations will change the pH of the samples and also affect electrode response. Electronic temperature correction must be used to correct for electrode response.

4.5 Salts of weak organic and inorganic acids will contribute to alkalinity. If the alkalinity is intended to be a measure of carbonate and bicarbonate only, the presence of these substances will cause high results.

4.6 The pH 4.5 is the routine endpoint for total alkalinity. This assumes the normal carbon dioxide/bicarbonate/carbonate/hydroxide mass action interrelationships for natural waters. Other types of waters can have other interrelationships that might dictate the use of a different endpoint.

4.7 Samples not in equilibrium with the atmosphere may exhibit changes in pH and in the distribution of the various forms of alkalinity when exposed to the atmosphere. The sample containers should be filled completely and kept closed until just prior to the analysis. The analysis should be performed as soon as possible.

4.8 Particulates in the sample may affect the alkalinity results. This interference can cause an error in the ion balance calculation.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Man-Tech Autotitator, consisting of:
  - Burivar I/2 Buret Module
  - Titrasip Titration Module
  - PC-Tis Interface Module
  - PC running PC-Titrate software
- Radiometer Autotitrator, consisting of:
  - SAC 80 Sample Changer
  - TIT 85 Titrator
  - ABU 80 Autoburette
- Combination pH Electrode – Epoxy-covered glass, with temperature correction, Ross Sure-Flow or equivalent.

6.2 Supplies

- Tubes to fit autosampler. These must be thoroughly rinsed to remove all traces of salt if reused.
- Pipette calibrated to 10.0 mL, and disposable tips.
- Miscellaneous laboratory apparatus and glassware.

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software/hardware located on G\QA\Read\Master List of Documents\Master List of Documents and Software/hardware.xls for the current software to be used for data processing.

7.0 Reagents and Standards

Reagents - All materials must be reagent grade or higher quality, unless otherwise specified

7.1 All of the standard materials are obtained from commercial sources, and must be NIST traceable.

7.2 **pH Buffers:** 4, 7, and 10.

7.3 **Alkalinity Standards:**

7.3.1 **Sodium Carbonate solution (1N)**
This standard is purchased commercially.

7.3.2 **Sodium Carbonate solution (200 mg/L as CaCO3)**
Pipette 4mL of the 1N Sodium Carbonate Solution into a 1 liter volumetric flask. Bring to volume with de-ionized water.

7.3.3 **0.02 N Sulfuric Acid (Alkalinity Titrant)**
Purchase from a commercially available source.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Method	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
2320B	HDPE	1 liter	Cool 4 ± 2°C	14 Days	40 CFR Part 136.3
9040B	HDPE	1 liter	None	Analyze Immediately	40 CFR Part 136.3

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit

Quality Controls	Frequency	Control Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	± 10% of the true value
Sample Duplicate - pH	1 in 20 or fewer samples[2]	≤0.05 units
Sample Duplicate - Alkalinity	1 in 20 or fewer samples	≤ 10% RSD

[1] MB is not possible for pH
[2] Some programs (e.g., South Carolina and North Carolina) require duplicates to be analyzed at a 10% frequency.

9.2 Instrument QC

Initial Check and Continuing Calibration Verification

- Calibration verification standard (CCV) and a continuing calibration blank (CCB) are to be analyzed after ten samples and at the end of the run.
- The 200 mg/L standard (7.3.2) is used for the alkalinity Initial check and CCV.
- The pH 7 buffer is used for the pH CCV.
- A blank is not appropriate for pH analysis.

10.0 Procedure

10.1 Sample Preparation

Allow the sample to come to room temperature before analyzing.

10.2 Calibration

10.2.1 Initial Calibration for pH

- The pH meter is calibrated each day of operation.
- Be sure that the reference electrode has been filled with 3M potassium chloride.
- Calibration of the pH meter is done using pH 4,7, and 10 buffers.
    - Fill the first four tubes in the autosamples with the following order of samples: pH 4 buffer, pH 7 buffer, pH 10 buffer, and deionized water.
    - Click on the button "PH CALIBRATION 4-7-10" and follow the screens to calibrate.
    - When calibration has finished, go to titrator and choose "examine calibrations." Print the calibration if it is valid.
    - Open up "documents" and select the bottom option (Alk-new) to update the buffers and standards. Update info and print this out.

10.3 Sample Analysis

10.3.1 Be sure the titrant reservoir of 0.02 N sulfuric acid is at least half full. Be sure that there are no air bubbles in the line. Fill deionized water container used for rinses to the top.

10.3.2 Click on the button "Conductivity – pH – Alkalinity". Click load template and load the appropriate template. Add the sample IDs to the schedule and save the template.

10.3.3 Begin to load the autosampler as indicated by the schedule, using approximately 40 mL in each tube. Click the button "start" when ready to begin analysis.

10.3.4 The titrator has been programmed to deliver a maximum volume of 25mL titrant. Samples requiring more than this should be reanalyzed using a smaller aliquot.

10.3.5 Calculate the results according to the calculation section below.

NOTE 1: Low level alkalinity (<20mg/L) is performed by the autotitrator checking measurements every 0.3 pH units.

NOTE 2: If there is a pH greater than 4.5 with an alkalinity result of zero than the high level alkalinity method needs to be performed (see DV-WC-0085)

11.0 Calculations / Data Reduction

11.1 Standardization of Alkalinity Titrant

$$N_{ACID} = \frac{N_{BASE} \times V_{BASE}}{V_{ACID}}$$

Where:

N_{ACID} = Normality of titrant
N_{BASE} = Normality of Sodium Carbonate
V_{BASE} = Volume of Sodium Carbonate titrated, mL
V_{ACID} = Volume of titrant needed for titration, mL

11.2 pH values are recorded directly from the titrator printout.

11.3 Three values for alkalinity will be printed if the pH is greater than 8.3. Record the values at pH 8.3 and 4.5 on the bench sheet. The third value is the difference and is ignored. Note that some results will be printed in scientific notation; be careful to check this when recording results.

11.4 If the pH is less than 8.3, only one value will be printed (at pH 4.5). Record this value on the bench sheet. P alkalinity is ND on these samples.

11.5 Calculate the concentration as follows:

$$\text{Total Alkalinity, mg/L } CaCO_3 = \left(\frac{(2B - C) \times N \times 50{,}000}{\text{mL of sample}} \right)$$

Where:

B = Volume of titrant to first recorded pH, in mL.
C = Total volume of titrant added to reach a pH level 0.3 units lower.
N = Normality of acid.

11.6 Calculate the individual forms of alkalinity as follows:

Result of Titration	Hydroxide Alkalinity	Carbonate Alkalinity	Bicarbonate Alkalinity
P = ND	ND	ND	T
P < T/2	ND	2P	T - 2P
P = T/2	ND	2P	ND
P > T/2	2P - T	2(T - P)	ND
P = T	T	ND	ND

Where:

T = Total Alkalinity = Alkalinity at pH 4.5
P = Phenolphthalein Alkalinity = Alkalinity at pH 8.3

11.7 Record the values for Total, Bicarbonate, Carbonate, and Hydroxide Alkalinity.

11.8 Reporting

- Alkalinity results less than 5mg/L are reported as ND. All forms of alkalinity are reported in mg/L as Calcium Carbonate.
- Report case narratives for South Carolina must include date and time of collection.
- All data are subject to two levels of review, which is documented on the checklist shown in attachments 3 and 4.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P) for each analyte.

- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy # DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows."

- Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use, has the required experience, and has successfully analyzed initial demonstration samples (see SOP # DV-QA-0024 for details).

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Titrated sample waste – Aqueous Acidic - Waste Stream F

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 pH:

- Method 9040B, "pH Electrometric Measurement", Test Methods for Evaluating Solid Waste, EPA SW-846 Third Edition, 1/95.
- Method 4500-H+ B, pH Value, "Standard Methods for the Examination of Water and Wastewater," 20th Edition, 1998.

15.2 Alkalinity:

- Method 2320B, Alkalinity – Titration Method, "Standard Methods for the Examination of Water and Wastewater," 20th Edition, 1998.
- U.S. EPA, Method for Chemical Analysis of Water and Waste, Method 310.1, Approved 1978.

16.0 Method Modifications:

Item	Method	Modification
1	310.1 & SM 2320B	The method requires an initial verification of the sulfuric acid titrant. The laboratory purchases the titrant from a commercial source and therefore does not perform additional verification.

17.0 Attachments

Attachment 1: Example Data Review Checklist for Titrations

18.0 Revision History

- Revision 6.2, dated 19 November 2010
  - Added reagent quality requirements to section 7.0
- Revision 6.1, dated 03 May 2010
  - Added section 6.3
  - Annual Review
- Revision 6, dated 27 March 2009
  - Deleted the Conductivity method from SOP
  - Updated other SOP references and formatting
  - Deleted Attachment 2 – Data Review Checklist for Direct Measurements.

- Added references to DV-WC-0085 (manual titration) for high alkalinity samples.

- Revision 5, dated 30 January 2008
  - Integration for TestAmerica and STL operations.
  - Updated formatting
  - Removed references to EPA method 305.1

- Revision 4.1, dated 20 November 2006
  - For this minor revision, the technical content of this SOP was not reviewed or updated. The purpose of this minor revision is to incorporate the Safety Bulletin information to ensure compliance with STL Corporate requirements. In addition, Section 9.1 was revised to reference Policy QA-024 for specific QC requirements for federal programs, any existing interim changes were incorporated, and formatting was updated consistent with Policy QA-001.

- Revision 4, dated 17 September 2002
  - Company name changed from Quanterra to STL Denver.
  - Definitions for conductivity and alkalinity were added to Section 3.
  - Details about the instrumentation were added to Section 6.
  - The pH meter is calibrated with 3 buffers, including a pH 7 buffer.
  - Details about the standards were added in section 7.
  - The old SOP made reference to LCS. Instead, this SOP refers to these solutions as calibration verification standards in Section 10.4
  - The high conductivity curve option was removed because current instrumentation does not allow adjustment of the cell constant.
  - The low level alkalinity option in the old SOP was redundant because all analyses use the 0.02N sulfuric acid, the low-level titrant.

Attachment 1.

Example Data Review Checklist for Titrations

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver Wet Chemistry Data Review Checklist
For Titration Methods

Test Name/Method #: _____ **Analysis Date:** _____

SOP #: _____ **Analyst:** _____ **Instrument:** _____

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Was the normality of the titrant verified and found acceptable?				
B. Sample Results				
1. Are all sample dilutions appropriate and do associated RLs/MDLs reflect required dilutions or limited sample volume?				
2. All reported results bracketed by in control LCS or QC Sample?				
3. Sample analyses done within holding time?				
4. Initial pH check documented for all samples (if required)?				
5. Preparation benchsheet completed and included in package (if applicable)?				
6. Special client requirements met?				
7. Was data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures?				
8. Do the prep and analysis dates in QuanTIMS reflect the actual dates?				
9. Are all data being reported highlighted on the benchsheet?				
10. Raw data copies prepared and scanned?				
C. Preparation/Matrix QC				
1. Method blank < RL for all reported samples > 10x method blank result?				
2. LCS run for batch and within QC limits?				
3. MS and/or MSD run at required frequency and within limits (if applicable)?				
4. Sample DUP run at required frequency and RPD within 10%?				

Analyst: _____ **Date:** _____
Comments: _____

2nd Level Reviewer : _____ **Date:** _____
Comments: _____

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Total Kjeldahl Nitrogen (TKN)

[EPA 351.2]

Approvals (Signature/Date):

David M. Elkin 2/11/10
Dave Elkin Date
Wet Chemistry Supervisor

2-11-10
Adam Alban Date
Health & Safety Manager / Coordinator

02/11/10
Karen Kuoppala Date
Quality Assurance Manager

Robert C Hanisch 2/11/10
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This procedure is used for the analysis of drinking and surface waters, domestic and industrial wastes, and soil samples for Total Kjeldahl Nitrogen (TKN), CAS# 5228003-90-0, by EPA Method 351.2.

1.2 During the digestion, amino acids, proteins, peptides and other nitrogenous compounds of biological origin are converted to ammonium sulfate. Nitrogenous compounds of some industrial waste such as amines, nitro compounds hydrazones, oximes, semicarbazones and some tertiary amines may not be converted.

1.3 The reporting limit is 0.50 mg/L for aqueous samples and 25 mg/kg for soil/waste samples. The range may be extended by sample dilution or by changing the detector sensitivity.

2.0 Summary of Method

Samples are digested by heating in the presence of sulfuric acid, potassium sulfate, and copper sulfate catalyst to a final temperature of 380°C. Free ammonia and organic nitrogen compounds are converted to ammonium sulfate. The ammonium is then reacted with salicylate and hypochlorite in a buffered alkaline solution in the presence sodium nitroprusside at a pH of 12.8 - 13.0 to form a blue-green compound whose intensity is measured at 660 nm.

3.0 Definitions

Total Kjeldahl Nitrogen (TKN) is the sum of free ammonia and organic nitrogen compounds.

4.0 Interferences

4.1 Some nitrogenous compounds such as amines, nitro compounds, hydrazones, oximes, and semicarbazones may not be broken down by the digestion procedure causing low TKN results.

4.2 During digestion, nitrate in excess of 10 mg/L can oxidize a portion of the ammonia released from the digested organic nitrogen, producing N_2O and resulting in a negative interference. When sufficient organic matter in a low state of oxidation is present, nitrate can be reduced to ammonia, resulting in a positive interference.

4.3 Inorganic salts and solids (from SM 4500-Norg A):
The acid and salt content of the Kjeldahl digestion reagent is intended to produce a digestion temperature of about 380°C. If the sample contains a very large quantity of salt or inorganic solids that dissolve during digestion, the temperature may rise above 400°C, at which point pyrolytic loss of nitrogen begins to occur. To prevent an excessive digestion temperature, add more H_2SO_4 to maintain the acid-salt balance. Not all salts cause precisely the same temperature rise, but adding 1 mL H_2SO_4/g salt in the sample gives reasonable results. Add the extra acid and the digestion reagent to both sample and reagent blank. Too much acid will lower the digestion temperature below 380°C and result in incomplete digestion and recovery. If necessary, add sodium hydroxide-sodium thiosulfate before loading onto the autoanalyzer to neutralize the excess acid.

4.4 The color reaction is very sensitive to the acid and salt content of the samples. All standards must be digested. Any dilutions performed after digestion must be made with the digested blank to maintain the proper matrix.

4.5 This procedure does not require distillation like some TKN determinations. See (67 FR 652198 – October 23, 2002) "Other TKN methods explicitly require alternate sample preparation procedures, such as the semi-automated block digestion (e.g., Method 351.2)."

4.6 Ammonia is included in the TKN result and is a common laboratory reagent and cleaning chemical. Contamination of samples with ammonia will give erroneously high results. Do not digest samples in the same hoods where ammonia is used as a reagent. Cleaning chemicals used in the laboratory should be ammonia-free. Glassware should be thoroughly rinsed with deionized water to remove any ammonia residues. After digestion, samples should be covered with foil in the tubes if they can not be promptly diluted and placed in capped vials.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.3 Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Nitroferricyanide	Poison	5 mg/m^3 as HCN gas	Very toxic. May be fatal if inhaled or swallowed. May cause irritation in contact with the skin. **Gives off HCN gas if combined with strong acids. Inhalation of HCN gas can cause irritation, dizziness, nausea, unconsciousness, and potentially death.**
Sodium Hydroxide	Corrosive	2 mg/m3 (Ceiling)	Sever irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and with greater exposures, it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/m3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Block digestor (e.g. Tecator 2040 Digester with Controller)
- Alpkem (OI analytical) auto-analyzer system consisting of sampler, pump, manifold, detector, and computer with software WinFLOW for data system.

OR

- Astoria® Analyzer automated continuous flow analysis equipment designed to deliver and react sample and reagents in the required order and ratios. System consists of autosampler, multi-channel pump, TKN analysis cartridge, detector and computer with FASPac software.
- Pump tubing of appropriate size for use on Astoria® Analyzer or ALPKEM autoanalyzer.
- Nitrogen gas supply and appropriate lines and fittings

6.2 Supplies

- Thermometer (400°C range).
- Digestion tubes.
- Vortex mixer.
- Hengar boiling stones.
- Re-pipettor, 25 mL.
- Graduated cylinder, Class A, 25 mL
- Miscellaneous laboratory apparatus and glassware, including volumetric flasks, volumetric class A pipettes, beakers, etc.

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

The automated salicylate method is utilized with this analysis; reagents vary depending on the instrument used. All reagents should be reagent grade unless manufactured specifically for this method. The reagents and their concentration listed below are for use with either the Alpkem autoanalyzer or Astoria autoanalyzer, as labeled.

7.1 Reagent water (ASTM type II or equivalent), distilled or deionized water, free of the analytes of interest.

7.2 **Digestion Reagents**
Dissolve 134 g of K_2SO_4 and 7.3 g of $CuSO_4$ in 800 mL of reagent water, **very slowly** and with constant stirring, add 134 mL of conc. H_2SO_4 acid. The solution will get **VERY HOT** on the addition of acid. Allow the solution to cool before diliuting to 1L in a volumetric flask. This reagent is also available commercially from RICCA Chemical.

7.3 **Manifold Startup Solution - Brij-35, 30% solution(ALPKEM or Astoria)**
Add approximately 1 mL Brij-35 (a solution purchased from commercial sources) to 500 mL of de-ionized water and mix gently.

7.4 **10 N Sodium Hydroxide (1 L)(ALPKEM or Astoria)**
Dissolve 250g of sodium hydroxide in approximately 800 mL of deionized water, allow to cool, then dilute with de-ionized water to 1L in a volumetric flask. Store tightly capped in a plastic container. Different volumes of 10 N sodium hydroxide can be prepared depending on requirement by increasing proportions. This reagent is available commercially from Fisher or VWR Scientific.

7.5 **Stock Buffer, Sodium Phosphate dibasic (1L)(ALPKEM or Astoria)**
Dissolve 134g of sodium phosphate-dibasic in approximately 800 mL of de-ionized water. Add 50 mL of 10N sodium hydroxide and dilute to 1L in a volumetric flask, mix well

7.6 **Stock Potassium Sodium Tartrate Solution (1L)(ALPKEM or Astoria)**
Dissolve 200g sodium potassium tartrate in approximately 800 mL of de-ionized water and dilute to volume in a 1L volumetric flask. Dilute to mark and mix well.

7.7 **Working Buffer (1L)(ALPKEM)**
Add 200 mL of stock phosphate buffer (7.5) to 200 mL of de-ionized water in a 1 L volumetric flask and mix. While stirring, add 250 mL of stock potassium sodium tartrate (7.6). Continue stirring and slowly add 60 mL of 10 N sodium hydroxide (7.4). Dilute the solution to the mark and mix well. Degas the solution, then add 0.5 mL of Brij-35, 30% and mix gently to prevent foaming. Degas the solution prior to use.

7.8 **Working Buffer (1L)(Astoria)**
Add 200 mL of stock phosphate buffer (7.5) to 200 mL of de-ionized water in a 1 L volumetric flask and mix. While stirring, add 250 mL of stock potassium sodium tartrate (7.6). Continue stirring and slowly add 88 mL of 10 N sodium hydroxide

(7.4). Dilute the solution to the mark and mix well. Degas the solution, then add 10 drops of of Brij-35, 30% and mix gently to to prevent foaming. Filter if baseline is noisy.

7.9 Sodium Salicylate/Sodium Nitroprusside Solution(500 mL)(ALPKEM or Astoria)

Dissolve 75g of sodium salicylate and 0.3g of sodium nitroferricynide in approximately 300 mL of de-ionized water and dilute to volume with deionized water in a 500 mL volumetric flask. Mix well to completely dissolve all sodium nitroprusside. This solution must be stored in a dark (light-resistant) container.

7.10 Sodium Hypochlorite Solution(ALPKEM or Astoria)

Add 12 mL of sodium hypochlorite solution to approximately 180 mL of de-ionized water in 200 mL volumetric flask. Dilute to mark and mix well. Prepare fresh daily from commercial liquid bleach. Do not use "ultra" or scented bleach. Store in an amber bottle.

7.11 Calibration Standards

7.11.1 Ammonia Cal Stock Standard, 1000 mg/L as N

Dry ammonia chloride at 105 C. Dissolve 3.819 g in 5600 mL of deionized wate, add 2 mL of concentrated sulfuric acid, and dilute to 1000 mL with deionized water. This solution is normally obtained from commercial vendors also.

7.11.2 Ammonia Cal Intermediate Cal Standard, 100 mg/L as N

Add 50 mL of Ammonia Cal standard to a 500 mL which also contains 1.0 mL of 1:1 sulfuric acid. Dilute to the mark with deionized water.

7.11.3 Ammonia Cal Intermediate Cal Standard, 25 mg/L as N

Add 25 mL of Ammonia cal intermediate standard to a 100 mL which also contains 1.0 mL of 1:1 sulfuric acid. Dilute to the mark with deionized water.

7.11.4 Second-Source Calibration Verification Standard (ICV)

Purchase a commercially prepared concentrate with a certified value equal to or greater than 2 mg/L. This should be prepared according to manufacturer's instructions and has a true value that varies with individual lot of standard. Normally, ERA Complex Nutrients is the ICV, but equivalent standards may be used.

7.11.5 Calibration Standards

All standards must be digested. New standards must be prepared and digested at least monthly. Store digested standards in tightly sealed containers to prevent absorption of ammonia. Alternatively, these can be prepared by spiking directly into digestion tubes.

Prepare standards as indicated below using the 25 mg/L intermediate stock:

Volume (mL) Standard (7.9.1)	Final Volume (mL)	Concentration as mg/L N
0.25	25	0.25
0.5	25	0.5
1.0	25	1.0
2.0	25	2.0
*5.0	25	5.0
10.0	25	10.0

*This level is prepared as the Continuing Calibration Verification (CCV).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE or Glass	1 Liter	H_2SO_4, pH < 2; Cool 4 ± 2°C	28 Days	40 CFR Part 136.3
Soils	Glass	4 oz	Cool 4 ± 2°C	28 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and

trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.1.1 Method Blank

A method blank (de-ionized water) is required with every batch of 20 or less samples.

Acceptance Criteria: Method blanks must be less than two times the reporting limit.

Corrective Action: If this level is exceeded, the batch must be reprepared and reanalyzed.

9.1.2 Laboratory Control Samples (LCS)

An LCS is required with every batch of 20 or less samples. The LCS standard is 6.0 mg/L made from the 6.0 mL of intermediate stock standard diluted to 25 mL with de-ionized water.

Acceptance Criteria: LCS control limits are based on historical data (see Policy DV-QA-003P for details), and the control limits are available in LIMS.

Corrective Action: If the acceptance criteria are not met, the batch must be reprepared and reanalyzed.

9.1.3 Matrix Spike and Matrix Spike Duplicates (MS/MSD)

An MS and MSD are required with every 10 samples or less. Add 3 mL of 25 mg/L standard to 25 mL of sample and digest. The spiking concentration is 3 mg/L.

Acceptance Criteria: MS/MSD recovery and relative percent difference control limits are based on historical data, and the control limits are available in LIMS.

Corrective Action: If MSD/MSD recoveries exceed allowable levels and the LCS is in control, the data will be flagged as outside of control limits. Document the results in an exception report. If the RPD is greater than RPD limit the samples should be reanalyzed.

9.1.4 Troubleshooting Guide

- Check for contamination in the reagents and standards. Be sure all reagents were made correctly and have not exceeded their expiration dates.
- All samples and standards must contain the same salt and acid concentrations.
- Check system for obvious problems such as plugs and leaks.
- Low LCS recovery usually indicates incomplete digestion.
- If you are unable to locate and solve the problem, consult your supervisor.

9.2 Instrument QC

9.2.1 Initial calibration verification standard (ICV): Immediately following the initial calibration, a mid-range second-source initial calibration verification standard (ICV) is analyzed. The recovery for this standard must be within 10% of the true value. If this is not achieved, the instrument must be recalibrated.

9.2.2 Continuing calibration verification standard (CCV): A CCV is analyzed at a frequency of at least 10% between samples, and analyzed at the end of the analytical sequence. The acceptance criteria for the CCV is ± 10% of the true value. If this is not achieved, the instrument must be recalibrated and the samples run since the last successful CCV must be reanalyzed.

9.2.3 Continuing calibration blank (CCB): A CCB is analyzed after every CCV. The CCB consist of deionized water. The CCB results must be less than the reporting limit. If the blank is greater than the reporting limit, check for carry-over from high level samples, clean the system, recalibrate, and rerun all samples analyzed since the last successful CCB.

9.2.4 Linear Calibration Range (LCR): The LCR is performed initially by each analyst and then verified every analysis. A blank, LCS, CCV, and ICV are performed each analysis, this fulfills the method requirement of a blank and three standards for verification of linearity.

Acceptance Limits: The acceptance limits for the LCR standards are ± 10% of the true value.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, and chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.1 Interference Checks

The measuring and recording the pH of water samples with pH paper is done at the time of receipt of the samples in sample receiving. The pH should be less than 2. If it is not, prepare a nonconformance describing the problem and check with the lab PM before proceeding.

10.2 Sample Preparation(ALPKEM or Astoria)

10.2.1 Prepare a block digestor lay-out sheet to determine what will be loaded into each digestion tube position.

10.2.2 Place 25 mL of sample, blank, LCS and calibration standards into the appropriate digestion tubes. For calibration standards and LCS, pipette the appropriate amount of stock or intermediate standard to each tube and rinse it down the side well with de-ionized water to an approximate total volume of 25 mL. Use volumetric class A pipettes or micro-liter pipettes.

10.2.3 For solid samples, weigh 0.5 g sample into the tube and rinse down any sample that clings to the side walls of the tube using deionized water.

10.2.4 Samples which contain high levels of salt or organic nitrogen may need to be diluted prior to digestion. Record any dilutions performed on the bench sheet.

10.2.5 Add 5 mL of digestion reagent.

10.2.6 Add 4-6 Hengar Granules and place the tubes on the block digestor. Use a digestor lay-out sheet to record the sample IDs located in each position.

10.2.7 Heat the samples for 2 hours at 140 °C followed by 2 1/2 hours at 380 °C. Depending on the model used, it may be possible to temperature program the block digestor. See the block digestor manual for specific programming instructions.

10.2.8 After digestion, allow the tubes to cool to room temperature.

10.2.9 Carefully and accurately add 25mL of de-ionized water using a class A graduated cylinder to measure the water. Point the tube toward the back of the hood when doing this in case the mixture splatters out of the tube. Samples may be allowed to sit for a short time with the deionized water in order to aid in dissolution of the salts before proceeding to 10.2.10.

10.2.10 Mix the samples on the vortex mixer and ensure that all salts in the bottom of the tubes have completely dissolved. Transfer to a labeled vial and seal. Analyze the samples as soon as possible after digestion.

10.3 System Startup: ALPKEM

NOTE: Consult the Alpkem operating instructions located near the instrument for guidance on start up. This method is somewhat generalized - the specific detail for analysis on the Alpkem Auto-Analyzer systems can be found in operation manual on line.

10.3.1 Be sure that the instrument is set up with the correct manifold, flow cell, detector wavelength (660 nm), temperature, etc. A generalized flow diagram is attached (Attachment 1); detailed information can be found in the instructions for the specific instrument being used. The temperature on the TKN heating coil should be set to 37°C.

10.3.2 Check reagent levels and replenish as needed.

10.3.3 Allow the sytem to flow with start-up solution (7.3) for approximately 10-15 minutes.

10.3.4 Once the flow is stable, place the reagent lines into the reagent containers, except for the salicylate line. **CAUTION:** If the salicylate reagent is pumped too soon, a precipitate may form inside the lines.

10.3.5 The salicylate line may be placed in its proper container after the other reagents have been pumped through the entire system.

NOTE: If a precipitate forms after the addition of salicylate, immediately stop the proportioning pump and flush the coils with water using a syringe. Precipitation of salicylic acid is caused by a low pH. Before restarting the system, check the concentration of the sulfuric acid solutions and/or the working buffer solution.

10.4 System Startup: Astoria

NOTE: Consult the Astoria operating manual located near the instrument for guidance on start up. This method is somewhat generalized.

10.4.1 Be sure that the instrument is set up with the correct manifold as shown in the flow diagram included in Attachment 2. The temperature on the TKN heating coil should be set to 50°C.

10.4.2 Turn on the power to all units including heating coil and place all reagent lines in startup solution.

10.4.3 Latch the pump platens on both the main and auxiliary (traveling wash reservoir) pumps and begin flow. Verify that the bubble size and spacing is consistent.

10.4.4 Allow the sytem to flow with start-up solution (7.3) for approximately 10-15 minutes.

10.4.5 Once the flow is stable, place the reagent lines into the reagent containers, except for the salicylate line. **CAUTION:** If the salicylate reagent is pumped too soon, a precipitate may form inside the lines.

10.4.6 The salicylate line may be placed in its proper container after the other reagents have been pumped through the entire system.

NOTE: If a precipitate forms after the addition of salicylate, immediately stop the proportioning pump and flush the coils with water using a syringe. Precipitation of salicylic acid is caused by a low pH. Before restarting the system, check the concentration of the sulfuric acid solutions and/or the working buffer solution and make sure all lines are pumping properly. Check reagents for any contamination caused by backflow when the manifold was clogged.

10.5 Calibration(ALPKEM or Astoria)

Calibration standards are prepared as described in Section 7.11, and digested as described in Section 10.2. The instrument start up is also described in Section 10.3. Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

The instrument is initially calibrated (ICAL) using the six concentrations shown in 7.9. The correlation coefficient r for the linear regression equation should be 0.995 or more. If the r is less than 0.995, investigate and correct problem before recalibrating.

10.6 Sample Analysis(ALPKEM or Astoria)

10.6.1 Prepare the sample table with the appropriate calibration and with the intended sequence of samples.

10.6.2 Load the calibration standards and samples into the autosampler.

10.6.3 Initiate the autosampler and start with the analysis of the sync peak. Verify that an acceptable calibration curve has been obtained before proceeding.

10.6.4 The ICV, ICB, LCS and MB should be analyzed next.

10.6.5 Analyze samples. Any samples which exceed the response of the high standard must be diluted with digested blank solution and reanalyzed. Samples must not be diluted with deionized water but with digested blank solution, or an incorrect value will be reported. Do not over-dilute the samples.

10.6.6 Analyze a continuing calibration verification and continuing calibration blank after every 10 or less samples, and again at the end of the run.

10.6.7 Carefully monitor the reagent to ensure that you do not run out of buffer. Running out of buffer will cause salicylate to precipitate out in the lines.

10.7 Instrument Shut-Down(ALPKEM)

10.7.1 Remove the salicylate line first and place in start-up solution. Allow reagents to flow for several minutes until all salicylate has been flushed from the system.

10.7.2 Place all other reagent lines in start-up solution and flush the system for at least 10 minutes.

10.7.3 Turn off the instrument modules and release the pump platens.

10.8 Instrument Shut-Down(Astoria)

10.8.1 Remove the salicylate line first and place in start-up solution. Allow reagents to flow for several minutes until all salicylate has been flushed from the system. Turn off the heating coil.

10.8.2 Place all other reagent lines in start-up solution and flush the system for at least 10 to 15 minutes.

10.8.3 Turn off the instrument modules and release the pump platens from both the main and auxiliary pumps.

11.0 Calculations / Data Reduction

NOTE: The Alpkem and Astoria systems automatically perform all data acquisition.

11.1 Enter the standard concentrations and peak heights into a linear least squares program. The Alpkem software performs these calculations automatically.

11.2 All calibration equations can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005. Use the least squares equation to calculate the results for the samples from the peak heights. Multiply the result obtained by any dilutions made during prep or analysis. The Alpkem software does this automatically, so long as the dilution factor is entered into the dilution factor field in the sample table.

11.3 Total Organic Nitrogen can be determined by subtracting ammonia nitrogen from the TKN result. TKN and ammonia are often analyzed on the same sample. The TKN result should always be greater than or equal to the ammonia result, taking into account the possible analytical errors.

11.4 Reporting

- Reporting units are mg/L as N for aqueous samples and mg/kg as N for soil/waste samples.
- Results less than the reporting limit is reported as ND. The detection limits must be raised if dilutions were made during sample prep or due to interferences.
- All results are subject to two levels of data review, which are documented on the checklist shown in Attachment 3.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method Detection Limit Study an initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Prepare and analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy DV-QA-005P) for each analyte.
- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

- Four aliquots of the QC check sample (independent source from the calibration) are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

12.3.1 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Acid Waste (Wet Chemistry) - Waste Stream F

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Method Source: EPA Method 351.2, Revision 2.0, August, 1993

15.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition, 4500-Norg A

15.3 Related Documents

- Tecator Manual, 2000 Digestion System
- Alpkem (OI Analytical) Automated Ion Analyzer Manual

16.0 Method Modifications:

Item	Method	Modification
1	EPA 351.2	Method 351.2 states the calibration standards are made with ammonium chloride. The laboratory uses L-glutamic acid to calibrate for verification of the digestion process. A series of three ammonium chloride standards were prepared at levels of 5 ppm and 0.2 ppm, and then analyzed against the L-glutamic acid curve. The standards were found to be comparable to the organic nitrogen standards at the 5 ppm level with ± 10% and ± 50% at the MDL concentration. The results are kept on file for proof of verification.
2	EPA 351.2	The initial temperature for digestion was reduced to 140°C to prevent overheating and splattering of the samples, which occurred at the higher temperature (160°C) specified in the source method. The time was increased to be sure that the water is completely driven off before ramping to the high temperature.
3	EPA 351.2	The dynamic range has been changed to 0.25 - 7 mg/L from 0.1 - 20 mg/L to improve linearity.

17.0 Attachments

Attachment 1: Flow Chart
Attachment 2: Manifold Flow Diagram
Attachment 3: Example Data Review Checklist

18.0 Revision History

- Revision 6, dated 12 February 2010
  - Added Section 6.3
  - Updated Sections 7.11.1 to 7.11.3
  - Updated Section 7.11.5 (standards table)
  - Updated Section 9.1.2

- Revision 5, dated 24 July 2009
  - Fixed multiple section references throughout SOP
  - Deleted Attachment 1 Digestor instructions
  - Added reference to the digestor manual to section 10.2.7
  - Added Attachment 2 Manifold Flow diagram
  - Updated Attachment 3 to current checklist

- Revision 4, dated 04 May 2009
  - Added the Linear Calibration Range information in Section 9.2.4
  - Added method modification #1
  - Clarified that IDOCs need to be performed with a second source standard.
  - Corrected minor grammatical and formatting errors.

- Revision 3, dated 30 September 2008
  - Added Astoria technical information throughout SOP.

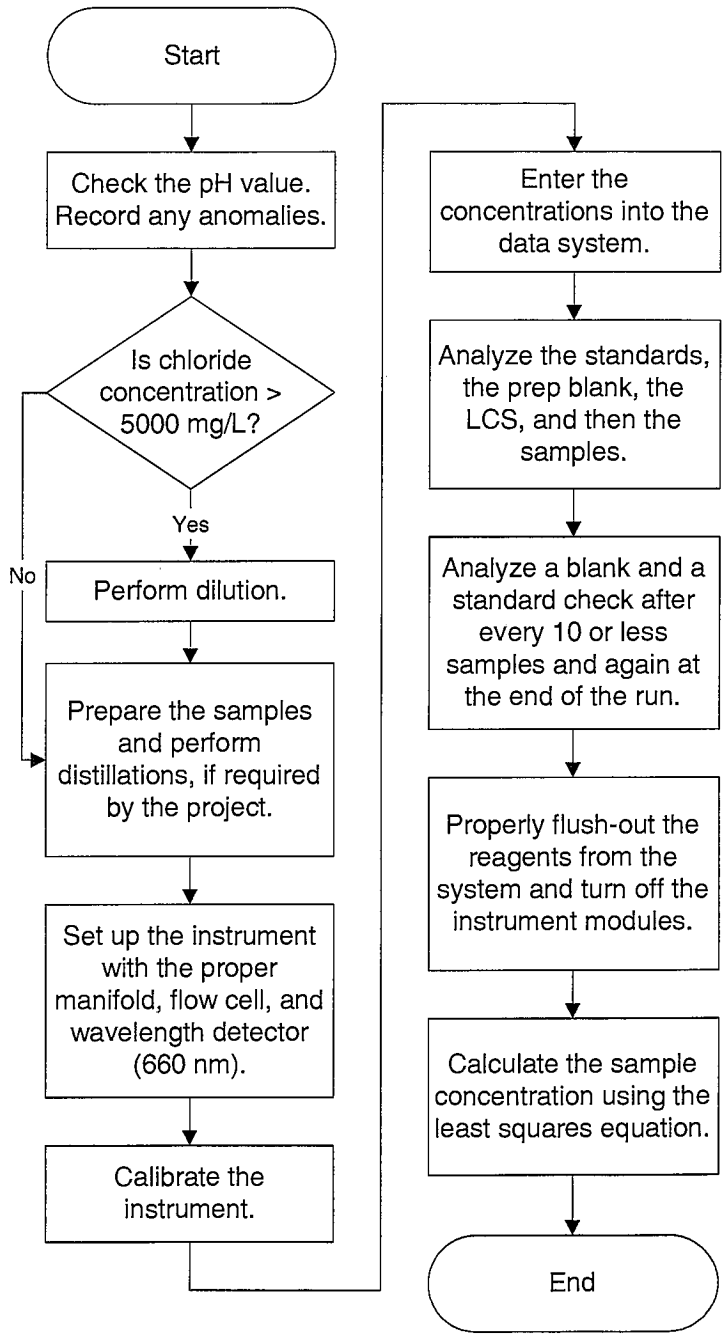
- Revision 2, dated 29 February 2008
  - Integration for TestAmerica and STL operations.
  - Updated formatting
  - Changed the nomenclature for SOPs throughout the SOP to match the new format.

- Revision 1.1, dated 20 November 2006
  - For this minor revision, the technical content of this SOP was not reviewed or updated. The purpose of this minor revision is to incorporate the Safety Bulletin information to ensure compliance with STL Corporate requirements. In addition, Section 9.1 was revised to reference Policy QA-024 for specific QC requirements for federal programs, any existing interim changes were incorporated, and formatting was updated consistent with Policy QA-001.

- Revision 1.0, dated 15 January 2003
  - Safety and Waste Management sections were updated to reflect current format and practice.
  - Initial calibration levels were updated.
  - A reference to a distillation for New Jersey was removed, and a citation noting that distillation is not part of this procedure was added as section 4.5.
  - An example Data Review Checklist was added as Attachment 2.
  - Section 13, Method Performance, was updated to reflect current content and format.

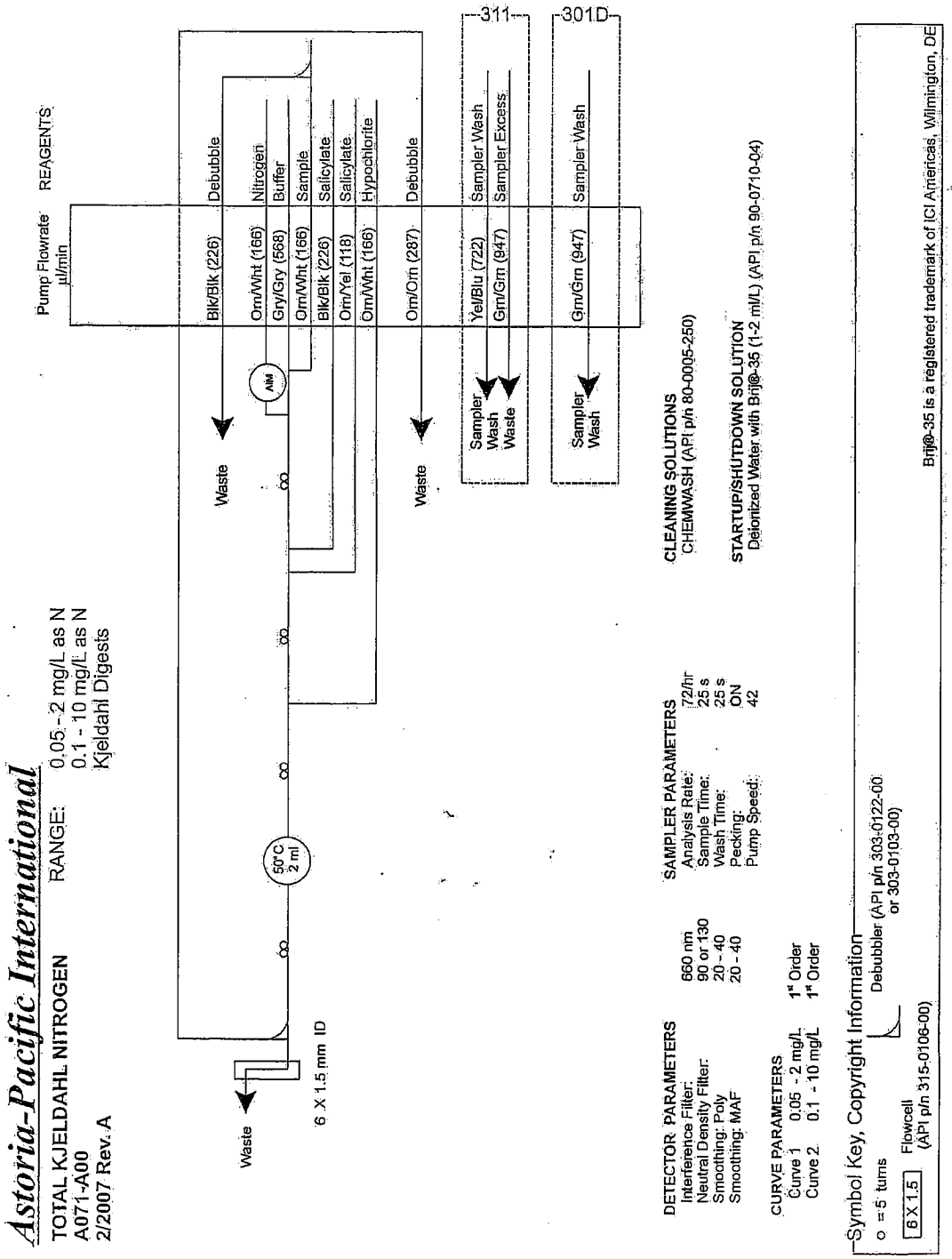
Attachment 1.

Flow Chart



Attachment 2.

Manifold Flow Diagram



Attachment 3.

Example Data Review Checklist

TESTAMERICA Denver

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Lot / Sample Numbers	Matrix	Batch	Method	QC	Special Inst

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Minimum of five standards in ICAL or as specified in method?				
2. Correlation coefficient ≥ 0.995 ?				
3. Second-source ICV analyzed, and recovery 90-110%?				
4. ICB analyzed immediately after the ICV & results < the RL				
5. CCV analyzed after every ten samples & recovery ± 10% of true value?				
6. CCB analyzed after every CCV & results < RL?				
7. Absolute value of the intercept is < ± ½ the RL?				
B. Sample Results				
1. All samples greater than highest calibration standard diluted and reanalyzed?				
2. Do associated RLs/MDLs reflect dilutions or limited sample volume?				
3. Are reported results bracketed by in control CCV results?				
4. Sample analyses done within holding time?				
5. Initial pH check documented for all samples?				
6. Preparation benchsheet completed and included in package?				
7. Client requirements reviewed and met?				
8. Were data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures and correct units?				
9. Do the prep and analysis dates in QuanTIMS reflect the actual dates? Lots/Dates report checked?				
10. Are all data being reported highlighted on the benchsheet?				
11. Raw data copies prepared and scanned?				
12. Manual integrations done properly and initialed and dated?				
13. STD/True Value sheet is updated and included?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10x blank have NCM?				
2. Method blank < ½ RL or NCM provided?				
3. LCS/LCSD run for batch and within QC limits?				
4. MS/MSD run at required frequency and within limits or NCM written?				
5. DUP run at required frequency and RPD within 20% or NCM written?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

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-Title: Ammonia Nitrogen by Autoanalyzer [EPA 350.1]

Approvals (Signature/Date):

Dave Elkin — Date 12/21/10
Wet Chemistry Supervisor

Adam Alban — Date 21 Dec 10
Health & Safety Manager / Coordinator

John P. Morris — Date 12/21/10
Quality Assurance Manager

Robert C. Hanisch — Date 12/21/10
Laboratory Director

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 This procedure describes the automated phenolate analysis of water samples for ammonia by EPA Method 350.1.

1.2 The reporting limit for ammonia is 0.1 mg/L.

1.3 This method covers the determination of ammonia in drinking, ground, surface, and saline water, domestic and industrial wastes.

2.0 Summary of Method

2.1 The automated phenolate method involves the reaction of alkaline phenol and hypochlorite with ammonia to form indophenol blue. The color is enhanced by the addition of sodium nitroprusside.

2.2 The intensity of the color at 660nm is proportional to the ammonia concentration.

3.0 Definitions

There are no terms requiring definition unique to this procedure. Refer to the Glossary of the QAM for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Residual chlorine will react with ammonia to form chloramines. Residual chlorine must be removed at the time of sample collection by pretreatment of the sample with sodium thiosulfate or other reagent before distillation.

4.2 Wastewater discharge monitoring and other Clean Water Act compliance testing requires either distillation or demonstration of equivalency between distilled and undistilled samples. If significant interferences are encountered, a preliminary distillation may be necessary.

4.3 Calcium and magnesium may precipitate as hydroxides or carbonates in alkaline solutions and clog the ammonia channel or cause turbidity problems. EDTA is added to minimize this effect.

4.4 Color and turbidity in the samples will interfere. If color or turbidity are observed the sample is filtered through GFC filter media. If color remains after filtration, the sample is diluted until the color absorption at 660nm is minimal.

4.5 Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out.

4.6 Method interference may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that biases analytical response.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 Sodium nitroferricyanide will generate hyrdogen cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness, and potentially death.

5.1.3 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.4 Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ammonium Chloride	Corrosive	10 mg/m^3 (TWA)	Causes irritation to respiratory tract; symptoms may include coughing, shortness of breath. Ingestion may cause nausea, vomiting, and diarrhea. Causes irritation to skin and eyes.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Hydroxide	Corrosive	2 mg/m3 (Ceiling)	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and with greater exposures, it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Sodium Nitroferricyanide	Poison	5 mg/m3 as HCN gas	May cause irritation in contact with the skin. Gives off HCN gas if combined with strong acids. Inhalation of HCN gas can cause irritation, dizziness, nausea, unconsciousness, and potentially death.
Phenol	Corrosive	5 ppm (TWA)	Breathing vapor, dust or mist results in digestive disturbances. Will irritate, possibly burn respiratory tract. Rapidly absorbed through the skin with systemic poisoning effects to follow. Discoloration and severe burns may occur, but may be disguised by a loss in pain sensation. Eye burns with redness, pain, blurred vision may occur. May cause severe damage and blindness.
EDTA	Flammable	None	Mild irritant to respiratory tract. Symptoms may include coughing and sneezing. Low toxicity by ingestion. Prolonged contact with skin may cause redness or inflammation.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

- Auto-analyzer equipped with the proportioning pump, autosampler, and colorimeter with 660 nm filters and 5-mm flow cell.
- Analytical balance, capable of accurately weighing to the nearest 0.0001 g. The accuracy of the balance is verified each day it is used in accordance with SOP DV-QA-0014.
- An all glass distilling apparatus (Kontes or WESTCo).

6.2 Supplies

- Volumetric Flasks (Class A): varying volumes
- Eppendorf Pipettes, varying volumes

6.3 Computer Software and Hardware

Please refer to the master list of documents and software/hardware located on G\QA\Read\Master List of Documents\Master List of Documents and Software/hardware.xls

7.0 Reagents and Standards

Reagents - All materials must be reagent grade or higher quality, unless otherwise specified

7.1 Reagent Water
Reagent grade deionized water, Milli-Q or equivalent. The water must have less than 0.1 mg/L ammonia.

7.2 Sulfuric Acid (H_2SO_4), 0.04 N
Dilute 1.0 mL of concentrated H_2SO_4 to 1 liter. This is the distillation collection solution.

7.3 Sulfuric Acid (H_2SO_4), 5 N:
Carefully add 139 mL of concentrated sulfuric acid to approximately 500 mL of reagent water. Cool to room temperature and dilute to 1 liter with deionized water. This is the air scrubber solution used in the autoanalyzer.

7.4 Sulfuric Acid, 0.2%
Slowly and with constant mixing add 4 mL concentrated sulfuric acid to 2 liters of de-ionized water.

7.5 Sodium Phenolate
Add 80 mL of 10 N sodium hydroxide to 700 mL of reagent water. Slowly add 83g of phenol (94 mL of liquid phenol) in small amounts. Bring to a final volume of 1 liter with deionized water. Store in the dark and prepare fresh every week.

7.6 Sodium Hypochlorite Solution (NaOCl)
Dilute 250 mL of a bleach solution containing 5.25% NaOCl (such as Clorox) to 500 mL with deionized water. Analysts must remain alert to detecting any variation in the formulation as the percentage of NaOCl changes in Clorox. Due to the instability of this product, solution should be prepared fresh daily.

7.7 Sodium Nitroferricyanide Solution
Dissolve 0.5 g of sodium nitroferricyanide in 1 liter of deionized water. Store in an amber container and prepare fresh monthly.

7.8 Dechlorinating reagents;

7.8.1 Sodium Thiosulfate ($Na_2S_2O_3$)
Dissolve 3.5g of $Na_2S_2O_3 \cdot 5H2O$ in reagent water and dilute to 1 liter.

7.8.2 Sodium Sulfite (Na_2SO_3)
Dissolve 0.9g of Na_2SO_3 in reagent water and dilute to 1 liter.

7.9 Brij-35 Solution
This solution is obtained from commercial sources.

7.10 Borate Buffer
Add 88 mL of 0.1 N NaOH solution to 500 mL of 0.025 M sodium tetraborate solution (5 g anhydrous $Na_2B_4O_7$ or 9.5 g of $Na_2B_4O_7 \cdot 10H_2O$ per liter) and dilute with reagent water. This solution is also available from commercial sources.

7.11 Sodium Hydroxide Solution, 1.0 N
Reagent grade solution is available from commercial sources. Alternatively, it is prepared by dissolving 40g of NaOH in reagent water and diluting to 1 liter.

7.12 Disodium EDTA
Add 50 g of disodium EDTA and 6 pellets of sodium hydroxide to 800mL of deionized water. Cool to room temperature and bring to a final volume of 1 liter. Add 20 drops or 1 mL of Brij-35 and mix.

7.13 Ammonia CAL Stock Standard, 1000mg/L
Dry ammonium chloride at 105 °C. Dissolve 3.819 g in 600 mL of deionized water, add 2 mL of concentrated sulfuric acid, and dilute to 1000 mL with deionized water. This solution is normally obtained from commercial vendors also.

7.14 Ammonia ICV Stock Standard, 1000 mg/L
Dry ammonium chloride at 105 °C. Dissolve 3.819 g in 600 mL of deionized water, add 2 mL of concentrated sulfuric acid, and dilute to 1000 mL with deionized water. This solution is normally obtained from commercial vendors also. This solution should be prepared from a separate source from that used for the CAL standard.

7.15 Intermediate CAL Standard, 100 mg/L
Pipette 10 mL of stock standard into a 100 mL volumetric flask, dilute to volume with distilled water and add 2 drops concentrated sulfuric acid.. This standard can also be purchased from a commercial vendor.

7.16 Intermediate ICV Standard, 100 mg/L
Pipette 10 mL of stock standard into a 100 mL volumetric flask, dilute to volume with distilled water, and add 2 drops of concentrated sulfuric acid. This standard can also be purchased from a vendor.

7.17 Working Calibration Standards
Prepare dilutions as indicated below. These dilutions can be made by the auto-analyzer system. All dilutions are made with distilled water and are remade daily. The following table shows suggested concentrations:

Volume of Intermediate Standard (mL)	Final Volume (mL)	Concentration (mg/L as N)
0.05	100	0.05
0.10	100	0.10
0.50	100	0.50
1.0	100	1.0
5.0	100	5.0
10.0	100	10.0

NOTE: If nitrate/nitrite is being run at the same time as ammonia, a mixed intermediate and mixed calibration standards may be prepared that contain both ammonia and nitrate at the above concentrations.

7.18 Initial Calibration Verification Standard (ICV)
This is a second-source standard, different vendor than the calibration standards. It is prepared at a concentration of 2.0 mg/L by pipetting 2.0 mL of the 100 mg/L intermediate ICV standard and diluting to 100 mL with distilled water. This solution must be remade daily.

7.19 Laboratory Control Sample (LCS)
The LCS is identical to the 5.0 mg/L standard prepared as a working calibration standard. The LCS for soil samples is prepared by adding 1 mL of the Cal Stock Standard 1000 mg/L (7.13) to 10 g of solid matrix (Ottawa sand, boiling chips, glass beads, etc.).

7.20 Matrix spike/matrix spike duplicate (MS/SD)
The Matrix Spike and Matrix Spike Duplicate (MS/MSD) samples are prepared by adding 0.20 mL of the Intermediate CAL Standard, 100 mg/L(7.15) to 4.8 mL of sample. The true value of the spike is 4.0 mg/L. The MS/MSD for soil samples is prepared by adding 1 mL of the Cal Stock Standard, 1000 mg/L (7.13) to 10 g of sample.

7.21 Kleenflow Solution - Acidic
Add 50 mL of Isopropyl Alcohol to a 1 liter flask. Carefully add approximately 950 mL of 1N Hydrochloric Acid (HCL).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass	1 Liter	H_2SO_4, pH < 2; Cool 4 ± 2°C	28 Days	40 CFR Part 136.3
Soil	Glass	4 oz	Cool 4 ± 2°C	28 Days	N/A

NOTE: Samples collected for ammonia nitrogen must be dechlorinated in the field. 1 mL of sodium thiosulfate or sodium sulfite solution removes 1 mg/L of residual chlorine per 500 mL of sample. If necessary, neutralize to approximately pH 7 with dilute acid or base, using a pH meter.

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

9.1.1 Method Blank

A method blank of deionized water is required with every batch of 20 or fewer samples. The MB consists of reagent water that is carried through the entire analytical procedure, including preparation and analysis. When analyzing soils, the MB is prepared in the same manner as the samples by a solid matrix (Ottawa sand, boiling chips, glass beads, etc.). The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

Acceptance Criteria: The blank result must be < ½ RL, although samples more than ten times the blank concentration may be reported with a footnote or narrative comment.

Corrective Action: If the blank criterion is not met, the source of the contamination should be investigated and corrected. All associated samples should be reanalyzed or the data evaluated and reported with the appropriate qualifiers.

9.1.2 Laboratory Control Sample (LCS)

One LCS sample is required for each batch of 20 or fewer samples. The LCS for aqueous sample batches consists of reagent water to which a known amount of target analyte has been added. The LCS for soil batches consists of 10 grams of a solid matrix (Ottawa sand, boiling chips, glass beads, etc.)

to which a known amount of the target analyte is added. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Preparation of the LCS is described in Section 7.19.

Acceptance Criteria: The recovery for the LCS must be within the laboratory's statistical (three standard deviations) control limits, not wider than 90-110%.

Corrective Action: Samples analyzed along with an LCS that is determined to be "out of control" are considered suspect and the samples must be reprocessed and reanalyzed, or the data reported with appropriate data qualification. If the LCS result does not fall within statistical control limits, check calculations, check instrument performance, reanalyze the LCS, and if still outside of control limits, re-prepare and reanalyze all samples in the QC batch. It is acceptable to report the data if the LCS recovery is out high and any analyte of concern was not detected in any of the samples.

9.1.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Samples

An MS/MSD pair is required for every 10 or fewer samples. Preparation of matrix spikes and matrix spike duplicates is described in Section 7.20.

Acceptance Criteria: The recovery for each matrix spike must be within the laboratory's statistical (three standard deviations) control limits, not wider than 90-110%. The relative percent difference (RPD) for the MS/MSD pair must be within 10%.

Corrective Action: If these limits are not attained, associated sample results must be qualified on the final report. Check special program or project requirements on this point because some require additional corrective actions.

NOTE: Some programs (North and South Carolina) require spiking at 10% rate and a spike duplicate is not sufficient for these programs. Refer to client requirement for these special instructions.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV)

The result for a second-source calibration verification standard must be within ± 10% of the expected value. If this is not attained, reprepare the initial calibration standards and/or optimize the instrument and recalibrate.

9.2.2 Continuing Calibration Verification (CCV)

A mid-point calibration standard is analyzed after every ten samples and at the end of the run. The results for the CCVs must be within ± 10% of the expected value. If routine corrective action fails to produce a second consecutive (immediate) CCV within acceptance limits, than two consecutive successful CCVs need to be performed or the instrument should be recalibrated and all samples tested since the last successful CCV reanalyzed.

NOTE: If a quadratic curve is used 2 CCV concentrations must be performed every 10 samples. The concentrations used will be 1.0 mg/L and 5.0 mg/L.

9.2.3 Continuing Calibration Blank (CCB)
A deionized water blank is analyzed after every ten samples and at the end of the run. The results for the CCBs must be less than 0.1 µg/L. If the CCB results are over this limit, maintenance must be performed to decontaminate the system and all samples run since the last successful CCB reanalyzed.

9.2.4 Linear Calibration Range (LCR)
The LCR must be determined initially and verified every 6 months or whenever a significant change in instrument response is observed or expected. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ± 10%, the linearity must be re-established.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 The pH of all samples are checked in log-in at the time of receipt. If the pH is above 2, results may not be acceptable – A Condition Upon Receipt (CUR) form is completed to notify the laboratory Project Manager. Because results for improperly preserved samples may be biased low, analysis should not proceed without authorization and the final report must qualify the result.

10.1.2 The laboratory **does not** distill all water samples for Method 350.1, but will perform as requested by the client. If distillation is requested the sample will be logged with the distillation prep code.

10.1.3 The laboratory distills **all** soil samples for method 350.1.

10.1.4 Sample Distillation

10.1.4.1 Test sample for residual chlorine with KI paper. If residual chlorine is detected, it means that the sample was not collected properly and the laboratory Project Manager must be notified. The client should then be consulted to determine how to proceed. If the client gives the order to proceed, an NCM must be prepared and the final report case narrative must describe the problem.

10.1.4.2 Add 8 mL of borate buffer to the sample adjusted to pH 9.5 and transfer to a 500 mL distillation flask

10.1.4.3 Add 1 N NaOH, until the pH is 9.5. Check the pH during the addition with a pH meter or by use of a short-range pH paper.

10.1.4.4 Place 200 mL of sample in a distillation flask and set up distillation apparatus.

10.1.4.5 For soil samples, place 10 g of soil in a distillation flask, add 200 mLs DI water and set up distillation apparatus. Then add the borate buffer (Section 7.10) until the pH is 9.5

10.1.4.6 Distill at a rate of 6-10 mL/min. Distill ammonia into 50 mL of 0.04 N H2SO4. Collect at least 100 mL of distillate. Dilute distillate to 200 mL with reagent water.

10.2 Calibration

10.2.1 Instrument Set-up

Follow the specific instrument set up directions from the equipment vendor.

10.2.2 Initial Calibration

The instrument is calibrated at six concentration levels from 0.05 to 10.0 mg/L (see Section 7.17). A calibration curve is constructed using a linear least squares regression. A minimum correlation coefficient of $r \geq 0.995$ is required. If this is not met, reprepare standards and/or optimize the instrument and recalibrate.

10.2.3 If a linear squares regression is not the best curve fit, alternatively a quadratic curve fit may be used (2^{nd} order curve). The instrument software uses a quadratic regression to relate the concentration of ammonia in each standard and the associated absorbance reading, as follows:

$$x = cyy + by + a \qquad \textbf{Equation 1}$$

Where:

x = *analyte concentration*
y = *analyte height*
c = *definition of the curve*
b = *Slope.*
a = *Y-intercept.*

10.2.4 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

10.3 Sample Analysis

10.3.1 Samples are loaded on the autosampler, processed. The instrument data system prints a report of results calculated in mg/L.

10.3.2 Samples with results exceeding the highest calibration standard are diluted and rerun. Appropriate dilutions produce results in the upper one-half of the calibration range.

10.3.3 Following is a typical analytical sequence:

ICAL and/or ICV and ICB

LCS and LCSD
Method Blank
7 injections
5 mg/L CCV, (1 mg/L CCV if 2nd order) and CCB
10 injections
5 mg/L CCV, (1 mg/L CCV if 2nd order) and CCB
10 injections
5 mg/L CCV, (1 mg/L CCV if 2nd order) and CCB

10.3.4 Disconnect the reagent lines and put all of them into DI water, EXCEPT the buffer line. The buffer line should be placed into bridge solution.

10.3.5 Rinse all tubes for at least 5 minutes, and then rinse with Kleenflow Base for 5 minutes. Rinse all tubes with the appropriate rinses for another 5 minutes.

10.3.6 Turn off the instrument and pump, rinse platens, and empty waste when finished.

11.0 Calculations / Data Reduction

11.1 Accuracy

ICV / CCV, LCS % Recovery = observed concentration x 100

known concentration

MS % Recovery = (spiked sample) - (unspiked sample) x 100

spiked concentration

11.2 Precision (RPD)

Matrix Duplicate (MD) = |orig. sample value - dup. sample value| x 100

[(orig. sample value + dup. sample value)/2]

11.3 Concentration

$$\text{Ammonia (as mg/L N)} = \frac{(C \times DF)}{V}$$

Where:

C = Concentration measured at the instrument
DF = Dilution factor
V = Volume of sample used for analysis

- Normally this calculation is performed automatically by the instrument data system.

- If an extract of a non-aqueous sample was analyzed, multiply the result obtained by the prep dilution factor to obtain the final result.

11.4 See Attachment 1, "Wet Chemistry Review Checklist for Tests with Calibration Curves," for first and second-level data review documentation requirements.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 Method Detection Limit Study an initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Prepare and analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P) for each analyte.
- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy # DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

- Initially the analyst must perform an MDL study (see section 12.1).
- Four aliquots of the QC check sample (independent source from the calibration) are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Distillation waste – Aqueous Alkaline - Waste Stream E
- Distillate and Instrument Waste – Aqueous Acidic - Waste Stream F

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 EPA 350.1, Determination of Ammonia Nitrogen by Semi-Automated Colorimetry, Revision 2.0, August 1993

15.2 Alpkem manual for Enviroflow 3500 (P/N 000156 & P/NO 00157)

16.0 Method Modifications:

Item	Method	Modification
1	EPA 350.1	Method 350.1 requires all samples to be distilled. The laboratory only distills samples as requested by the client.
2	EPA 350.1	Method 350.1 states to place the calibration standards in the sampler "in order of decreasing concentration." The standards are placed in the sampler in increasing concentration per the instrument manufacturer's specifications.

17.0 Attachments

Attachment 1: Example Data Review Checklist

18.0 Revision History

- Revision 6, dated 23 December 2010
  - Added soil matrix to Section 8.0
  - Updated Sections 7.19 and 7.20 to include soils preps.
  - Updated Sections 10.1.3 and 10.1.4.5 to include references to soil samples.

- Revision 5.2, dated 19 November 2010
  - Added reagent quality requirements to section 7.0

- Revision 5.1, dated 24 June 2010
  - Annual Technical Review.

- Revision 5, dated 24 August 2009
  - Deleted section 4.5 due to all samples, standards and rinse water use DI water not acidified solutions.
  - Deleted 0.2% sulfuric acid – not used in standard preparation
  - Added Kleenflow recipe to section 7.
  - Adjusted the Intermediate standards (CAL and ICV) to be brought to volume with DI water.
  - Added the use of Ottawa sand to the LCS and MB.
  - Changed pH check to be performed by log-in (section 10.1.1).
  - Added sections 10.3.4 – 10.3.6.
  - Fixed the Concentration calculation.
  - Updated the checklist attachment.

- Revision 4, dated 29 May 2009
  - Updated minor formatting and grammatical errors.
  - Changed the preparation time for Sodium Phenolate from monthly to weekly.
  - Changed the concentrations of the calibration standards.
  - Changed the concentration for the LCS.
  - Changed the CCV concentrations.
  - Added 2nd CCV concentration requirement for quadratic curves.
  - Added reference to corporate calibration SOP (section 10.2.4).
  - Added the performance of an MDL study for IDOC.

- Revision 3, dated 14 March 2008
  - Integration for TestAmerica and STL operations.
  - Updated formatting.

- Updated method references.
- Technical review and update throughout the SOP.

- Revision 2.0 and 2.1, dated 30 August 2002 and 21 November 2006
  - For the minor revision, the technical content of this SOP was not reviewed or updated. The purpose of this minor revision is to incorporate the Safety Bulletin information to ensure compliance with STL Corporate requirements. In addition, Section 9.1 was revised to reference Policy QA-024 for specific QC requirements for federal programs, any existing interim changes were incorporated, and formatting was updated consistent with Policy QA-001.

- Revision 1.0, dated 15 March 2002
  - Updated standards including ability to make mixed nitrate/ammonia standards, and added details for preparing ICV standards.
  - Changed Quanterra to STL.
  - Added requirement that hypochlorite solution be made daily.
  - Added Appendices
  - Updated pollution prevention and waste sections.

Attachment 1.

Example Data Review Checklist

TESTAMERICA Denver

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Lot / Sample Numbers	Matrix	Batch	Method	QC	Special Inst

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Minimum of five standards in ICAL or as specified in method?				
2. Correlation coefficient ≥ 0.995 ?				
3. Second-source ICV analyzed, and recovery 90-110%?				
4. ICB analyzed immediately after the ICV & results < the RL?				
5. CCV analyzed after every ten samples & recovery ± 10% of true value?				
6. CCB analyzed after every CCV & results < RL?				
7. Absolute value of the intercept is < ± ½ the RL?				
B. Sample Results				
1. All samples greater than highest calibration standard diluted and reanalyzed?				
2. Do associated EDLs/MDLs reflect dilutions or limited sample volume?				
3. All reported results bracketed by in control CCV results?				
4. Sample analyses done within holding time?				
5. Initial pH check documented for all samples?				
6. Preparation benchsheet completed and included in package?				
7. Client requirements reviewed and met?				
8. Were data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures and correct units?				
9. Do the prep and analysis dates in QuanTIMS reflect the actual dates? Lots/Plates reported checked?				
10. Are all data being reported highlighted on the benchsheet?				
11. Raw data copies prepared and scanned?				
12. Manual integrations done properly and initialed and dated?				
13. STD True Value sheet is updated and included?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10x blank have NCM?				
2. Method blank < ½ RL or NCM provided?				
3. LCS/LCSD run for batch and within QC limits?				
4. MS/MSD run at required frequency and within limits or NCM written?				
5. DUP run at required frequency and RPD within 20% or NCM written?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

TestAmerica
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TestAmerica
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Title: Total Organic Carbon in Soil [SW 9060]

Approvals (Signature/Date):

Dave Elkin — Date: 5/3/10
Wet Chemistry Supervisor

Adam Alban — Date: 04 May 10
Health & Safety Manager / Coordinator

Karen Kuoppela — Date: 5-4-10
Quality Assurance Manager

Robert C. Hanisch — Date: 5/4/10
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This procedure describes the determination of total organic carbon in soils, sludge, and sediments using the LECO C632 TOC analyzer. The LECO instrument uses 0.20 gram quantities of sample, and so results are less prone to precision problems that are typical of the trace TOC instruments that use sample aliquots in the 10-100 mg range. The method referenced for this procedure is EPA Method 9060.

1.2 The reporting limit (RL) is 0.2% carbon or 2,000 mg/kg.

2.0 Summary of Method

The sample is treated with 6N (1:1) HCL to drive off inorganic carbonates and then dried to remove moisture and acid. Organic carbon in the sample is converted to carbon dioxide (CO_2) by catalytic combustion. The CO_2 formed is measured by an infrared detector. The amount of CO_2 is directly proportional to the concentration of carbonaceous material in the sample.

3.0 Definitions

Total Organic Carbon (TOC):

The carbon measured as a result of oxidation of the sample after the removal of inorganic carbon.

4.0 Interferences

4.1 Oily samples will cause erratic results. This is minimized by homogenization of the sample.

4.2 Due to the initial purging step to remove inorganic carbon, purgeable organic carbon compounds are not effectively analyzed by this procedure.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Spent crucibles must be allowed to cool to room temperature prior to disposal.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the

method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- LECO C632 Analyzer.
- Hot Plate. The temperature must be sufficient to drive off water and HCl and completely dry the samples

6.2 Supplies

- Porcelain Combustion Boats

  NOTE: Porcelain Combustion Boats are a radioactive material.
- Small Beakers
- Spoons or spatulas
- Miscellaneous volumetric glassware

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

7.1 6N Hydrochloric Acid (1:1)

Slowly and carefully and with stirring, add 500 mL of concentrated HCL to 500 mL of deionized water. Allow to cool before use.

7.2 TOC Calibration Standard (MS/SD/CCV)

Low Level: Calcium Carbonate; 12.00%C
High Level: Potassium Biphthalate, 47.05%C

7.3 TOC Initial Calibration Verification (ICV) This standard is from a different source than that of 7.2.

Low Level: Calcium Carbonate; 12.00%C
High Level: Potassium Biphthalate, 47.05%C

7.4 TOC Calibration Standard (LCS)

This standard is purchased from an outside vendor. The true value will be dependent on the lot received.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils	Glass	4 oz	Cool 4 ± 2°C	N/A	N/A

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

A batch is a group of no greater than 20 samples excluding QC samples (LCS, MS, MSD, Method Blanks), which are processed similarly, with respect to the procedure. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.3 Method Blank (MB)

One method blank (MB) must be processed with each batch. The method blank consists of a solid blank matrix containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is

used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

Acceptance Criteria: The method blank should not contain any analyte of interest at or above the reporting limit.

Corrective Action: If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are re-prepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data must be taken in consultation with the client and must be addressed in the project narrative.

If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the project narrative.

If all samples associated with a blank greater than the RL are greater than 10 times the blank value, the samples may be reported with an NCM to qualify the high blank value.

9.4 Laboratory Control Sample (LCS)

One LCS must be processed with each batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

The LCS for TOC in soils is performed by analyzing a 0.20 g aliquot of a purchased standard.

Acceptance Criteria: The LCS recovery must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean. The control limits are maintained in the LIMS.

Corrective Action: If any analyte is outside established control limits, the system is out of control, and corrective action must occur. Corrective action will be re-preparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.5 Matrix Spike and Matrix Spike Duplicate (MS/MSD) Samples

One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) that is prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to an MS/MSD pair. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked.

The MS and MSD for the automated method are prepared by placing 0.20 g of the soil sample to be spiked into a porcelain boat and adding an identical weight of calcium carbonate. These are mixed and combusted as a sample with the weight of the soil (0.20 g) used as the sample weight in the sample table.

Acceptance Criteria: The recovery of the analyte in the MS and MSD must fall within established control limits, which are set at ± 3 standard deviations around the historical mean. The relative percent difference between the MS and MSD must be no greater than the established RPD limit, which is based on historical data.

Corrective Action: If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include re-preparation and reanalysis of the batch.

If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the MS/MSD RPD limits.

9.6 Initial Calibration Verification (ICV)

The ICV standard is analyzed immediately following the ICAL. The ICV is a second-source calcium carbonate standard with a true value of 12% carbon. The analyte recovery must fall within the 90-110% range. If it is outside the acceptance limits, check the equipment and standards, correct any problems, and then recalibrate.

9.7 Continuing Calibration Verification (CCV)

The calibration is checked at the beginning of an analytical sequence (ICV), after every ten samples (CCV), and at the end of the sequence (CCV) by measuring a CCV standard.

The CCV is calcium carbonate with a true value of 12% carbon.

The CCV recovery must be within the 90-110% range. If it is outside the acceptance limits, check the equipment and standards, correct any problems, recalibrate, and rerun all samples analyzed since the last successful CCV.

9.8 Initial and Continuing Calibration Blank (ICB and CCB)

System cleanliness is checked at the beginning of an analytical sequence (ICB), after every ten samples (CCB), and at the end of the sequence (CCB) by analyzing a blank.

The ICB/CCB for the automated method is a solid sample matrix.

Results must be less than the reporting limit. If the blank result is greater than the reporting limit, check for carry-over from high level samples, clean the system, recalibrate, and rerun all samples analyzed since the last successful CCB.

10.0 Procedure

One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 Samples should be homogenized, and ground to uniform consistency if necessary. Leave out any extraneous artifacts, i.e., glass shards, large twigs, leaves, etc.

10.1.2 In a small beaker, aliquot approximately 2-3 g of sample. Slowly add 6N HCl until sample is completely moistened. If sample fizzes excessively, more 6N HCl may be needed.

10.1.3 Heat samples on a hot plate until they appear dry; then dry in oven at 104°C for at least one hour.

10.2 Calibration

10.2.1 Instrument and furnace should be left on at all times. Be sure that the furnace is reheated to 1350°C before beginning analysis.

10.2.2 If the furnace has been shut down due to maintenance or a power failure, ramp the temperature up slowly to 600°C to minimize the thermal stresses on the combustion tube.

10.2.3 Turn on the compressed air to the autosampler and the oxygen to the combustion analyzer.

10.2.4 Check that the incoming oxygen pressure is 20-40 psi and the combustion pressure is <15 psi.

10.2.5 Initial Calibration: The LECO analyzer is calibrated with calcium carbonate, a solid with a true value of 12% carbon and blanks.

10.2.5.1 Analyze three blanks

10.2.5.2 Analyze a five point standard curve (0.05g, 0.010g, 0.075g, 0.15g, and 0.25g) with the 12% carbon standard.

10.2.6 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

10.3 Sample Analysis

10.3.1 Weigh 0.20 g of dried sample into a tared porcelain weighboat. Spread the sample evenly throughout the boat. Transfer the exact sample weight into the sample table and load the boat into the autosampler.

10.3.2 When samples are loaded, use the software to begin analysis.

10.3.3 Sample results should be less than 20% carbon so that they use the calibrated low-range IR cell. Sample results of greater than 20% carbon should be reanalyzed with a smaller aliquot.

11.0 Calculations / Data Reduction

Results are reported from the instrument as a final concentration expressed as % carbon. Multiply by 10,000 to convert to mg/kg. Samples less than 0.2% are reported as ND.

11.1 Accuracy

$$\textbf{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\textbf{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\textbf{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Concentration = $\frac{\text{mg/kg or L} = C \times V \times D}{W}$

Where:

C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4 Control limits are stored in and accessed from LIMS.

11.5 The detection limit for this method will vary based on the results of detection limit studies performed. Samples less than the method detection limit are reported as ND. Please refer to LIMS for current reporting limit information.

11.6 First and second level data reviews are recorded on the Wet Chemistry Data Review Checklist shown in Attachment 1.

11.7 Additional calibration calculation information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method Detection Limit Study an initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Prepare and analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then,

calculate the MDL (single-tailed, 99% confidence level, as described in Policy DV-QA-005P) for each analyte.

- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

- Four aliquots of the QC check sample (independent source from the calibration) are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs (independent source from the calibration), or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produce when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Acidic waste – Waste Stream F
- Porcelain Combustion Boats: Contact Radioactive Waste Coordinator

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

- Method 9060, "Total Organic Carbon", SW-846, Third Edition, 9/86.

16.0 Method Modifications:

Item	Method	Modification
1	SW 9060	Method 9060 is designed for water samples, and requires quadruplicate analysis to overcome potential precision problems. This procedure is exclusively for soil samples, and the LECO instrument is designed for soil analysis. The sample aliquots are 10-100 times larger than are practical with most other non-dispersive IR instruments, and so the precision is acceptable with single analyses.
2	SW 9060	Method 9060 requires the use of a blender to homogenize samples. Since this procedure is for soils, the samples are ground to a uniform consistency.

17.0 Attachments

Attachment 1: Example Data Review Checklist

18.0 Revision History

Revision 3.1, dated 15 May 2010

- Annual Review
- Added section 6.3

- Revision 3, dated 15 May 2009
  - Changed aliquot size from 0.25g to 0.20g throughout the SOP.
  - Changed LCS to reflect it is a purchased standard.
  - Deleted Methanol from the safety section.
  - Modified the oven temperature in section 10.1.3 to 104°C.
  - Changed section 11.5 to read samples less than the MDL are reported as ND.
  - Updated calibration section 10.2 to reflect current practices.

- Revision 2, dated 04 April 2008
  - Integration for TestAmerica and STL operations.

- Revision 1, dated 08 August 2007
  - Method was changed to be specific to the LECO C632 instrument. Sample aliquot was changed from 0.5g to 0.25g at manufacturer's recommendation.

Attachment 1.

Example Data Review Checklist

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

WET CHEMISTRY COVERSHEET
Revision 6 – 8/8/07

*ANALYTE:*_____ *ANALYST:*_____ *ANALYSIS DATE:*

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

1) Special instructions followed (obtained if necessary)?		Y	N/A
2) Prep sheet complete and reviewed?		Y	N/A
3) Blanks, DCSs and CCVs within limits or properly anomalized?		Y	N/A
4) DCS limits:_____ , RPD limits:_____			
5) (if applicable) MSQC limits:_____ , RPD limits:_____		N/A	
6) Client specific criteria met?	Y	N/A	
7) Chromatograms, tapes, printouts checked?		Y	N/A
8) Significant figures reported correctly?	Y	N/A	
9) Reporting limits reflect dilutions and/or limited sample volume?		Y	N/A
10) Comments, exceptions, anomalies and SOP/method variances recorded?		Y	N/A
11) Holding times met; if not, NCM form submitted, PM/PM notified and form copied and included?	Y	N/A	
12) Rework documented; original and rework data copied and included?	Y	N/A	
13) All data being reported highlighted in blue on benchsheets?		Y	N/A
14) All data (including QC) entered into LIMS and checked against the data review printout?	Y	N/A	
15) All MS/MSD data defaulted into LIMS?		Y	N/A
16) Completed dates in LIMS reflect actual prep, reprep or analysis dates?		Y	N/A
17) Run log/benchsheet and maintenance entries entered in instrument logbook?	Y	N/A	
18) Were scheduled required due dates met?		Y	N
If not, why?:_____			

*Analyst:*_____ *Date:*

1) Calculations and chromatograms checked?		Y	N/A
2) Correlation coefficient is 0.995 or greater?		Y	N/A
3) All QC (blanks, DCSs, CCVs, LCBs) within limits or properly anomalized?		Y	N/A
4) Significant figures and reporting limits correct?		Y	N/A
5) NTVs (NCM form), rework, comments and anomalies documented and included?	Y	N/A	
6) Data checked against data review printout?		Y	N/A
7) For soils, is dry-weight flag set correctly in QuantIMS (run query)?		Y	N/A
8) Project and raw data copies prepared and filed?	Y	N/A	
9) All raw data copies legible?	Y	N/A	

*Reviewed by:*_____ *Date:*

Comments:

QA/Forms/Benchst/WC/ BS0022.DOC

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TestAmerica
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Title: Color
[EPA 110.2, SM 2120B]

Approvals (Signature/Date):

Dave Elkin — Date 5/3/10
Wet Chemistry Supervisor

Adam Alban — Date 04 May 10
Health & Safety Manager / Coordinator

Karen Kuoppala — Date 5-4-10
Quality Assurance Manager

Robert C. Hanisch — Date 5/4/10
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This method is used for measuring the color of water derived from naturally occurring materials, i.e., vegetable residues such as bark, roots, humus and peat materials. This method is NOT applicable to color measurement on waters containing highly colored industrial wastes.

1.2 This method is usable in the 5 to 50 unit range. Higher level samples can be analyzed by dilution of the sample.

2.0 Summary of Method

2.1 Color is measured by visual comparison of a sample to platinum-cobalt standards.

2.2 One unit of color is that produced by 1 mg/L platinum in the form of chloroplatinate ion.

3.0 Definitions

There are no terms requiring definition unique to this procedure. Refer to the Glossary of the QAM for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Even slight amounts of turbidity will interfere. Samples with turbidity may be clarified by centrifugation or filtration. If turbidity is removed, the results are reported as "true color." Otherwise, the results are reported as "apparent color."

4.2 The method is pH dependent.

4.3 Absorption of ammonia by the standards will cause an increase in color.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm - ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat and upper respiratory tract and in severe cases, pulmonary edema, circulatory failure and death. Can cause redness, pain and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Balance: analytical, capable of accurately weighing to the nearest 0.0001 g. The balance accuracy is verified each day of use in accordance with SOP DV-QA-0014.

6.2 Glassware: Class A volumetric flasks and pipettes as required.

6.3 Nessler tubes, matched tall form, 50 mL capacity.

6.4 Rack for Nessler tubes.

6.5 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

7.1 Chloroplatinate Stock Standard, 500 units

7.1.1 To a 1000 mL volumetric flask containing approximate 500 mL of deionized water, slowly add 100 mL concentrated hydrochloric acid. Dissolve 1.246 g of potassium chloroplatinate (K2PtCl6) and 1.0 g of cobaltous chloride monohydrate (CoCl2•H20) in this mixture and dilute to 1000 mL with deionized water.

7.1.2 Alternatively, a commercially prepared standard may be used and diluted to the appropriate concentrations.

7.2 Working Standards

7.2.1 In the 50 mL Nessler tubes, prepare the following standards:

Volume Stock Standard (7.1)	Final Volume	Color Units
0.0 mL	50 mL	0
0.5 mL	50 mL	5
1.0 mL	50 mL	10
1.5 mL	50 mL	15
2.0 mL	50 mL	20
2.5 mL	50 mL	25
3.0 mL	50 mL	30
3.5 mL	50 mL	35

4.0 mL	50 mL	40
4.5 mL	50 mL	45
5.0 mL	50 mL	50

7.3 Laboratory and commercially prepared stock standards are stored at room temperature. Laboratory prepared stock standard is assigned an expiration of one year or less, and the manufacturer's expiration for commercially prepared stock standard is observed. Working standards are assigned an expiration of no more than 6 months from preparation. Working standards are stored in the Nessler tubes tightly stoppered.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	HDPE or Glass	500 mLs	Cool 4 ± 2°C	48 Hours	40 CFR Part 136.3

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Method Blank (MB)

9.1.1 The laboratory must analyze at least one MB with each batch of 20 samples. Data produced are used to assess contamination from the laboratory environment.

Acceptance Criteria: The MB result must be less than ½ the reporting limit or less than one-tenth of the concentration measured in samples.

Corrective Action: If the method blank exceeds these levels, the problem should be investigated and all associated samples reanalyzed. If elevated blank levels are encountered and color is not detected in samples, it may be possible to report the sample results together with an NCM.

9.2 Laboratory Control Sample (LCS)

9.2.1 There is no LCS associated with this method.

9.3 Sample Duplicate

9.3.1 The laboratory must analyze at least one sample duplicate with each batch of 20 samples. The Relative Percent Difference (RPD) of the sample duplicates must be within QC limits.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.1 Calibration

This method relies on the visual comparison of the color of a sample to the color of each of 11 standard solutions, which are prepared as described in Section 7.0.

10.2 Sample Analysis

If the samples are turbid, centrifuge until the supernatant liquid is clear. Up to one hour may be required. Alternately, samples may be filtered to remove turbidity. Samples with turbidity removed are reported as "true color" instead of "apparent color".

Fill a Nessler tube to the mark with sample and place the tube into the rack between the two standards with the most similar color values. Look vertically downward through the tube, on a white surface, to determine which standard the sample most closely resembles. The sample turbidity should be low enough that it is possible to see down the entire length of the tube to the reflective surface. Be certain to consider only sample color, not any slight darkening from any remaining sample turbidity.

Dilute any samples with more than 50 units of color and reanalyze.

11.0 Calculations / Data Reduction

Multiply the observed color by any dilution made to bring the color below 50 units.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

- There is no MDL study for color.

12.2 Demonstration of Capabilities

- Each analyst performing the method must complete a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.
- Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Contents of Nessler tubes – Aqueous Acidic (Reactivity) - Waste Stream F

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Method Source: Methods for the Chemical Analysis of Water and Wastes, EPA Method 110.2, 600/4-79-020

15.2 Method 2120B: "Standard Methods for the Examination of Water and Wastewater", 20th Edition.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 110.2	The upper standard prepared is 50 units.

17.0 Attachments

Attachment 1: Example Laboratory benchsheet
Attachment 2: Example Data Review Checklist

18.0 Revision History

- Revision 2.2, dated 15 May 2010
  - Annual Review
  - Added section 6.5

- Revision 2.1, dated 15 May 2009
  - Technical review performed.
  - Updated formatting.
  - Updated method and SOP references.
  - Updated the attachments.

- Revision 1, dated 07 July 2007
  - Company name was changed from STL to TestAmerica .

Attachment 1.

Example Laboratory Bench Sheet

TestAmerica Denver

Laboratory Bench Sheet
Color (Methods 110.2, SM 2120 E)
Revision 2 – June 28, 2007

Analyst:	Date:	SOP # DEN-WC-0058 Rev. 0.1

Calibration Standard Information

Source/Verification Lot#: _____ Prep Date: _____ Made by: _____

Concentration: _____(mg/L or ug/L) Expiration Date: _____

Sample	pH	Observed Color	Dilution Factor	Turbidity Removed*?	Final Color
Blank				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	
				Yes No	

**Note: If turbidity is removed, the results are reported as "true color". Otherwise, results are reported as "apparent color".*

Comments:_____

Attachment 2.

Example Data Review Checklist

TestAmerica Denver

Wet Chemistry Data Review Checklist
Direct Measurement Methods (pH, Conductance, etc.)

Test Name/Method #: _____ **Analysis Date** _____

SOP #: _____ **Analyst:** _____ **Instrument:** _____

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

	Yes	No	N/A	2nd Level
A. Calibration/Instrument Run QC				
1. Was the instrument properly standardized?				
2. Second-source ICV analyzed immediately after instrument standardization & recovery ± 10% of true value?				
3. ICB analyzed immediately after ICV & results < the RL?				
4. CCV analyzed after every ten samples & recovery ± 10% of true value?				
5. CCB analyzed after every CCV & all results < the RL?				
B. Sample Results				
1. Are all sample dilutions appropriate and do associated RLs reflect required dilutions or limited sample volumes?				
2. All reported results bracketed by in control CCV results?				
3. Sample analyses done within holding time?				
4. Preparation benchsheet completed and included in package (if applicable)?				
5. Special client requirements met?				
6. Was data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures?				
7. Do the prep and analysis dates in QuanTIMS reflect the actual dates?				
8. Are all data being reported highlighted on the benchsheet?				
9. Raw data copies prepared and scanned?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10x blank ?				
2. LCS run for batch and within QC limits?				
3. Sample DUP run at required frequency and RPD within established limits?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

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Phone: 303-736-0100
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Title: Hardness by Titration [EPA 130.2 and SM 2340C]

Approvals (Signature/Date):

Dave Elkin — 5/3/10 Date
Wet Chemistry Supervisor

Karen Kuoppala — 5-4-10 Date
Quality Assurance Manager

Adam Alban — 04 May 10 Date
Health & Safety Manager / Coordinator

Robert C. Hanisch — 5/4/10 Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This method is applicable to drinking, surface and saline waters, and domestic and industrial wastes by EPA method 130.2 and SM 2340C with normal to high hardness.

1.2 The lower limit of quantitation using this SOP is 5 mg/L.

2.0 Summary of Method

Ethylenediaminetetraacetic acid (EDTA) and its sodium salts form a chelated soluble complex when added to a solution containing metal cations such as calcium and magnesium. If a small amount of a dye such as Calmagite is added to an aqueous solution containing calcium and magnesium ions at a pH of 10, the solution becomes wine red. If EDTA is added as a titrant, the calcium and magnesium will be complexed, and when all of the calcium and magnesium ions have been complexed, the solution turns from wine red to blue, marking the end point of the titration. Magnesium ion must be present to yield a satisfactory end point, so a small amount of complexometrically neutral magnesium salt of EDTA is added to the buffer.

3.0 Definitions

Total hardness: The sum of the calcium and magnesium concentrations expressed as calcium carbonate in mg/L.

4.0 Interferences

4.1 Some metal ions interfere by causing fading or indistinct endpoints or by stoichiometric consumption of EDTA. This interference can be reduced by adding certain inhibitors before titration (See Attachment 1). The figures in the table are intended as a rough guide only. For samples with a large amount of metal interference, the hardness calculation method should be considered as an alternative.

4.2 Suspended or colloidal organic matter also may interfere with the endpoint.

4.3 Large amounts of suspended solids may interfere with the determination of the endpoint if the color of the solution can not readily be determined. These samples may require dilution prior to analysis.

4.4 This method is suitable for a wide range of concentrations, but in order to avoid large titration volumes, select a sample volume that requires less than 15 mL of EDTA titrant.

4.5 Because calcium carbonate can precipitate out of solution at high pH, the titration must be completed within 5 minutes of buffer addition to minimize the tendency toward calcium carbonate precipitation.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Nitirc Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Sodium Sulfide	Toxic	53 mg/kg (intraperitoneal)	Contact with acids liberates toxic gas. Toxic by inhalation, in contact with skin, and if swallowed. Causes burns in contact with skin and eyes. Material is extremely destructive to the tissue of the mucous membranes and upper respiratory tract.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Sodium Cyanide	Corrosive Poison	25 mg/m^3 IDLH (as CN) 5 mg/m^3 - TWA	May be fatal if inhaled, absorbed through the skin, or swallowed. May cause severe respiratory and digestive tract irritation with possible burns. May cause central nervous system effects and blood abnormalities. May cause severe eye and skin irritation, with possible burns. Contact with eyes may cause burns, chemical conjunctivitis, and corneal damage.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Balance: analytical, capable of accurately weighing to the nearest 0.0001g. The accuracy of the balance is checked each day it is used in accordance with DV-QA-0014.

6.2 Supplies

- Glassware: Class A volumetric flasks, graduated cylinders and pipettes as required.
- Mechanical pipettes
- Titration setup: Buret, Erlenmeyer flasks, stir plate and stir bar.
- Miscellaneous laboratory glassware and equipment.

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Buffer Solution

Dissolve 16.9 g of ammonium chloride (NH_4Cl) in 143 mL of concentrated ammonium hydroxide (NH_4OH). Add 1.25 g of magnesium EDTA (Ethylenediamine tetraacetic acid, magnesium salt) and dilute to 250 mL with deionized water. This solution is commercially available. Store tightly stoppered and replace after one month. Discard when 1 or 2 mL added to a sample fails to produce a pH of 10.0 ± 0.1 at the endpoint of the titration.

7.2 Calmagite Indicator

Use Calmagite powder or a commercially available Calmagite indicator solution.

7.3 1 N Ammonium Hydroxide

Dilute 35 mL of concentrated ammonium hydroxide to 500 mL with deionized water.

7.4 Inhibitor I: Sodium Cyanide

7.5 Inhibitor II: Sodium Sulfide nonahydrate.

Dissolve 5.0 g of sodium sulfide nonahydrate in 100 mL of deionized water. This solution deteriorates rapidly through air oxidation and should not be stored.

7.6 Stock Calcium Carbonate Standard, 1000 mg/L as Calcium Carbonate.

Use a commercially available source. This standard should be stored according to the manufacturer's specification. This standard expires as per the vendor specified expiration date.

7.7 EDTA Titrant, 0.02 N.

Dissolve 3.723 g of disodium EDTA dihydrate in deionized water and dilute to 1000 mL with deionized water. Standardize versus the stock calcium carbonate standard. Alternatively, a commercially available EDTA titrant may be used.

7.8 Reagent Water, $\leq$ 1 µmho-cm

7.9 Matrix Spike/Matrix Spike Duplicate

Prepare the MS/MSD by adding 10 mL of the calcium carbonate standard to 25 mL of sample. The spiking concentration is 400 mg/L.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Plastic/Glass	25 mLs	HNO_3, pH < 2; Cool 4 $\pm$ 2°C	180 Days	40 CFR Part 136.3

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Batch Definition

A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD). As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.2 Method Blank (MB)

One method blank (MB) must be processed with each batch. The MB consists of reagent water that is carried through the entire analytical procedure, including preparation and analysis. When analyzing soils, a soil MB is prepared using washed Ottawa sand. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The MB is prepared in the same manner as the samples using a DI water leach in accordance with SOP DV-WC-0036.

Acceptance Criteria: The MB should not contain any analyte of interest at or above the reporting limit (RL).

Corrective Action: If the analyte level in the MB exceeds the reporting limit for the test, all associated samples are re-prepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data must be taken in consultation with the client and must be addressed in the project narrative.

If the analyte concentration is greater than the reporting limit (RL) in the samples associated with an unacceptable MB, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be

addressed in the project narrative.

If all samples associated with a blank greater than the RL are greater than 10 times the blank value, the samples may be reported with an NCM to qualify the high blank value.

9.3 Laboratory Control Sample (LCS)

At least one LCS must be processed with each batch. The LCS consists of reagent water to which a known amount of target analyte has been added. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process.

Acceptance Criteria: In-house control limits, set at ± 3 standard deviations around the historical mean, are used as long as they are at least as tight as the regulatory limits. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

Corrective Action: If LCS recoveries are outside established control limits, the analytical system is out of control and corrective action must be taken.

If recoveries are above control limits and sulfate is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

In other circumstances, the entire batch must be re-prepared and reanalyzed.

For hardness analysis, prepare the LCS by adding 10 mL of the calcium carbonate standard (7.9) to 25 mL of deionized water. The spiking concentration is 400 mg/L.

9.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) that is prepared and analyzed along with the sample and matrix spike.

One MS/MSD pair must be processed for each batch. Some client-specific data quality objective (DQOs), may require an MS per 10 samples. This is listed in the client requirement checklist.

The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing on only the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

Preparation of the MS/SD is described in Section 7.9.

Acceptance Criteria: In-house control limits, set at ± 3 standard deviations around the historical mean, are used for recovery

acceptance as long as they are at least as tight as the regulatory limits.

Corrective Action: If the calculated recovery or relative percent difference (RPD) is outside the acceptance range, the recovery of that analyte in the LCS must be within the established control limits. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.1 Calibration and Standardization

10.1.1 Standardization of EDTA Titrant: The EDTA titrant must be standardized daily.

10.1.1.1 Place 50 mL of deionized water in a 125 mL Erlenmeyer flask.

10.1.1.2 Add 10.0 mL of stock calcium carbonate standard, 1000 mg/L (7.6)

10.1.1.3 Add 2 mL of buffer solution (7.1)

10.1.1.4 Add a small amount of powdered Calmagite or Calmagite indicator solution.

10.1.1.5 Titrate slowly with EDTA titrant (7.7), stirring continuously until the last red color disappears. The end point color is blue. Complete the titration within 5 minutes of adding the buffer solution.

10.1.1.6 Repeat and use the average of the two results as the EDTA titrant concentration.

10.2 Sample Analysis

10.2.1 Use a 25-ml sample volume, or select a volume of sample that will require less than 15 mL of EDTA titrant, but at least of 3 mL of EDTA titrant. The titration must be completed within 5 minutes of the addition of the buffer solution.

10.2.2 Place the sample aliquot into a 125-mL Erlenmeyer flask. If the aliquot used is less than 25 mL, add sufficient deionized water to bring the volume to 25 mL.

10.2.3 Add 2 mL of buffer solution.

10.2.4 If interferences are known or suspected, add one of the inhibitors at this point. For details, see Table I. If inhibitor I (sodium cyanide) must be used, be certain to do all work in a hood. Waste generated when inhibitors are used must be disposed of separately from normal waste. (See Section 15, Waste Management, for details).

10.2.5 Add a small amount of Calmagite powder or approximately 4 drops of Calmagite indicator solution.

10.2.6 Titrate with standard EDTA titrant, slowly and with continuous stirring, until the last reddish tint disappears. Solution should be blue at the endpoint. The titration must be completed within 5 minutes of the addition of the buffer solution.

NOTE: If the endpoint is not clear, interferences may be present. The sample is diluted and qualified. If a client specifically indicates that the sample contains a significant amount of heavy metals, the laboratory will use the appropriate inhibitor.

11.0 Calculations / Data Reduction

11.1 Titrant Standardization

$$\text{Normality of EDTA Titrant} = \frac{0.2}{\#\ mL\ of\ EDTA\ used\ for\ standardization}$$

11.2 Calculation of Hardness:

$$HARDNESS = \left(\frac{(VOLEDTA)(N)(50000)(DF)}{mL\ sample} \right)$$

Where:

HARDNESS	=	Hardness, mg/L as $CaCO_3$.
VOLEDTA	=	Volume of EDTA required for titration of sample, mL
N	=	Normality of EDTA
mL sample	=	mL of sample aliquotted
DF	=	Dilution factor of sample before it was aliquotted

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial MDL study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven standards at three to five times the estimated MDL concentration.
- Analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in mg/L, and the standard deviation of the concentration(s) in mg/L. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P).
- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy # DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows"

- Four aliquots of the QC check sample (independent source from the calibration) are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director.

DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually. Initial Demonstration of Capability

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program".

14.2 For this method, the following waste streams have been identified:

- All titration waste – Aqueous Alkaline (E)
- Expired standards and chemicals: Contact Waste Coordinator.

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated from this procedure.

15.0 References / Cross-References

15.1 Method Source: Methods for the Chemical Analysis of Water and Wastes, EPA Method 130.2

15.2 Method 2340C: "Standard Methods for the Examination of Water and Wastewater", 20th Edition, 1998.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 130.2 and SM 2340C	The reporting level for this method is 5mg/L. Therefore a lower level titration is not performed. If a lower level is required the samples will need to be analyzed by a different method (ICP).
2	EPA 130.2 and SM 2340C	The source method calls for the use of inhibitors for heavy metals. Do to the hazardous potential of the inhibitor solutions, samples will be analyzed at a dilution to minimize the affect of inhibitors unless otherwise indicated by the client.

17.0 Attachments

Appendix I: Maximum Concentration of Interferences Permissible with Various Inhibitors
Appendix II: Example Laboratory Bench Sheet
Appendix III: Example Checklist

18.0 Revision History

- Revision 3.2, dated 15 May 2010
  - Annual Review
  - Added section 6.3

- Revision 3.1, dated 15 May 2009
  - Added method modifications (section 16.0)
  - Updated method references
  - Updated formatting
  - Made minor spelling and grammatical corrections

- Changes from previous revisions
  - Section 11.4: Instruction to neutralize the sample with ammonium hydroxide was deleted.

Appendix I

Interfering Substance	Maximum Interference Concentration[a], mg/L	
	Inhibitor I	Inhibitor II
Aluminum	20	20
Barium	b	b
Cadmium	b	20
Cobalt	over 20	0.3
Copper	over 30	20
Iron	over 30	5
Lead	b	20
Manganese (Mn^{2+})	b	1
Nickel	over 20	0.3
Strontium	b	b
Zinc	b	200
Polyphosphate		10

NOTES:

a. Table concentrations are based on a 25 mL of sample volume diluted to 50 mL for analysis.

b. Titrates as hardness.

Inhibitor use:

Inhibitor I: Use 25 mg of sodium cyanide. Add sufficient buffer to achieve pH 10.0 and make sure to use this inhibitor in a hood.

Inhibitor II: Add 1mL of Inhibitor II.

Appendix II: Example Laboratory Bench Sheet

Laboratory Bench Sheet
Hardness Titration
Revision 3 - Sept 200[illegible]

TestAmerica Denver

HARDNESS BY TITRATION

Analyst:	RDAVIS	Titration Solutions		SOP Information:	Rep. / Detection Limits
Date:	5/14/2007	Solution 1:	EDTA	Number: DEN-WC-0060	MDL 0
QC Batch:	7134274	Source:	LABCHEM	Revision: 2	Rep. Limit 3
MS Run:	7134193	Lot #:	1611170	Calibration Information	
		Normality:	0.01984		
		Exp. Date:	4/30/2008	Lot # 6312-07	
				Made By: LabChem	
				Concentration:1000	
				STD# 1247-07	
				LCS True Value: 400	400.00
				Spike True Value: 200	200.00

Lot Number	Work Order	Sample Volume	Buret Start	Buret Stop		Final mL	Concentratn mg/L	Prep D.F.	Analyt. D.F.	Final Conc mg/L	% Rec.
	titrant stdz	10	0.00	10.05		10.05	997.033	1	1	997.03	0.01990
	titrant stdz	10	0.00	10.11		10.11	1002.985	1	1	1002.99	0.01978
	BLANK	25	0.00	0.00		0.00	0.000	1	1	0.00	
	LCS	25	0.00	10.44		10.44	414.289	1	1	414.29	
	LCS	25	0.00	9.99		9.99	396.432	1	1	396.43	
D7E080137-1	JWGKC	2	0.00	1.13		1.13	560.521	1	1	560.52	
D7E050135-1	JWDJQ	25	0.00	9.82		9.82	389.686	1	1	389.69	
D7E090168-1	JWJ40	10	0.00	0.50		0.50	49.604	1	1	49.60	
2	JWJ49	10	0.00	0.36		0.36	35.715	1	1	35.71	
D7E120139-1	JWVJQ	1	0.00	0.85		0.85	843.261	1	1	843.26	
2	JWVJ2	1	0.00	0.79		0.79	783.737	1	1	783.74	
3	JWVJ3	25	0.00	0.00		0.00	0.000	1	1	0.00	
4	JWVJ9	10	0.00	4.79		4.79	475.203	1	1	475.20	
5	JWVKE	0.5	0.00	0.40		0.40	793.658	1	1	793.66	
6	JWVKN	10	0.00	0.72		0.72	715.284	1	1	715.28	
7	JWVKQ	1	0.00	2.63		2.63	2609.150	1	1	2609.15	
8	JWVKV	5	0.00	3.40		3.40	674.609	1	1	674.61	
1S	JWVKV	5	0.00	13.60		13.60	2698.437	1	1	2698.44	
1SD	JWVKV	5	0.00	14.17		14.17	2811.533	1	1	2811.53	0.05882
											0.01471

Appendix III: Example Checklist

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TestAmerica Denver Wet Chemistry Data Review Checklist For Titration Methods

Test Name/Method #: _____ **Analysis Date** _____

SOP #: _____ **Analyst:** _____ **Instrument:** _____

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

	Yes	No	N/A	2nd Level
A. Calibration/Instrument Run QC				
1. Was the normality of the titrant verified and found acceptable?				
B. Sample Results				
1. Are all sample dilutions appropriate and do associated RLs/MDLs reflect required dilutions or limited sample volume?				
2. All reported results bracketed by in control LCS or QC Sample?				
3. Sample analyses done within holding time?				
4. Initial pH check documented for all samples (if required)?				
5. Preparation benchsheet completed and included in package (if applicable)?				
6. Special client requirements met?				
7. Was data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures?				
8. Do the prep and analysis dates in QuanTIMS reflect the actual dates?				
9. Are all data being reported highlighted on the benchsheet?				
10. Raw data copies prepared and scanned?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10x method blank result?				
2. LCS run for batch and within QC limits?				
3. MS and/or MSD run at required frequency and within limits (if applicable)?				
4. Sample DUP run at required frequency and RPD within 10%?				

Analyst: _____ **Date:** _____

Comments: _____

2nd Level Reviewer : _____ **Date:** _____

Comments: _____

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Title: Determination of solids in waters and wastes [EPA160.1, EPA160.2, EPA160.3, EPA160.4,SM 2540B, SM 2540C, SM 2540D, and SM 2540E]

Approvals (Signature/Date):

Dave Elkin — 11/1/10 — Date
Wet Chemistry Supervisor

Adam Alban — 01 Nov. 10 — Date
Health & Safety Manager / Coordinator

John P. Morris — 11/2/10 — Date
Quality Assurance Manager

Robert C. Hanisch — 11/1/10 — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This SOP is applicable to the determination of total solids, total suspended solids, total dissolved solids, volatile solids, volatile dissolved solids, and volatile suspended solids using gravimetric techniques. This SOP is applicable to drinking, surface, and saline waters and domestic and industrial wastes.

1.2 The methods cover a practical range of 10 mg/L to 20,000 mg/L (TSS: 4 mg/L - 20,000 mg/L). As a practical matter, the final residue weight should be limited to about 200 mg.

1.3 The procedure for settleable solids is in SOP # DV-WC-0032.

1.4 The procedure for percent moisture in solid samples is in SOP # DV-WC-0023.

2.0 Summary of Method

2.1 **Total Solids (TS):** A well-mixed aliquot of the sample is quantitatively transferred to a preweighed evaporating dish and evaporated to dryness at 103-105 °C. The increase in weight over that of the empty dish represents the total solids.

2.2 **Total Dissolved Solids (TDS):** A well-mixed sample is filtered through a glass fiber filter. The filtrate is quantitatively transferred into a preweighed evaporating dish and is evaporated to dryness and then dried to constant weight at 180 °C. The increase in weight over that of the empty dish represents the total dissolved solids. The filter from this procedure may also be used for TSS/VSS determination.

2.3 **Total Suspended Solids (TSS):** A well-mixed sample is filtered through a pre-weighed glass fiber filter. The residue on the filter is dried to constant weight at 103-105 °C. The increase in weight over that of the pre-weighed filter represents the TSS content. The filtrate from this procedure may be used for TDS determination. The filter from this procedure may also be used for VSS analysis.

2.4 **Volatile Solids (VS):** The residue obtained from the determination of total solids is ignited at 550 °C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile solids.

2.5 **Volatile Dissolved Solids (VDS):** The residue obtained from the determination of total dissolved solids is ignited at 550 °C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile dissolved solids.

2.6 **Volatile Suspended Solids (VSS):** A well-mixed sample is filtered through a glass fiber filter to separate the suspended material. The filter is dried and weighed, then ignited at 550 °C and reweighed. Volatile suspended solids is determined from the weight loss after ignition. The filter from the analysis of TSS may be used for the determination of VSS.

3.0 Definitions

3.1 **Total Solids (TS):** The term applied to the residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at 103-105 °C. Total solids includes "total suspended solids," the portion of solids retained by a filter, and "total dissolved solids," the portion that passes through the filter. The reporting limit is 10 mg/L.

3.2 **Total Dissolved Solids (TDS):** Those solids capable of passing through a glass fiber filter and dried to constant weight at 180 °C. TDS is also referred to as filterable residue.

3.3 **Total Suspended Solids (TSS):** Those solids which are retained by a glass fiber filter and dried to constant weight at 103-105 °C. TSS is also referred to as non-filterable residue.

3.4 **Volatile Solids (VS):** The portion of total solids which is lost on ignition at 550 °C.

3.5 **Volatile Dissolved Solids (VDS):** The portion of total dissolved solids which is lost on ignition at 550 °C.

3.6 **Volatile Suspended Solids (VSS):** The portion of suspended solids which is lost on ignition at 550 °C.

3.7 **Aliquot:** A representative portion of a sample.

3.8 **Reagent Water:** Deionized water which is free of the analyte(s) of interest.

4.0 Interferences

4.1 Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. All these materials must be routinely demonstrated to be free from interferences under the conditions of analysis by running method blanks.

4.2 Non-homogeneous samples may give erroneous results. Samples should be mixed as thoroughly as possible before removing an aliquot for analysis.

4.3 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the benchsheet.

4.4 Samples containing large amounts of solids may filter slowly. Prolonged filtration times resulting from filter clogging may produce high TSS results due to increased colloidal materials captured on the clogged filter.

4.5 Oil and grease in the samples will cause unreliable results due to difficulty in drying to constant weight. Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before aliquoting.

4.6 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperatures are specified because these variables have been shown to affect the results.

4.7 The temperature at which the residue is dried has an important bearing on the results because weight losses due to volatilization of organic matter, mechanically occluded water, water of crystallization, and gases from heat-induced chemical decomposition, as well as weight gains due to oxidation, depend on temperature and time of heating.

4.8 Each sample requires close attention to desiccation after drying. Minimize opening the desiccator because moist air enters. Some samples may be stronger desiccants than those used in the desiccator and may take on water.

4.9 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

4.10 Samples containing high concentrations of bicarbonate may require careful and possibly prolonged drying to ensure that all the bicarbonate is converted to carbonate.

4.11 Too much residue in the drying vessel will crust over, entrapping water that will not be driven off during drying. Total residue should be limited to about 200 mg.

4.12 Some samples may have fine suspended solids which will pass through the glass fiber filter causing high TDS results.

4.13 Aluminum pans should not be used for TS or TDS analyses. Components in some samples may react to form aluminum compounds, causing unreliable results.

4.14 For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material prior to TSS determination.

4.15 The volatile solids tests are subject to many errors due to the loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics and decomposition of mineral salts during combustion. The results should not be considered an accurate measure of organic carbon in the sample.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Eye protection that satisfies ANSI Z87.1 (as per the Environmental Health and Safety Manual), laboratory coat, and nitrile or latex gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2 Primary Materials Used

There are no materials used in this method that have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

6.1 Analytical balance capable of weighing to 0.0001 g. The balance calibration is checked each day of use with three Class 1 weights that bracket the range of use. Details for this procedure are covered in SOP DV-QA-0014.

6.2 Vacuum filtration apparatus.

6.3 Vacuum pump equipped with moisture trap.

6.4 Glass fiber filter disks, 47 mm, without organic binder (Gelman Type A/E) or equivalent. The laboratory currently uses pre-washed and pre-weighed filters purchased from an outside vendor. If these pre-prepared filters are not available, filters can be prepared as stated below.

6.4.1 Preparation of Glass Fiber Filter Disc for TSS/VSS

6.4.1.1 Place the glass fiber filter discs, one at a time, on the membrane filter apparatus with wrinkled surface up.

6.4.1.2 While vacuum is applied, wash the disc with three successive (approximately) 20 mL volumes of distilled water.

6.4.1.3 Remove all traces of water by continuing to apply vacuum after water has passed through and discard washings.

6.4.1.4 Remove filter from membrane filter apparatus and place in a labeled, aluminum weighing dish and dry in an oven at 103-105 °C for one hour.

6.4.1.5 Remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

6.4.1.6 Weigh the cooled filter and aluminum weighing dish to the nearest 0.1 mg using an analytical balance. Record the weight and the dish identification number on the benchsheet.

6.5 Glass beakers for TDS, minimum 150 mL volume, must be thoroughly cleaned, rinsed with de-ionized water and baked at 180 ± 2 °C for TDS and 104 ± 2 °C for TS at least one hour before use. Transfer to a desiccator and allow to cool completely before use.

Note: Glass beakers may not be used for procedures requiring a muffle furnace. In that case, porcelain dishes, pre-dried and weighed, must be used.

6.6 Desiccators providing sufficient space for storage of samples in process separate from filters and evaporating dishes.

6.7 Desiccant containing a color indicator of moisture concentration or an instrumental indicator.

6.8 Drying ovens set at 103-105 °C and 180 ± 2 °C. Separate ovens should be maintained at appropriate temperatures if possible.

6.9 Muffle furnace (550 °C ± 50 °C).

6.10 Thermometers, NIST traceable.

6.11 Conductivity meter and associated apparatus.

6.12 Graduated cylinders, assorted sizes.

6.13 Volumetric flasks, Class A, assorted sizes.

6.14 Aluminum weighing dishes large enough to hold a 47 mm filter.

6.15 Forceps for handling filters.

6.16 Crucible tongs.

6.17 Zetex gloves or other gloves capable of providing protection at 550 °C.

7.0 Reagents and Standards

7.1 Reagent water must be produced by a Millipore DI system or equivalent (see also Section 10.1.3).

7.2 LCS solution (500 mg/L TDS):

Place 500.0 mg of sodium chloride into a 1000 mL volumetric flask and dilute to volume with deionized water. Mix well. Prepare fresh every three months. Alternatively, a commercially available LCS solution may be used.

7.3 Commercially available reference materials are used for the TSS/TS LCS.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Method	Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
TS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
TDS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
TSS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
VS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
VDS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
VSS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3

9.0 Quality Control

9.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL) as described in Section 12.1.

9.2 In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument they will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12.2 for more details.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See QC Policy (DV-QA-003P) for further details.

9.4 Method Blank

A method blank is required with every batch of 20 or less samples. The blank is deionized water taken through the procedure like a sample. The general requirement is that the method blank result must be less than the reporting limit or one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL). Note that some agencies (e.g., South Carolina) require that the blank must be less than the MDL – see special requirements in LIMS to determine which criterion applies.

Corrective Action: If the method blank exceeds allowable levels, all associated samples must be reanalyzed

9.5 Laboratory Control Sample (LCS)

One LCS is required with each analytical batch. Historical control limits are based on three standard deviations of past results, and must be 80-120% or tighter. The process of establishing control limits is described in more detail in the QC Policy DV-QA-003P.

Corrective Action: If the LCS exceeds allowable levels, all associated samples must be reanalyzed.

9.6 Duplicate Sample Analysis

Two duplicate pairs are required with each analytical batch. The relative percent difference (RPD) must be within 10%. Note that the control limits only apply to samples with results greater than 5 times the RL. The process of establishing control limits is described in more detail in the QC Policy DV-QA-003P.

Corrective Action: If the RPD is greater than 10 the sample should be reanalyzed.

10.0 Procedure

- One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo (see SOP # DV-QA-0031 for details) and must be approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.
- Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.
- All samples are to be checked out of sample control with the chain of custody documentation filled out completely.
- Proper sample identification is extremely important in any analytical procedure. Labeling of evaporating dishes and filters holders must be done in a manner to ensure connection with the proper sample.
- If possible, analyze all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.
- Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the benchsheet.
- If samples are visibly oily, this should be noted on the benchsheet.
- If there is limited sample volume or high solids content, smaller amounts of sample may need to be processed than detailed in the following sections. This occurrence must be noted on the benchsheet and reporting limits must be adjusted appropriately.

10.1 Calibration

10.1.1 Since this method is based on gravimetric techniques, there is no calibration in the usual sense. Proper balance operation will be verified daily or prior to sample analysis by following the lab-specific balance calibration SOP. Analytical balance calibration verification must be performed daily (every 24 hours).

10.1.2 Oven temperature must be checked daily and recorded either on the benchsheet or in an oven temperature logbook.

10.1.3 Conductivity of the water must be monitored and recorded in the Conductivity Logbook daily. The maximum permissible conductivity is 1.0 umhos/cm (at 25 °C). If the conductivity reading on the water system exceeds this level, do not use the water for these procedures and notify the supervisor immediately.

10.2 Sample Preparation

10.2.1 Total Solids

10.2.1.1 If only total solids are to be measured, heat clean dish to 103-105 °C for one hour. If volatile solids are to be measured in addition to total solids, ignite the clean evaporating dish at 550 °C for one hour in a muffle furnace.

10.2.1.2 Remove the dish from the muffle furnace using tongs and heat resistant gloves.

10.2.1.3 Cool and store dish in desiccator until dish reaches room temperature or until needed.

10.2.1.4 Weigh immediately before use to the nearest 0.1 mg. Record the weight into the LIMS benchsheet.

10.2.2 Total Dissolved Solids

Preparation of beakers/evaporating dishes.

- If only total dissolved solids are to be measured, heat clean beakers to 180 ± 2 °C for one hour. If volatile dissolved solids are to be measured in addition to TDS, ignite the clean evaporating dish at 550 ± 50 °C for one hour in a muffle furnace.
- Heat resistant gloves and tongs must be used when removing items from the muffle furnace.
- Store and cool dish in desiccator until dish reaches room temperature or until needed.

Note: Analyst must transfer the dish with gloves or tongs to prevent added weight due to oil from fingerprints.

- Weigh immediately before use to the nearest 0.1 mg. Record the weight of the dish into the LIMS benchsheet.

10.2.3 Total Suspended Solids

The pre-washed and pre-weighed filters come in aluminum pans that have scan bars that are associated with an ID and weight of each filter. These IDs are scanned into the LIMS system. If filters need preparation, see Section 6.4.1.

10.3 Sample Analysis

10.3.1 Total Solids

10.3.1.1 Transfer a measured aliquot of well-mixed sample to the pre-weighed, labeled dish. Record the volume of sample (to the nearest mL) on the benchsheet.

10.3.1.2 Choose an aliquot of sample sufficient to contain a residue of at least 10 mg but less than 200 mg.

10.3.1.3 If the sample is known to contain > 2000 mg/L dissolved solids, it should be diluted. Prescreening should be performed using a conductivity meter to determine the required sample volume or dilution. Dilution should be the smallest dilution sufficient to bring the approximate conductivity to under 3000 umho/cm.

10.3.1.4 For the LCS, measure 100 mL of the LCS Solution (Section 7.3) and pour into the dish.

10.3.1.5 For the MB, measure 100 mL of reagent water and pour into the dish.

10.3.1.6 Place the tray of samples into a drying oven and evaporate to dryness.

10.3.1.7 Dry the evaporated sample for at least one hour at 103-105 °C.

10.3.1.8 Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.1.9 Remove the tray of weighing dishes from the oven using heat-resistant gloves. Place in a desiccator and cool to room temperature.

10.3.1.10 Weigh the dish to the nearest 0.1 mg. Record the weight on the LIMS benchsheet.

10.3.1.11 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight difference is ≤ 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

Note: When weighing dried sample, be alert to change in weight due to air exposure and/or sample degradation.

10.3.1.12 If volatile solids are to be determined, treat the residue according to Section 10.3.4.

10.3.1.13 Calculate results according to the equation provided in Section 11.3.1. Use the final weight achieved for calculating TS.

10.3.2 Total Dissolved Solids

10.3.2.1 Thoroughly rinse the entire filtration apparatus with reagent water before filtering each sample.

10.3.2.2 Assemble the filtering apparatus, place a glass fiber filter in the apparatus, pre-wet the filter using reagent water, and begin suction.

Note: If the sample also requires TSS, pre-weigh the prepared filter and refer to Section 10.3.3 for additional guidance.

10.3.2.3 Transfer 100 ml or a larger volume to yield between 10 and 200 mg dried residue to the funnel by means of a graduated cylinder. If more than 10 minutes are required to complete filtration, decrease sample volume or increase filter size.

Note: Multiple filters may be used if performing only TDS analysis.

10.3.2.4 The conductance of each sample may be used to determine the appropriate sample volume to process.

Note: TDS is typically 55-90% of the conductance result. The exact relationship depends on the compounds present in the samples and may not hold for very high concentrations or samples containing non-ionic species or samples with conductance greater than 10,000 umho/cm or less than 50 umho/cm.

10.3.2.5 If the sample has a conductance less than 3,000 umhos/cm, 100 mL should be used.

10.3.2.6 If the conductance is > 3,000 umhos/cm, the smallest dilution that would bring the conductance to under 3,000 umhos/cm should be used.

10.3.2.7 A smaller amount should be filtered if the sample is high in TSS or is otherwise slow to filter. Filter 25 mL at a time until filtration slows. Record on the benchsheet the reason that a smaller volume had to be used and any sample observations.

10.3.2.8 Record the volume of sample used (to the nearest mL) on the benchsheet.

10.3.2.9 For the method blank, process 100 mL of reagent water as the sample. Blank result should be less than method detection limit.

10.3.2.10 For the LCS, process 100 mL of the LCS Solution. Refer to Section 7.2 for instructions on how to prepare the LCS.

10.3.2.11 Filter the sample through the glass fiber filter.

10.3.2.12 Rinse the graduated cylinder, funnel walls, and filter with three successive 10 mL portions of reagent water and allow

for complete drainage between washings. Continue to apply vacuum until the filter is completely dried.

10.3.2.13 Transfer the filtrate (including the washings) to a pre-weighed evaporating dish on a tray. Rinse the receiving flask with 10-25 mL of reagent water and transfer washings into the dish to ensure complete transfer of the sample.

10.3.2.14 Evaporate the samples to dryness in a drying oven.

10.3.2.15 Dry the evaporated sample in an oven for at least one hour at 180 ± 2 °C.

10.3.2.16 Record the date, time, and oven temperature on the LIMS benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.2.17 Use heat resistant gloves to remove the tray of samples from the oven. Place in a desiccator and cool to room temperature.

10.3.2.18 Weigh the dish to the nearest 0.1 mg. Record the combined weight of the dried residue and the dish on the LIMS benchsheet.

Note: If the sample residue is over 200 mg (0.2 g) the sample needs to be re-analyzed at a dilution or an NCM needs to be written.

10.3.2.19 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating and weighing cycle until a constant weight is obtained or weight difference is ≤ 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

10.3.2.20 Calculate results according to the equation in Section 12.2. Use the final weight achieved for calculating TDS.

10.3.3 Total Suspended Solids

10.3.3.1 Assemble the filtering apparatus, place the pre-weighed glass fiber filter in the apparatus, pre-wet the filter using reagent water and begin suction.

Note: Handle the filters only with forceps.

10.3.3.2 Selection of Sample Volume

- For a 47 mm diameter filter, sufficient sample to yield between 10 mg and 200 mg of dried residue. Some clients or agencies (e.g. South Carolina) require increasing standard volume from 250 mL up to 1L to achieve a minimum residue of between 10 and 200 mg.

- Because it can be difficult in some samples to determine the amount of TSS present visually, record on the benchsheet all observations on samples for which the entire volume could not be filtered due to slowing of filtration.
- If during filtration of this initial volume, the filtration rate drops rapidly or if filtration time exceeds 5-10 minutes, a smaller volume of sample should be processed.

  Note: If the sample appears high in TSS, start with a smaller sample volume.

10.3.3.3 Invert and shake the sample vigorously, then quickly aliquot the sample. It is important to pour out the sample immediately after shaking so that the solids do not have time to settle. A smaller amount should be filtered if the sample is high in TSS or is otherwise slow to filter. Filter 25 mL at a time until filtration slows. Record the volume of sample filtered (to the nearest mL) on the benchsheet.

Note: If Total Dissolved Solids (TDS) is also required, the filtrate may be used. Refer to Section 10.3.2 for additional guidance.

10.3.3.4 Remove all traces of water by continuing to apply vacuum after the sample has passed through.

10.3.3.5 With suction on, rinse the graduated cylinder, filter, suspended solids residue, and filter funnel wall with three 10 mL portions of reagent water allowing complete drainage between washings.

10.3.3.6 Remove all traces of water by continuing to apply vacuum for about three minutes after the sample has passed through.

10.3.3.7 Carefully remove the filter from the filter support and transfer to an aluminum weighing dish. If the filter is torn or damaged during this process, the sample must be reanalyzed. Take care to keep the filter face-up during the transfer so that the residue does not fall off.

10.3.3.8 Dry the filter at least one hour at 103-105 °C.

10.3.3.9 Record the date, time, and oven temperature on the LIMS benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.3.10 Use heat resistant gloves to remove the tray of dishes from the oven. Place in a desiccator and cool to room temperature.

10.3.3.11 Cool the samples in a desiccator (minimum of 1 hour) weigh (to the nearest 0.1 mg), and record the weight on the benchsheet.

10.3.3.12 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight difference is ≤ 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

10.3.3.13 If volatile suspended solids are to be determined, treat the residue according to Section 10.3.6.

10.3.3.14 Calculate the results using the formula given in Section 11.3.2. Use the final weight achieved for calculating TSS.

10.3.4 Volatile Solids

10.3.4.1 Heat muffle furnace up to temperature (550 ± 50 °C).

10.3.4.2 Place evaporating dish containing residue generated by Total Solids protocol (Section 10.3.1) in muffle furnace to ignite the residue.

10.3.4.3 Record the date, time, and oven temperature on the LIMS benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.4.4 Typically, 15-20 minutes ignition is required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

10.3.4.5 Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace.

10.3.4.6 Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling.

10.3.4.7 Return the samples to the muffle oven for another cycle repeating steps 10.3.4.2 – 10.3.4.6 until a constant weight is obtained or weight difference is less than 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

10.3.4.8 Calculate the results using the formula given in Section 11.3.4. Use the lowest weight achieved for calculating VS.

10.3.5 Volatile Dissolved Solids

10.3.5.1 Heat muffle furnace up to temperature (550 ± 50 °C).

10.3.5.2 Place evaporating dish containing residue generated by Total Dissolved Solids protocol (Section 10.3.2) in muffle furnace to ignite the residue. The dish used should be a ceramic weighing dish, not a glass beaker.

10.3.5.3 Record the date, time, and oven temperature on the LIMS benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.5.4 Typically, 15-20 minutes ignition is required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

10.3.5.5 Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace.

10.3.5.6 Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling to room temperature.

10.3.5.7 Return the samples to the oven for another cycle of weighing. Repeat steps 10.3.5.2 to 10.3.5.6 until a constant weight is obtained or weight difference is less than 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

10.3.5.8 Calculate the results using the formula given in Section 11.3.5. Use the lowest weight achieved for calculating VDS.

10.3.6 Volatile Suspended Solids

10.3.6.1 Heat muffle furnace up to temperature (550 ± 50 °C).

10.3.6.2 Place glass fiber filter disc containing residue generated by Total Suspended Solids protocol (Section 11.12) in muffle furnace to ignite the residue.

10.3.6.3 Record the date, time, and oven temperature on the LIMS benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

10.3.6.4 Typically, 15-20 minutes ignition is required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

10.3.6.5 Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace.

10.3.6.6 Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling to room temperature.

10.3.6.7 Return the samples to the oven for cycle of weighing, repeating steps 10.3.6.3 – 10.3.6.6 until a constant weight is obtained or weight difference is less than 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

10.3.6.8 Calculate the results using the formula given in Section 11.3.6. Use the final weight achieved for calculating VSS.

11.0 Calculations / Data Reduction

- If smaller or larger sample volumes are processed than are specified in the method, the reporting limit must be adjusted accordingly.
- All data are subject to two levels of review, which is documented on a checklist (see example Data Review Checklist in Attachment 3).
- If multiple weighing cycles are required, the lowest final sample weight is used for calculating solids content.

11.1 Conversion equation

All sample are weighed in grams but reported in milligrams. Use the following equation before computing further calculations:

Weight in grams X 1000 = Weight in milligrams

11.2 Accuracy

$$\text{LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.3 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

11.4 Concentration

11.4.1 Total Solids =

$$\text{Total Solids, mg/L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

11.4.2 Total Dissolved Solids =

$$\text{Total Dissolved Solids, mg/L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

11.4.3 Total Suspended Solids =

$$\text{Total Suspended Solids, mg/L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of filter + residue (mg)
B = weight of filter (mg)
C = volume of sample filtered (mL)

11.4.4 Volatile Solids =

$$\text{Volatile Solids, mg/L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of residue + dish before ignition (mg)
B = weight of residue + dish after ignition (mg)
C = volume of sample (mL)

11.4.5 Volatile Dissolved Solids = Volatile Dissolved Solids, mg/L $= \frac{(A - B) \times 1000}{C}$

Where: A = weight of residue + dish before ignition (mg)
B = weight of residue + dish after ignition (mg)
C = volume of sample (mL)

11.4.6 Volatile Suspended Solids = Volatile Suspended Solids, mg/L $= \frac{(A - B) \times 1000}{C}$

Where: A = weight of residue + filter before ignition (mg)
B = weight of residue + filter after ignition (mg)
C = volume of sample (mL)

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P) for each analyte.
- The MDL studies and concentrations can be found at L:\QA\Read\MDL.
- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy # DV-QA-005P for further details concerning MDL studies.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows"

- Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.
- Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need

to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP# DV-QA-0024.

13.0 Pollution Control

Prescreening is performed to determine the required sample volume or dilution in order to minimize laboratory waste. In addition, standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents to be disposed.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

The following waste streams are produce when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Acidic sample waste – Waste Stream F
- Filter and filter residue – nonhazardous waste
- Solid soil waste – Waste Stream S

Note: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated from this procedure.

15.0 References / Cross-References

15.1 Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1979: Methods 160.1, 160.2, 160.3, and 160.4.

15.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1992: Methods 2540A, 2540B, 2540C, 2540D, and 2540E.

16.0 Method Modifications:

None

17.0 Attachments

Attachment 1: Example TDS/TS/TSS Benchsheet

Attachment 2: Example VSS/VDS/VS Benchsheet

Attachment 3: Data Review Checklist

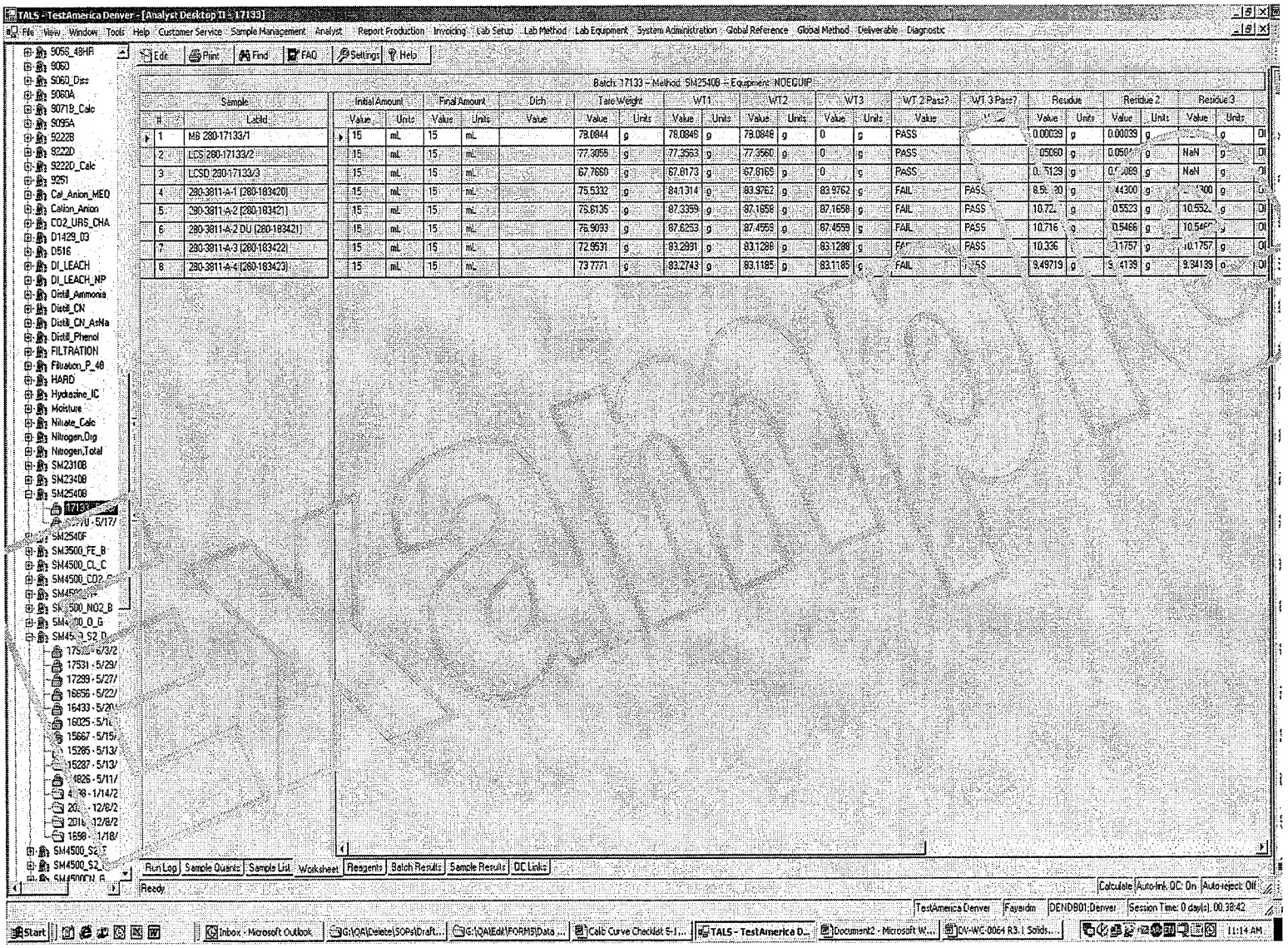
18.0 Revision History

- Revision 5.1, dated 19 November 2010
  - Corrected the duplicate acceptance criteria in section 9.6.
- Revision 5, dated 08 October 2010
  - Added Note to section 10.3.2.18 concerning sample residue weights.
- Revision 4, dated 18 June 2010
  - Annual Technical Data Review
  - Updated SOP to include the use of pre-washed & weighed filters.
  - Updated all attachments to include the new LIMS bench sheets.
- Revision 3.1, dated 19 June 2009
  - Corrected minor grammatical and formatting errors.
  - Technical and method review performed.
- Revision 3, dated 16 June 2008
  - Fixed formatting
  - Added EPA references to SOP
- Revision 2, dated 21 December 2007
  - Integration for TestAmerica and STL operations.
  - Updated formatting.
  - Updated to include the 10% sample duplicate requirement from Standard Methods.
- Revision 1.1, dated 30 December 2005
  - Safety and Waste section updates. The technical interim changes are maintained, and the revision was increased by "X.1"
- Revision 1, dated 26 August 2002
  - Definition for TS in 3.1 corrected to have drying temperature (103-105 degrees C) match text.
  - Standard volume for TSS and VSS changed from 250 ml to 100 ml.
  - Changed references to STL from Quanterra.
  - Updated references in Quality Control Section.
  - Added Appendices with bench sheet and data review checklist.
  - Changed volume required for TSS to be up to 1L of sample, to yield 1 mg of residue weight.
  - Added requirement of some clients for a duplicate 1 per 10 samples to 9.4.3.2.
  - Added requirement of some clients to control results down to the MDL to 9.4.1.1.
  - Added reference to the balance SOP to 6.1.
  - Changed "weight loss" to "weight difference" in description of cyclical weighings.

- Removed commercially prepared filters for TSS from the procedure.
- Update to section 13 to include information on MDLs & IDCs.
- Removal of reference to MS and MSD.

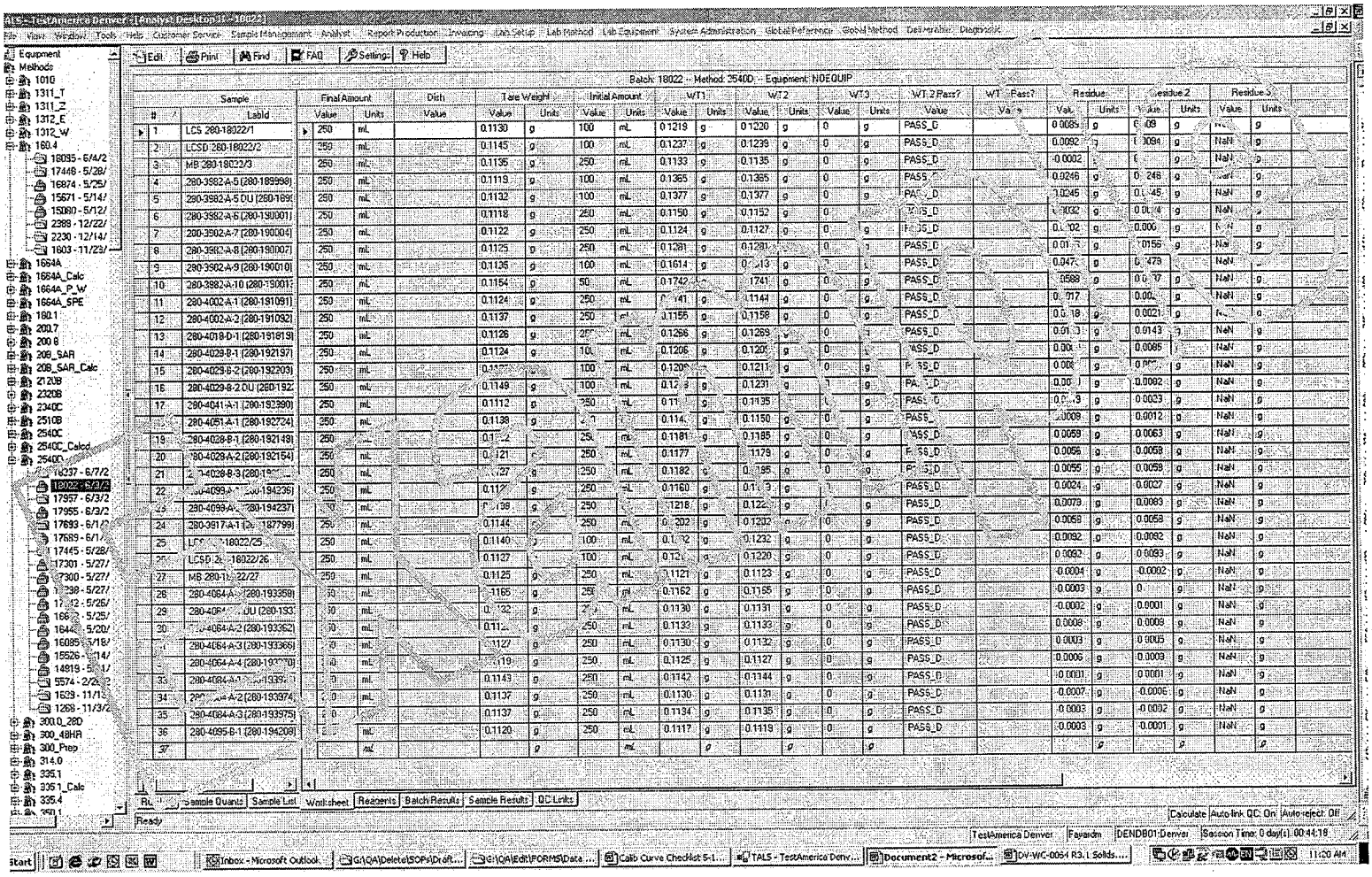
Attachment 1.

Example TDS/TS/TSS Benchsheet



Attachment 2.

Example VSS/VS/VDS Benchsheet



Attachment 3.

Data Review Checklist

TestAmerica Denver

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Wet Chemistry Data Review Checklist For Gravimetric Methods

Test Name/Method #: _____ **Analysis Date:** _____

SOP #: _____ **Analyst:** _____ **Instrument:** _____

Lot / Sample Numbers	Matrix	Prep Batch	Batch	Method	Special Inst

A. Balance, Oven, and DI Water QC Checks	Yes	No	N/A	2nd Level
1. Was the balance calibration verified before and after processing samples and noted in the "Balance Calibration Log" for the date(s) the samples were processed?				
2. Was the oven temperature within method requirements and recorded in the "Oven Temperature" logbook for the date(s) the samples were processed?				
3. Was the daily conductivity check of the deionized water recorded in the "Conductivity Logbook"?				
B. Method Requirements				
1. If sample is visibly oily, was this noted on the benchsheet?				
2. Was final residue weight within minimum/maximum requirements?				
3. Were the initial and final drying dates and times recorded on the benchsheet and were all samples dried for at least one hour?				
C. Sample Results				
1. TDS/Conductivity ratio or historical data checked?				
2. For % Moisture, was the Final Dried Weight < the Initial Pan Weight or is the result greater than 100%?				
3. Were sample analyses done within holding time?				
4. Were special client requirements met?				
5. Were data that were manually transcribed from instrument printouts into TALS verified 100% including significant figures and units?				
6. Do the prep and analysis dates in TALS reflect the actual dates? Lots/Dates report checked?				
7. STD/True Value information is updated and included?				
8. Are raw data copies prepared, scanned, and uploaded?				
D. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10 X RL?				
2. Method blank < ½ RL or NCM provided?				
3. LCS/LCSD run for batch and within QC limits?				
4. DUP run for batch and RPD < 20% for samples >5 X RL?				

Analyst: _____ **Date:** _____

Comments: _____

2nd Level Reviewer : _____ **Date:** _____

Comments: _____

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Total and Amenable Cyanide by SW-846 9010C, 9012A and 9013

Approvals (Signature/Date):

Dave Elkin — Date
Wet Chemistry Supervisor

Adam Alban — Date
Health & Safety Manager / Coordinator

Karen Kuoppala — Date
Quality Assurance Manager

Robert C. Hanisch — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This procedure is for the determination of:

Analyte	CAS Number
Total Cyanide	57-12-5

1.1.2 This procedure is applicable to soils, domestic and industrial wastes and leachates.

1.1.3 This procedure detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine total and amenable cyanide as described in SW-846 9012A.

1.1.4 This procedure describes the reduced volume version of the methods and uses the same reagents and molar ratio to meet the quality control and performance requirement stated in the method.

1.1.5 Dynamic Range
The approximate working range extends from 0.01 mg/L to 0.4 mg/L (0.5 mg/kg to 20 mg/kg). Samples with higher concentrations are analyzed with dilution. Current method detection limits are given in the laboratory LIMS system.

2.0 Summary of Method

2.1 Total cyanide – Cyanide, as hydrocyanic acid (HCN) is released from samples by means of reflux-distillation under acidic condition and absorbed in a scrubber containing sodium hydroxide (NaOH) solution. The cyanide concentration in the scrubber solution is determined using an automated analyzer. The cyanide is converted to cyanogen chloride by reactions with Chloramine-T that subsequently reacts with pyridine and barbituric acid to give a red-colored complex. The color intensity which is proportionate to the cyanide concentration is measured at 570 nm.

3.0 Definitions

3.1 **Cyanide:** The term "cyanide" refers to all of the CN groups in cyanide compounds that can be determined as the cyanide ion, CN- by the various chemical methods. These compounds include both simple and complex cyanides.

4.0 Interferences

4.1 Oxidizing agents such as chlorine will destroy cyanide. Sodium arsenite is used to remove chlorine interferences.

4.2 Samples that contain sulfide compounds may produce hydrogen sulfide during the distillation and interfere with color development. This is treated by adding an excess of bismuth nitrate to the sample prior to distillation.

4.3 Chlorine added to the sample for amenable cyanide must be completely destroyed before distillation. Otherwise, it may distill over and destroy the non-amenable cyanide. Chlorine present in samples from chlorinated sources will also destroy cyanide and must be destroyed before distillation.

4.4 Nitrate and/or nitrite may react with organic compounds during distillation to form cyanide. Sulfamic acid is added to remove the nitrate and/or nitrite interference.

4.5 Samples containing surfactants may foam excessively during distillation.

4.6 High carbonate concentrations may react violently when sulfuric acid is added to the samples during distillation.

4.7 Amino acids may distill with the cyanide and interfere with the analysis.

4.8 Fatty acids may interfere by forming soaps in the absorption solution.

4.9 Thiocyanate greater than 10 mg/L may interfere.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, latex or nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Potassium cyanide and sodium cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, and dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Potassium Hydroxide	Corrosive Poison Reactive	2 mg/m3 - ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting and diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness and swelling. Greater exposures cause severe burns with possible blindness.
Hydrochloric Acid	Corrosive Poison	5 ppm - ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat and upper respiratory tract and in severe cases, pulmonary edema, circulatory failure and death. Can cause redness, pain and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric acid	Corrosive Poison Irritant Carcinogen	1mg/m3 TWA	Inhalation symptoms may include irritation of the nose and throat, and labored breathing. Swallowing can cause severe burns of the mouth, throat, and stomach, leading to death. Can cause sore throat, vomiting, and diarrhea. Skin contact can cause redness, pain, and severe burn. Eye contact can cause blurred vision, redness, pain and severe tissue burns.
Calcium hypochlorite	Strong oxidizer	None listed	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Glacial acetic acid	Corrosive Poison Flammable Irritant	10 ppm TWA	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Swallowing can cause severe injury leading to death. Skin contact may include redness, pain, and skin burns. Eye contact may cause severe eye damage followed by loss of sight.
Sulfamic acid	Corrosive Irritant	None listed	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.
Chloramine-T	Irritant	None listed	May cause irritation to the mucous membranes and upper respiratory tract, skin and eyes.
Barbituric acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as a potential health hazard; do not ingest.
Bismuth Nitrate	Oxidizer	None	May cause irritation to the respiratory tract, skin and eyes.
Silver Nitrate	Corrosive Poison Oxidizer	0.01 mg/m3 (TWA) for silver metal dust and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns, and eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3 Build-up of pressure in the distillation apparatus will cause the hot, acidic solution to spray out of the thistle tube. In case vacuum is lost, the condensers must be opened to prevent build-up of pressure. If the solution overflows onto the heating block, turn it off. Unplug and replace the heating mantle when it cools.

5.4 All distillations are to be performed with adequate ventilation.

5.5 Exposure to chemicals must be maintained as low as reasonable achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit. For cyanide amenable to chlorination, the chlorination step will also be performed in a fume hood.

5.7 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Denver associate. The situation must be reported immediately to a laboratory supervisor and the Health and Safety Officer.

6.0 Equipment and Supplies

6.1 Instrumentation

- Alpkem Automated Segmented Flow Analyzer
  - Autosampler
  - Proportioning pump
  - Injection module
  - Colorimeter with 570 nm filter and 10 mm flow cell
  - WinFlow Software
  - Debubblers
  - Miscellaneous tubing and reaction coils.

- Midi-distillation apparatus consisting of 100 mL distillation tubes, cold-finger condensers, absorption tubes, and associated apparatus.
- Vacuum pump.
- Recirculating chiller.

6.2 Supplies

- Disposable auto-sampler vials or culture tubes for samples.
- Eppendorf pipettes, various sizes.
- Volumetric flasks, class A, various sizes.
- Volumetric pipettes, class A, various sizes.
- Miscellaneous laboratory apparatus and glassware.

7.0 Reagents and Standards

All Standards purchased from vendors are NIST traceable. Reagents used in the lab for standard preparation must meet ACS specifications.

7.1 Cyanide Calibration Stock Standard, 1,000 mg/L

7.1.1 This standard is purchased commercially.

7.1.2 *Alternatively*, it can be made as described here.

7.1.2.1 Dissolve 2.51 g of dried (103 °C) potassium cyanide and 2.0 g potassium hydroxide in water and dilute to 1,000 mL.

7.1.2.2 This stock solution will be standardized initially and every 30 days thereafter as described below.

7.1.2.3 All intermediate and working standard concentrations are adjusted from the nominal concentrations shown below to the exact concentrations based on the results of the weekly standardization.

7.2 Standardization of the Stock Cyanide Solution

7.2.1 Indicator: Dissolve 20 mg p-dimethylaminobenzalrhodanine in 100 mL acetone.

7.2.2 Silver nitrate ($AgNO_3$) titrant: 0.0192 N: Obtain a commercially prepared certified standard.

7.3 Cyanide Standardization

7.3.1 Add 10 mL of the 1000 mg/L Cyanide Stock Standard (Section 7.1) solution to a 500 mL Erlenmeyer flask. Dilute to 250 mL with deionized water.

7.3.2 Add 5-7 drops of p-dimethylaminobenzalrhodanine indicator solution.

7.3.3 Titrate with standard silver nitrate, $AgNO_3$, to the first change in color from a canary yellow to a salmon hue.

7.3.4 Prepare a blank in a similar fashion. Add 250 mL of deionized water to a 500 mL Erlenmeyer flask.

7.3.5 Add 5-7 drops of p-dimethylaminobenzalrhodanine indicator solution. Titrate with standard AgNO3 to the first change in color from a canary yellow to a salmon hue. The standardization should be done in duplicates.

7.3.6 Calculate the true stock cyanide concentration as follows:

$$\text{mg/L Stock Cyanide} = \frac{[(A - B) \times 1000]}{\text{mL of sample}}$$

A = Volume of $AgNO_3$ for titration of sample, mL

B = Volume of $AgNO_3$ for titration of blank, mL

7.3.7 If the verification result is within 97% of the initial value, place the date and initials of the analyst verifying the standard in the Comment box of the standard in the Standards Log. A new Standards Log entry is not required.

7.3.8 If the verification result is less than 97% of the initial value, place the date and initials of the analyst verifying the standard in the Comment box of the standard in the Standards Log.

7.3.9 Create a "new" standard in the Standard Log for this standard using the new concentration from the verification process.

7.3.10 Prepare a label for the stock solution with the name of the analyst, date of verification, expiration date (one month beyond the date of re-standardization), lot number, 1% (0.25N) NaOH, and the actual stock cyanide concentration.

7.3.11 The true concentration of the stock cyanide solution after standardization with silver nitrate is used to prepare intermediate and working standards. This concentration does not always equal 1000 mg/L. The dilution factor relative to the stock standard is provided to aid in the calculations, where:

[True Working] = [True Stock] / dilution factor

7.4 Intermediate Standard I, 50 mg/L (20x dilution from stock)

7.4.1 Pipette 5.0 mL 1,000 mg/L calibration stock standard (Section 7.1) into a 100 mL volumetric flask.

7.4.2 Dilute to volume with 1% (0.25 N) sodium hydroxide. Prepare every 7 days.

7.5 Calibration Standards

7.5.1 Working Standard, 1.0 mg/L
Pipette 2.0 mL intermediate standard I (Section 7.4) into a 100 mL volumetric flask.

7.5.2 Dilute to volume with 1% (0.25N) sodium hydroxide and mix. Prepare daily.

7.5.3 Initial Calibration Standards
Dilute the 1.0 mg/L cyanide working standard with 1% (0.25N) sodium hydroxide as follows:

Standard Level	Intermediate	Aliquot (mL)	Final Volume (mL)	Concentration (mg/L)
1	1 mg/L	10.0	25	0.40
2	1 mg/L	10.0	50	0.20
3	1 mg/L	5.0	50	0.10
4	0.1 mg/L	2.0	4.0	0.05
5	1 mg/L	1.0	50	0.02
6	1 mg/L	0.5	50	0.01
7	N/A	0	50	0.00 (Blank)

7.5.4 Calculate the exact concentration for each calibration curve standard by dividing the exact working cyanide standard concentration by the dilution factors shown above if stock concentration is different from 1.0 mg = 1.0 ml.

7.6 Continuing Calibration Verification Standard (CCV), 0.2 mg/L:
The level 2 calibration standard described in 7.5 is used as the working CCV standard.

7.7 Second-Source Standards

7.7.1 Initial Calibration Verification (ICV) Stock Standard, 1,000 mg/L: The second-source standard is obtained from a different source than the calibration standards. It is prepared and standardized as described in Section 7.2 for the primary standard. This standard is available commercially.

7.7.2 Intermediate ICV (second-source) Standard, 10 mg/L: Pipette 1.0 mL 1,000 mg/L ICV (7.7.1) stock into a 100 mL volumetric flask. Dilute to volume with 1% (0.25N) sodium hydroxide.P

7.7.3 Prepare every 7 days.Working ICV (second-source) Standard, 0.10 mg/L

7.7.4 Spike 1 mL of the 10 mg/L intermediate (7.7.2) into a 100 mL volumetric flask and fill to the mark with 1% (0.25N) NaOH.

7.8 Pyridine-Barbituric Acid Solution (per OI Manual)

7.8.1 In a hood, place 15 g barbituric ($C_4H_4N_2O_3$) acid in a 1000 mL volumetric flask and add about 100 mL deionized water, rinsing down the sides of the flask.

7.8.2 Place on a magnetic stirrer and add a stir bar.

7.8.3 Add 75 mL pyridine while mixing.

7.8.4 Carefully add 15 mL concentrated hydrochloric acid while mixing.

7.8.5 Add 500 mL of DI water and stir until the barbituric acid is dissolved.

7.8.6 Dilute to volume with deionized water and filter through a 0.45 µm filter.

7.8.7 Store in an amber bottle.

7.8.8 Expires 6 months from preparation.

7.9 Phosphate Buffer Solution 1M (per OI Manual)

Dissolve 138 g sodium dihydrogen phosphate monohydrate ($NaH_2PO_4 \cdot H_20$) in deionized water and dilute to 1000 mL.

7.9.1 Add 4 mL of Brij-35 to the solution and mix gently.

7.9.2 Store at 4°C.

7.9.3 Filter the solution through a 0.45 µm filter.

7.9.4 This solution expires 3 months from preparation.

7.10 Chloramine-T (per OI Manual)

7.10.1 Dissolve 1.0 g Chloramine-T in deionized water and dilute to 250 mL.

7.10.2 Prepare fresh daily.

7.11 Sodium Hydroxide, 10N

7.11.1 Dissolve 400 g sodium hydroxide in deionized water.

7.11.2 Cool to room temperature, dilute to 1000 mL, and mix well.

7.11.3 Store in a plastic bottle. Solution is commercially available.

7.12 Sodium Hydroxide, 2% wt/wt (0.5N)

7.12.1 Dissolve 20 g sodium hydroxide in deionized water.

7.12.2 Cool to room temperature, dilute to 1000 mL, and mix well. Store in a plastic bottle.

7.13 Sodium Hydroxide Dilution Solution, 1% wt/wt (0.25N)

7.13.1 Dissolve 10 g sodium hydroxide in deionized water.

7.13.2 Cool to room temperature, dilute to 1000 mL, and mix well. Store in a plastic bottle.

7.14 Sulfuric acid, concentrated.

7.15 Sulfuric acid, 0.02 N

7.15.1 In a 2000 mL volumetric flask, carefully add 1 mL concentrated sulfuric acid to approximately 1900 mL deionized water.

7.15.2 Dilute to final volume of 2000 mL with deionized water and mix. Solution is commercially available.

7.16 Bleach
Fragrance free commercial liquid bleach is purchased and used in place of the Calcium Hypochlorite solution.

7.17 Magnesium Chloride solution
This reagent is purchased though an approved vendor.

7.18 Acetate buffer

7.18.1 Dissolve 410 g sodium acetate trihydrate in approximately 150 mL deionized water.

7.18.2 Adjust the pH to 4.5 with glacial acetic acid and dilute to final volume of 500 mL with deionized water.

7.19 Ascorbic acid crystals.

7.20 pH test strips.

7.21 Lead acetate test paper.

7.22 Potassium iodide-starch test paper.

7.23 Boiling chips.

7.24 Sulfamic Acid, 10% wt/wt
Dissolve 10 g sulfamic acid in 100mL of deionized water. Mix well. Expires 1 year from preparation.

7.25 Brij-35 Start-Up Solution
Concentrated Brij-35 is a buffer solution obtained from the equipment vendor. The start-up solution is prepared by diluting 4 mL of the Brij-35 concentrate to 500 mL with reagent water.

7.26 Bismuth Nitrate

7.26.1 Obtain a clean, dry 100 mL volumetric flask.

7.26.2 Add approximately 20 mL of DI water to the flask.

7.26.3 Add 3 g $Bi(NO)_3 \cdot 5H_2O$ to the flask and swirl to dissolve.

7.26.4 Slowly add 25 mL of glacial acetic acid, swirling frequently.

7.26.5 Swirl until completely dissolved.

7.26.6 Bring to volume with DI water.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters[1]	HDPE, Glass	500 mLs	NaOH, pH > 12; Cool 4 ± 2°C	14 Days	40 CFR Part 136.3

[1] Add 1.2 g of ascorbic acid per liter of sample if residual chlorine is present.

9.0 Quality Control

9.1 Sample QC: The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit or < 5% of sample concentration
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4], but no more than 85-115% recovery.
Matrix Spike (MS)[2]	1 in 10 or fewer samples	Statistical Limits [4],but no more than 75-125% recovery.
MS Duplicate (MSD)[2]	1 in 10 or fewer samples	Statistical Limits [4],but no more than 75-125% recovery.
High Distilled Standard (HDS) (0.4 mg/L)	1 in 20 or fewer samples	± 10% of true value
Low Distilled Standard (LDS) (0.10 mg/L)	1 in 20 or fewer samples	± 10% of true value

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client....predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.2 Method blank: Add 50 mL of 1% (0.25N) NaOH into a distillation flask immediately prior to distillation. For solid samples, weigh 1.0 g of Ottawa Sand into a distillation flask and add 50 mL 1% (0.25N) NaOH.

Corrective Action: The corrective action for method blank failures is re-distillation and reanalysis of all samples in the batch. If there is insufficient sample for reanalysis, a Nonconformance Memo must be prepared and the client contacted by the laboratory Project Manager.

9.3 LCS: Spike 5 mL of the 1.0 mg/L intermediate standard (7.5.1) into a distillation flask filled with 50 mL 1% (0.25N) NaOH. For solid samples, weigh 1.0 g of Ottawa Sand into a distillation flask and add 50 mL 1% (0.25N) NaOH.

Corrective Action: If the LCS fails, redistill and reanalyze all samples in the batch. If reanalysis is not possible, a Nonconformance Memo must be prepared and the client contacted by the laboratory Project Manager. See the TestAmerica Denver Policy, Quality Assurance Program, DV-QA-003P for additional guidance.

9.4 MS/MSD: Measure 50 mL of sample into a distillation flask. Spike the aliquot with 5 mL of the 1.0 mg/L cyanide standard (7.5.1). The matrix spike and matrix spike duplicate are prepared in the same manner. Both the matrix spike and matrix spike duplicate are taken through the distillation and analysis process.

Corrective Action: If MS/MSD recoveries exceed LIMS historical limits for total cyanide, the Method of Standard Additions may be employed to analyze all samples associated with a particular QC batch. Consult your Supervisor, a Technical Specialist, QA Manager, or if contractually required, the client before proceeding. Because MS/MSD results may not have a direct bearing on other samples in the batch, the appropriate corrective action is generally governed by specific project requirements. At a minimum, QC failures will be noted as anomaly and discussed in the final report.

9.5 High and Low Distilled Standards (HDS & LDS), 0.4 mg/L and 0.10 mg/L
Standards are distilled to monitor the efficiency of the distillation process. See section 7.5 for preparation instructions.

Corrective Action: Distilled standard failure results in re-distillation and reanalysis of all associated samples. One possible exception is the situation in which recoveries are greater than 110% and cyanide was not detected in the samples. In that case, a Nonconformance Memo should be prepared and the failure noted in the report together with the sample results without taking other corrective action.

9.6 Instrument QC

9.6.1 Initial Calibration Verification (ICV)

9.6.1.1 Immediately after the initial calibration, the calibration is verified using a second-source ICV standard and an initial calibration blank ICB (1% NaOH).

9.6.1.2 The measured result for the ICV must be within 10% of the expected value, and the ICB must be less than the reporting limit.

Corrective Action: If these criteria are not met, check the accuracy of the standards and recalibrate.

9.6.2 Continuing Calibration Checks (CCV / CCB)

9.6.2.1 A blank CCB (1% NaOH) and standard check CCV (see preparation in Section 7.6) are required after every 10 or fewer samples and at the end of the run.

9.6.2.2 Standard checks (ICV/CCV) must be within 10% of the expected value.

9.6.2.3 Blanks must be less than the reporting limit.

Corrective Action: If either continuing calibration check fails, all samples since the last successful calibration check must be reanalyzed.

10.0 Procedure

10.1 Sample Preparation

10.1.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, and chemistry, sample size, or other parameters.

10.1.2 Any variation in procedure shall be completely documented using a Nonconformance Memo (see SOP# DV-QA-0033) and is approved by a Technical Specialist and QA Manager.

10.1.3 Depending on the severity of the change and prior arrangements, the client shall be notified.

10.1.4 Check aqueous samples for sulfide prior to distillation using lead acetate paper.

10.1.4.1 Moisten the paper with 2 or 3 drops of acetate buffer, and then place 1 drop of sample on the paper.

10.1.4.2 A dark color indicates a positive test for sulfide.

10.1.4.3 Record the result as "positive" or "negative" on the bench sheet.

10.1.5 Samples that test positive for sulfide by lead acetate test paper must be set aside and distilled in a separate analytical batch (QC lot).

10.1.5.1 The entire QC lot (including quality control samples and a calibration curve containing a minimum of 3 standards) is treated for sulfide interference with bismuth nitrate (7.26) according to the method of standard addition.

10.2 Cyanide Amenable To Chlorination Sample Preparation

10.2.1 Two sample aliquots are required for the determination of cyanide amenable to chlorination. The first aliquot is distilled for total cyanide (see Section 10.3), the second aliquot is chlorinated under an alkaline condition prior to distillation and is used to determine cyanide not amenable to chlorination.

10.2.1.1 Check the pH of samples with pH test strips. Record the results on the bench sheet.

10.2.1.2 Measure the sample aliquots to be chlorinated (including all quality control samples) into 100 mL beakers.

10.2.1.3 For water samples, use 50 mL of sample.

10.2.1.4 For soil samples, use 1.0 g of sample and add 50 mL 1% (0.25N) NaOH.

10.2.1.5 Clearly label the samples with the proper identification and "chlorinated" as appropriate.

Note: The chlorination process must be performed in a fume hood.

10.2.1.6 Adjust the pH of the samples in the beakers to between 11 and 12 with the 10 N Sodium hydroxide solution.

10.2.1.7 Test the samples with KI-Starch paper and add bleach solution drop-wise to sample while mixing until a positive test is obtained. A positive test is indicated by a blue or black color on the paper.

10.2.1.8 Maintain the excess chlorine level in the sample for 1 hour while keeping the pH of the samples between 11 and 12 with constant mixing (use a magnetic stirrer). Add bleach solution and sodium hydroxide as necessary.

10.2.1.9 After 1 hour, add 0.1 g portions of ascorbic acid crystals until a negative test is obtained with KI-Starch paper. Add an additional 0.1 g of ascorbic acid crystals to the sample to ensure an excess of the reagent

10.2.1.10 Transfer the contents of the beakers into distillation flasks quantitatively, rinsing with deionized water.

10.2.1.11 Proceed to section 10.3 for the distillation process.

10.3 Total Cyanide Sample Preparation

10.3.1 Check the pH of aqueous samples with pH test strips. Record the results on the bench sheet.

10.3.2 Check aqueous samples for oxidizing agents such as chlorine.

10.3.2.1 Place one drop of sample on a strip of potassium iodide (KI)-starch test paper.

10.3.2.2 A blue color indicates the need for treatment.

10.3.2.3 Record the result as "positive" or "negative" on the benchsheet.

10.3.2.4 If a positive test is obtained, add a few crystals at a time of ascorbic acid until a drop of sample produces no color on the indicator paper.

10.3.2.5 Then add an additional 0.1 g of ascorbic acid in excess.

10.3.3 Measure sample aliquots into the distillation flasks as follows:

- For water samples use 50 mL of sample.
- For solid samples use 1.0 g of sample and add 50 mL 1% (0.25N) NaOH.

10.3.4 Place 25 mL 2% sodium hydroxide into the absorption tubes.

10.3.5 Assemble the distillation apparatus. All distillations are to be performed under the slot hood.

10.3.6 Turn on the vacuum pump and chiller. Also be sure that the slot hood is operating.

10.3.7 Adjust the vacuum to provide a flow rate of approximately 2-3 bubbles per second (i.e., this is approximately 1/8-1/4 inch of foam in the scrubber) in the distillation flask.

10.3.8 Verify that there are no leaks in the system by observing the flow into the absorber tube. The flow rate may not remain constant during the distillation; readjust as necessary.

10.3.9 If the test for sulfide is positive, add 5 mL of 0.062 M bismuth nitrate solution (7.26) through the thistle tube to every sample and QC sample in the analytical batch.

10.3.9.1 Samples testing positive for sulfide are distilled and analyzed as a separate analytical batch.

10.3.9.2 The samples are analyzed using the method of standard addition utilizing a 2-point spike and calculating the sample concentration using an EXCEL spreadsheet application.

10.3.10 Allow to mix for 3 minutes.

10.3.11 Add 2 mL of 10% sulfamic acid solution (7.24) through the thistle tube. Allow to mix for 3 minutes.

10.3.12 Slowly and carefully, add 2.5 mL concentrated sulfuric acid through the thistle tube.

10.3.13 Rinse the tube with a little deionized water and allow to mix for 3 minutes.

10.3.14 Add 2 mL of magnesium chloride solution (7.17) and mix. If excessive foaming is observed, add additional magnesium chloride.

10.3.15 Turn on the controller and heat the samples to boiling.

10.3.16 While distilling the samples, carefully watch to make sure that vacuum is maintained on all of the stills. Adjust the flow as necessary.

10.3.17 Allow samples to reflux for 1.5 hours.

10.3.18 After 1.5 hours of refluxing, allow the samples to cool for 15 minutes while air is flowing.

10.3.19 Remove the absorption tube from the distillation apparatus. Rinse the inside and outside of the bubbler into the tube with deionized water.

10.3.20 Remove the flask and rinse their contents down the drain with running water. Residue not removed by this method must be scrubbed out.

10.3.21 Dilute the sodium hydroxide in the absorption tube to 50 mL with deionized water and store in plastic vials.

10.3.22 Place each batch of distillates in a box and store the samples at 4°C until they are analyzed.

10.3.23 At the end of the day, turn off the hood, vacuum, and chiller.

10.3.24 Proceed to Section 10.4 for colorimetric analysis of the distillates.

10.4 Instrument Set-Up & Calibration

10.4.1 Instrument Stabilization (per Alpkem, OI Company, manual)

10.4.1.1 Connect the reagent pump tubes to a reagent bottle containing the start-up solution (7.25). Start the pump, allowing the start-up solution to flow through the entire system.

10.4.1.2 Make sure that the flow cell of the detector is purged of all bubbles and the flow is stable and free from surging.

10.4.1.3 Once a stable flow is achieved, connect the reagent pump tubes to their respective reagent bottles, as shown in the schematic in Appendix IV. Allow the reagents to flow through the entire system, then, once again, verify that the flow cell of the detector is purged of all bubbles.

10.4.2 Initial Calibration

10.4.2.1 Calibration is performed daily or each time the instrument is set up using the standards shown in Section 7.5.

10.4.2.2 A minimum of five standards and a blank are required for the calibration (the high standard may be dropped if needed and sample dilutions performed appropriately).

Note: If sulfide was detected during the sample preparation step and the samples are logged for 9010B/9012A, then bismuth nitrate must be used to precipitate the sulfide. A method of additions spike must be prepared, and all calibration standards must be treated and distilled in the same manner as the samples. A minimum of five standards and a blank shall be distilled. Use the same calibration levels as shown in the table above, Section 7.5.

10.4.2.3 The calibration function is calculated by least-squares linear regression, and the correlation coefficient must be > 0.995 and the absolute value of the intercept must be less than ± 1/2 the reporting limit.

Corrective Action: If the correlation coefficient is < 0.995 or the absolute value of the intercept is too large, locate

and correct the problem and re-calibrate the instrument.

Note: If the standard curve for samples with sulfide (distilled in same manner as the samples) is not acceptable, the QC batch must be reprepared. If the second preparation does not give an acceptable calibration curve, the samples may be reanalyzed on an undistilled calibration curve with a discussion in the final report narrative.

10.4.2.4 The peak height of the synchronization (sync) standard should be > 150,000.

Corrective Action: If the peak height is < 150,000, the flow cell of the instrument must be cleaned (consult manufacturer's instructions), and then the instrument must be recalibrated

10.4.2.5 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

10.4.2.6 Initial Calibration Checks: Immediately after the initial calibration, the calibration is verified using a second-source, initial calibration verification (ICV, see preparation in Section 7.7) standard and an initial calibration blank (ICB, 1% NaOH). The measured result for the ICV must be within 10% of the expected value, and the ICB must be less than the reporting limit.

Corrective Action: If these criteria are not met, check the accuracy of the standards and recalibrate.

10.4.2.7 Continuing Calibration Checks: A blank CCB (1% NaOH) and standard check CCV (see preparation in Section 7.6) are required after every 10 or fewer samples and at the end of the run. Standard checks (ICV/CCV) must be within 10% of the expected value. Blanks must be less than the reporting limit.

Corrective Action: If either continuing calibration check fails, all samples since the last successful calibration check must be reanalyzed.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	≥0.995 correlation coefficient
Initial Cal. Verification (ICV)	Immediately after the calibration	± 10% of the expected value
Initial Cal. Blank (ICB)	Immediately after the calibration	Less than the reporting limit
Cont. Cal. Verif. (CCV)	Prior to / after every 10 injections	± 10% of the expected value

Cont. Cal. Blank (CCB)	Prior to / after every 10 injections	Less than the reporting limit
Matrix Spike & Matrix Spike Duplicate (MS/MSD)	1 in 10 or fewer samples	≤ 20% RPD

10.5 Sample Analysis

Following instrument set up and calibration, the sample distillates are analyzed in exactly the same manner as the calibration standards. The routine run sequence is as follows:

Cal 0.00 ppm
Cal 0.01 ppm
Cal 0.02 ppm
Cal 0.05 ppm
Cal 0.10 ppm
Cal 0.20 ppm
Cal 0.40 ppm
Second-source ICV
ICB
High concentration distilled standard
Low concentration distilled standard
LCS / LCSD
Method blank
6 samples (may include MS/SD)
CCV
CCB
10 samples (may include MS/SD)
CCV
CCB
Additional cycles of 10 samples with CCV/CCB
Closing CCV
Closing CCB

10.6 Instrument Shut-Down

10.6.1 Disconnect the reagent lines and put all of them into the DI water, except the buffer line.

10.6.2 The Buffer line should be put into the bridge.

10.6.3 Rinse all tubes for at least 5 min.

10.6.4 Rinse with Kleenflow Base for 5 min.

10.6.5 Rinse tubes with appropriate rinses for 5 min.

10.6.6 Turn off instrument and pump.

10.6.7 Raise platens.

10.6.8 Empty Waste

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{ICV / CCV, LCS/ HDS, LDS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Total Cyanide Calculation:

All routine calculations for total cyanide are performed by the instrument data system, provided dilutions and other information have been correctly entered.

11.4 Cyanide Amenable to Chlorination:

Amenable Cyanide = Total CN Unchlorinated Result – Treated Result

If the chlorinated aliquot shows more cyanide than the unchlorinated aliquot, a corrective action and/or a discussion in the final report is required. Iron-cyanides can cause this to occur. Weak acid dissociable cyanide would be a better method for these types of samples.

11.5 Reporting Final Results:

Final results are routinely reported in mg/L for aqueous samples and in mg/kg for solid samples. Results can also be reported as ug/L or ug/kg if there are special project instructions requiring it.

Note: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.6 All data are subject to two levels of review, which is documented on the checklist shown in Attachment 2.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

12.1.1 Prepare seven samples at three to five times the estimated MDL concentration.

12.1.2 Digest and analyze the MDL standards as described in Section 10.

12.1.3 Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte.

Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P) for each analyte.

12.1.4 MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS. See Policy # DV-QA-005P for further details concerning MDL studies.

12.1.5 The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities:

All personnel are required to perform an initial demonstration of capability (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

12.2.1 Four aliquots of the QC check sample (independent source from the calibration) are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration standard.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use, has the required experience, and has successfully analyzed initial demonstration samples (see SOP # DV-QA-0024 for details).

13.0 Pollution Control

13.1 In general, the quantity of chemicals purchased by Test America Denver is based on expected usage during its shelf life. The volume of reagents and standards prepared for this procedure reflects anticipated usage.

13.2 Bismuth Nitrate is substituted for cadmium carbonate in the procedure.

13.3 Source reduction is achieved through the use of micro-distillation followed by an automated colorimetric determination.

13.4 The volume of hazardous waste is minimized through proper segregation and management of the various waste streams generated by this procedure

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

14.2 The following waste streams have been identified for this method:

- Cyanide standardization waste – Aqueous Alkaline (E)
- Distilled sample – Aqueous Acidic (F)
- Distillate – Aqueous Alkaline (E)
- Alpkem process waste – Aqueous Alkaline, contains pyridine (E)
- Contents of sampler cups – Aqueous Alkaline (E)
- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

Note: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

- Method 9010C, Total and Amenable Cyanide: Distillation, Revision 3, August 2002.
- Method 9012B, Total and Amenable Cyanide (Automated Colorimeteric, with Off-Line Distillation), Revision 2, August 2002.
- Method 9013, Cyanide Extraction Procedure for Solids and Oils, Revision 0, July 1992.

16.0 Method Modifications:

Item	Method	Modification
1	SW 9012A	Method 9012A states the amenable cyanide test must be performed under amber light. This SOP method is performed under normal laboratory conditions.
2	SW 9012A	The reflux time for Cyanide Amenable to Chlorination and Total Cyanide has been changed to 1.5 hours versus 1.0 hours to accommodate the reflux time for samples requiring distillation under the Clean Water Act (EPA Method 335.4).

Item	Method	Modification
3	SW 9012A	Calibration is verified with an independently prepared check standard (ICV) with every analytical run and a CCV is run after every 10 samples, instead of for every 15 samples.
4	SW 9012A	There are differences among the referenced methods concerning the sodium hydroxide concentration in working standards: Method 9012A states in Section 7.4.1 that calibration standards are prepared using 50 mL of 1.25N sodium hydroxide and diluting to 250 ml, which produces a 0.25N sodium hydroxide concentration. To be sure that the standards are stable, the working standards in this SOP are prepared in 0.25N.
5	SW 9012A	The Cyanide stock standard is verified upon receipt and monthly thereafter. The standard is generally depleted long before the average expiration date of 6 months. The monthly verifications are sufficiently frequent to monitor the concentration.

17.0 Attachments

Attachment 1: Cyanide Preparation Example Bench Sheet
Attachment 2: Data Review Checklist
Attachment 3: Cyanide Distillation Apparatus
Attachment 4: Alpkem Manifold Schematic

18.0 Revision History

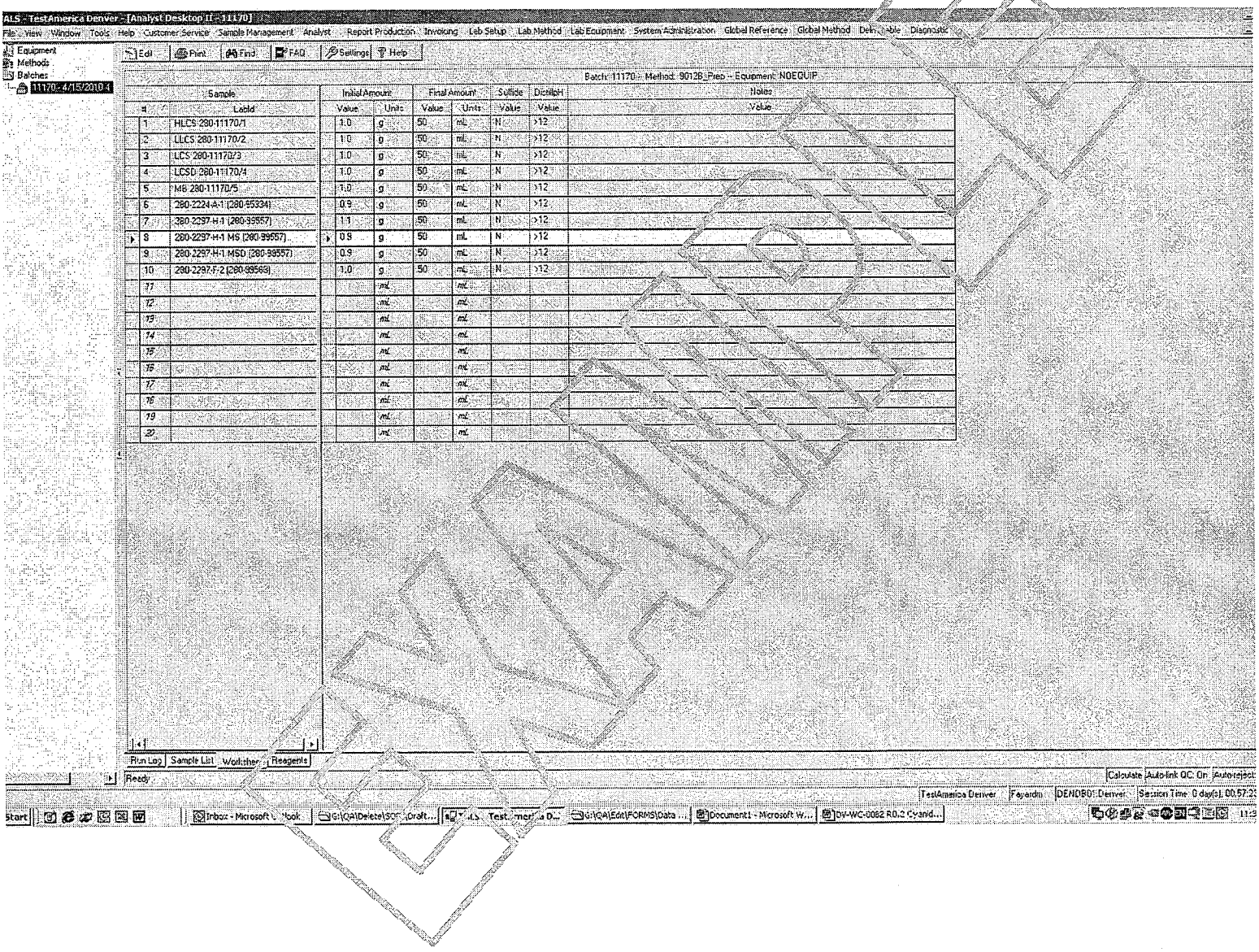
- Revision 0.2, dated 11 June 2010,
  - Annual Technical Review.
  - Updated Attachments 1 & 2.

- Revision 0.1, dated 12 June 2009, updated to changed calibration criteria to require that the intercept be less than the absolute value of the reporting limit.

- Revision 0, dated 27 February 2009

Attachment 1.

Cyanide Preparation Example Bench Sheet



Attachment 2.

Data Review Checklist

TESTAMERICA Denver

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Lot / Sample Numbers	Matrix	Prep Batch	Batch	Method	Special List

	Yes	No	N/A	2nd Level
. Calibration/Instrument Run QC				
. Minimum of five standards in ICAL as specified in method?				
. Correlation coefficient ≥ 0.995 ?				
. Second-source ICV analyzed, and recovery within acceptance limits?				
. ICB analyzed immediately after the ICV & results < the RL				
. CCV analyzed after every ten samples & recovery within acceptance limits?				
. CCB analyzed after every CCV & results < RL?				
. Absolute value of the intercept is < ½ the RL?				
. Sample Results				
. All samples greater than highest calibration standard diluted and reanalyzed?				
. Do associated RLs/MDLs reflect dilutions or limited sample volume?				
. All reported results bracketed by in control CCV results?				
. Sample analyses done within holding time?				
. Initial pH check documented for all samples? (If Applicable)				
. Preparation benchsheet completed and included in package?				
. Client requirements reviewed and met?				
. Were data manually transcribed from instrument printouts into TALS verified 100% including significant figures and correct units? (If Applicable)				
. Do the prep and analysis dates in TALS reflect the actual dates?				
1. Raw data copies prepared, scanned, and uploaded?				
2. Manual integrations done properly and initialed and dated?				
3. STD/True Value information is updated and included?				
. Preparation/Matrix QC				
. Method blank < RL or all reported samples > 10x blank have NCM?				
. Method blank < ½ RL or NCM provided?				
. LCS/LCSD run for batch and within QC limits?				
. MS/MSD run at required frequency and within limits or NCM written?				
. DUP run at required frequency and RPD within acceptance limits or NCM written?				

Analyst: _____ **Date:** _____

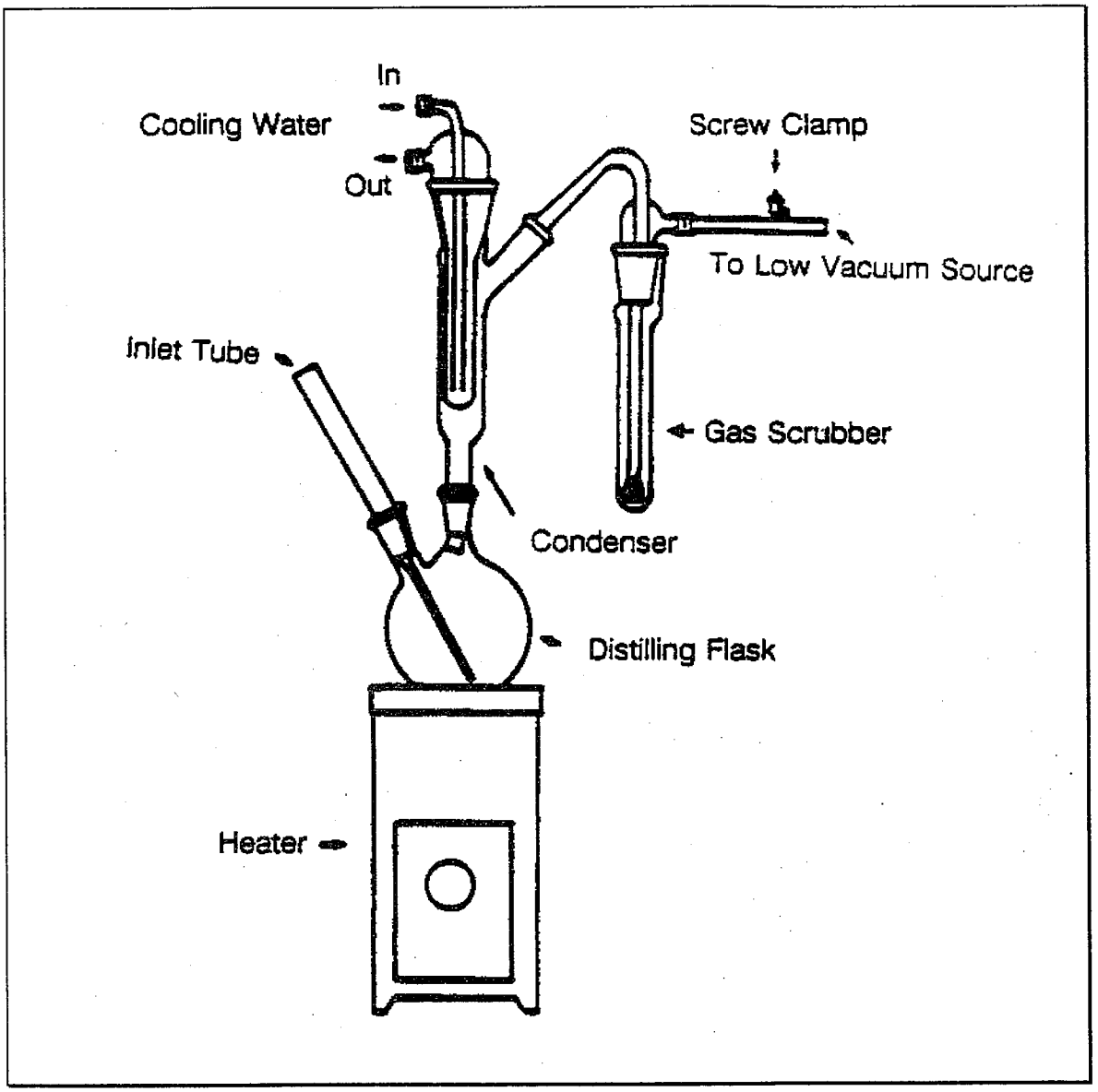
_____

_____

2nd Level Reviewer : _____ **Date:** _____

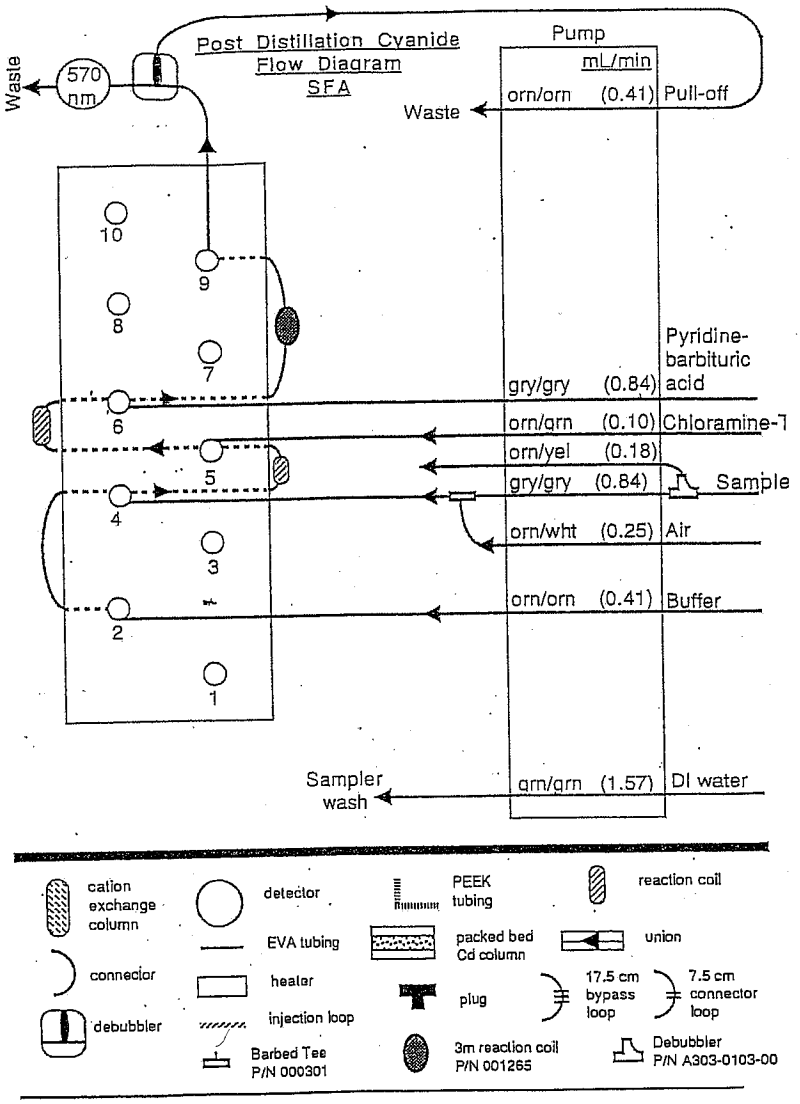
Attachment 3.

Cyanide Distillation Apparatus



Attachment 4.

Alpkem Manifold Schematic



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Title: Total Phenols, Automated Methods [EPA 420.2, EPA 420.4, SW 9066]

Approvals (Signature/Date):

Dave Elkin — Date: 5/24/10
Wet Chemistry Supervisor

Adam Alban — Date: 26 May 10
Health & Safety Manager / Coordinator

Karen Kuoppala — Date: 5-26-10
Quality Assurance Manager

Robert C. Hanisch — Date: 5/27/10
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the determination of total phenols that can be distilled and subsequently measured by the 4-aminoantipyrine (4-AAP) colorimetric method.

1.2 This procedure is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes, and soil samples.

1.3 The method determines phenol, ortho- and meta- substituted phenols, and under proper pH conditions, those para- substituted phenols in which the substitution is a carboxyl halogen, methoxyl, or sulfonic acid group. It does not determine those para- substituted phenols where the substitution is an alkyl, aryl, nitro, benzoyl, nitroso, or aldehyde group. The method does not differentiate between different types of phenol.

1.4 The routine working range for aqueous samples is 0.02 mg/L to 0.5 mg/L for the automated analysis and 0.005 mg/L to 0.1 mg/L for the chloroform extraction (manual) method. The routine working range for soil samples is 2.0 mg/kg to 50 mg/kg for the automated analysis and 0.5 mg/kg to 10 mg/kg for the chloroform extraction (manual) method. The working range can be extended by dilution of the distilled sample.

2.0 Summary of Method

2.1 The sample is acidified and distilled to separate phenolics from nonvolatile interfering compounds. A measured volume of an aqueous sample is distilled. A weighed aliquot of a solid sample is placed into a measured volume of water for the distillation. Phenolic compounds in the distillate react with 4-aminoantipyrene (4-AAP) in the presence of alkaline potassium ferricyanide to form a reddish-brown antipyrine dye.

2.2 The antipyrine dye formed by the reaction between the steam-distillable phenols and the 4-AAP is kept in the aqueous solution and the absorbance is measured at 505 nm.

3.0 Definitions

3.1 Total Phenols: The hydroxy derivatives of benzene and its condensed nuclei.

3.2 Ortho-substituted: Prefix used in organic chemistry in naming disubstitution products derived from benzene in which the substituent atoms or radicals are located in adjacent positions on the benzene ring. This is also called the 1,2 position. The ortho position on a phenol molecule is adjacent to the hydroxyl group.

3.3 Meta-substituted Phenols: Prefix used in organic chemistry in naming disubstitution products derived from benzene in which the second substituent atom or radical is located on the third carbon atom with respect to the first substituent. This is also called the 1,3 position. The meta position on a phenol molecule is separated from the hydroxyl group position by one carbon.

3.4 Para-substituted: Prefix used in organic chemistry in naming disubstitution products derived from benzene in which the substituent atoms or radicals are located in opposite positions on the benzene ring. This is also called the 1,4 position. The para position on a phenol molecule is opposite the hydroxyl group.

4.0 Interferences

4.1 Color response of phenolic materials with 4-aminoantipyrine is not the same for all phenolic compounds. Because phenolic-type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this

reason phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds present in the sample.

4.2 Most chemical interferences are eliminated by distillation of an acidified sample. However, some phenolic compounds are not steam-distillable and are not included in this analysis.

4.3 Many interferences from sulfur compounds (such as sulfide, thiosulfate, and sulfite from certain industrial treatment samples), phenol-decomposing bacteria, oxidizing and reducing substances, and alkaline pH can be eliminated by acidification and aerating briefly by stirring. Sulfite interferes but is not eliminated by this treatment.

4.4 Oxidizing agents such as chlorine will oxidize phenolic compounds and are removed by the addition of excess ferrous ammonium sulfate. If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.

4.5 Oils can distill and interfere with the analysis. The water phase can be separated and analyzed separately. The oil phase may have to be analyzed by other methods. In any case, the client should be consulted.

4.6 Aromatic amines may react with nitrite (if present) to produce phenolic compounds.

4.7 Background contamination from plastic tubing and sample containers is eliminated by filling the wash receptacle by siphon and use of glass tubes for the samples and standards.

4.8 Method interference may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The use of separatory funnels to extract aqueous samples with chloroform creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggle when it is performed.

5.1.3 Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.1.4 This method uses strong oxidizers, which can cause severe burns and tissue destruction.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phenol	Corrosive	5 ppm (TWA)	Breathing vapor, dust or mist results in digestive disturbances. Will irritate, possibly burn respiratory tract. Rapidly absorbed through the skin with systemic poisoning effects to follow. Discoloration and severe burns may occur, but may be disguised by a loss in pain sensation. Eye burns with redness, pain, blurred vision may occur. May cause severe damage and blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Phosphoric Acid	Corrosive	1 mg/m^3 (TWA)	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Hydroxide	Corrosive	2 mg/m3 (Ceiling)	Sever irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and with greater exposures, it can cause burns that may result in permanent impairment of vision, even blindness.
Ammonium Hydroxide	Corrosive Poison	50 ppm (TWA)	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Potassium Ferricyanide	Irritant	None	**This material will form hydrogen cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. However it does not break down into cyanide compounds in the body.** May cause irritation to the respiratory tract, skin and eyes

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

- Balance, analytical, capable of measuring to ± 0.0001 g. The accuracy of the balance is checked each day before use in accordance with SOP DV-QA-0014.
- Recirculating chiller.
- pH meter and electrode, with automatic temperature correction
- Alpkem Flow Solution IV autoanalyzer, which includes sampler, pump, colorimeter, 505 nm filter, 10 mm flowcell, and software.

6.2 Supplies

- All-glass distillation apparatus consisting of 500 mL round-bottom flask with side arm, coil condenser, heating mantle with controller, and associated adapters and hardware. Kontes midi distillation system is used to distill reduced volume for the modified procedure to the method.
- Eppendorf Pipettes, varying volumes
- Volumetric (Class A) glassware: varying volumes.
- Boiling stones.
- pH test strips
- Starch/iodide test strips

6.3 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent water (ASTM type II or equivalent), distilled or deionized water, free of the analytes of interest.

7.2 Sulfuric Acid, 50%:

Very slowly add 500 mL of concentrated sulfuric acid to 500 mL of deionized water with constant mixing, and allow the solution to cool completely before handling.

CAUTION: The reaction is very exothermic and should be done with extreme caution.

7.3 Sulfuric Acid, 1.0 N

This solution may be obtained from commercial sources. If prepared in the laboratory, slowly, carefully, and with stirring, add 2.78 mL of concentrated sulfuric acid to approximately 80 mL of deionized water. Dilute to 100 mL with deionized water. Allow to cool before use.

7.4 Phosphoric Acid Solution, 8.5%

Carefully add 10 mL of 85% phosphoric acid to approximately 80 mL of deionized water and bring to a final volume of 100 mL with deionized water.

NOTE: This solution is used ONLY if samples have been preserved with phosphoric acid, and the pH needs to be adjusted. Otherwise, this solution is not routinely used as part of this SOP.

7.5 Ferrous Ammonium Sulfate Solution

Add 1 mL of concentrated sulfuric acid to 500 mL deionized water. Add 1.1 g of ferrous ammonium sulfate, mix until dissolved, and dilute to 1000 mL.

7.6 Sodium Hydroxide, 2.5N

Dissolve 10 g of sodium hydroxide in approximately 80 mL of deionized water. Cool and dilute to 100 mL. Store in a plastic bottle.

7.7 Sodium Hydroxide, 1N

Dissolve 40 g of sodium hydroxide in approximately 800 mL of deionized water, cool, and dilute to 1000 mL with deionized water. Store in a plastic bottle.

7.8 Potassium Ferricyanide

Dissolve 1.0 g of potassium ferricyanide, 1.6 g of boric acid, and 1.88 g of potassium chloride in 400 mL of deionized water. Adjust the pH to 10.3 with 1 N sodium hydroxide, and dilute to 500 ml in a volumetric flask. Add 1.0 mL of Dowfax 2A1 and mix the solution gently. Prepare weekly.

7.9 4-Aminoantipyrine

Dissolve 0.065 g of 4-aminoantipyrine in approximately 80 mL of deionized water and dilute to 100 mL. Add 1.0 mL of Dowfax 2A1 and mix gently. Prepare daily.

7.10 Sampler Wash

Place deionized water under a strong vacuum for 15-20 minutes before use. Water may also be degassed with a stream of helium gas through a glass frit for approximately 5 minutes.

7.11 Dowfax Start-up Solution

To approximately 400 mL of deionized water, add approximately 1 mL of Dowfax 2A1. Dilute to 500 mL and mix gently.

7.12 Methyl Orange Indicator Solution

Dissolve 0.5 g of Methyl Orange in approximately 800 mL of deionized water. Dilute to 1 L with deionized water. This solution is also available commercially.

7.13 Phenol Stock Calibration Standard, 1000 mg/L

A commercially available standard is used.

7.14 Phenol Intermediate Calibration Standard, 10 mg/L

Dilute 1.0 mL of the 1000 mg/L Stock Calibration Standard (Section 7.13) to 100 mL with deionized water.

7.15 Working Calibration Standards

Dilute the 10 mg/L "automated" Intermediate Calibration Standard (Section 7.14) with deionized water as follows:

Automated Method Calibration Levels

Level	Volume of Intermediate Standard (mL)	Final Volume (mL)	Conc (mg/L)
1	2.0 of Level 2 standard	4.0	0.01

2	0.2	100	0.02
3	2.0 of Level 4 standard	4.0	0.05
4	1.0	100	0.1
5	2.0	100	0.2
6	5.0	100	0.5

NOTE: The standards are not distilled.

7.16 Phenol ICV Stock Standard, 1000 mg/L

This solution must be prepared using a standard obtained from a source different from the one that supplied the phenol calibration standard (Section 7.13). Dissolve 1.000 g of phenol in deionized water and dilute to 1000 mL. A commercially available standard can be used.

7.17 Phenol ICV Intermediate Standard, 10 mg/L

Dilute 1.0 mL of the 1000 mg/L ICV Stock Standard (Section 7.16) to 100 mL with deionized water.

7.18 ICV Standard

Dilute 1 mLs of the 10 mg/L ICV Intermediate Calibration Standard (Section 7.17) to 100 mL with deionized water. The true value is 0.1 mg/L.

7.19 Continuing Calibration Verification (CCV) Standard

Prepare the CCV in the same manner as the 0.2 mg/L calibration standard, as described in Section 7.15 As with the calibration standards, the CCV is not distilled.

7.20 Initial and Continuing Calibration Blank (ICB and CCB)

The ICB/CCB for the automated method is reagent water.

7.21 Phenol LCS Intermediate Standard, 10 mg/L

Dilute 1.0 mL of the 1000 mg/L Stock Standard (Section 7.14) to 100 mL with deionized water.

7.22 LCS Standard

Dilute 1 mLs of the 10 mg/L Intermediate Calibration Standard (Section 7.21) to 50 mL with deionized water. The true value is 0.2 mg/L. This standard is then distilled and analyzed in the same manner as a sample.

7.23 MS/MSD Standard

Dilute 1 mLs of 10 mg/L Intermediate Calibration Standard (Section 7.14) to 50 mL with the selected sample. The true value is 0.2 mg/L. These spiked samples are then distilled and analyzed in the same manner as all other samples.

7.24 Method Blank

One method blank (MB) must be processed with each batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[1]	Reference
Waters	HDPE	1 Liter	H_2SO_4, pH < 2; Cool 4 ± 2°C	28 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Sample QC - The following quality control samples are prepared with each batch of samples. See section 7 for standard preparation.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	90-110% for EPA 420.4
Matrix Spike (MS)[2]	1 in 10 or fewer samples	Statistical Limits [3]
MS Duplicate (MSD)[2]	1 in 10 or fewer samples	Statistical Limits [3]

Note: If all samples associated with a blank greater than the RL are greater than 10 times the blank value, the samples may be reported with an NCM to qualify the high blank value.

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client....predetermined by the extraction lab.
[3] Statistical control limits are updated annually and are updated into LIMS.

9.2 Instrument QC - The following quality control samples are prepared with each batch of samples (ICAL standards are prepared as listed). See section 7 for standard preparation.

Step	Standards	Type	Control Limit	Frequency
Initial Cal	6 Calibration standards. See section 7.18.1 for concentrations.	Linear	≥0.995 correlation coefficient	Initially or when continuing calibration verification fails acceptance criteria.
ICV/LCS	Initial calibration verification	Second source See section 7.21.1	90-110%	Initially or immediately after the ICAL.
ICB	Initial calibration blank	Reagent water	Less than RL	After the ICV.
CCV	Continuing Calibration Verification	See section 7.22.1	90-110%	Every 10 or fewer samples and after the last sample.
CCB	Continuing Calibration blank	Reagent water	Less than RL	After every CCV.
Linear Calibration Range	blank and three standards	Three calibration standards analyzed with analytical run	±10% of standards true value	Every six months

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.1 Sample Preparation

10.1.1 Measure and record the pH of each water sample. pH test strips may be used.

10.1.2 Samples from chlorinated sources must be checked for residual chlorine with starch/iodide test strips. A blue to black color indicates a positive test. Record the result on the bench sheet and generate an NCM anomaly for all positive results.

10.1.3 If the chlorine test was positive, add ferrous ammonium sulfate solution until a negative test is obtained.

10.1.4 Distillation for Automated Method

10.1.4.1 For the MIDI distillation setup, measure 50 mL of sample into a distillation flask and add a few boiling stones. For soil and waste samples, use 0.5 g and add 50 mL of deionized water. Record the exact weight on the bench sheet.

10.1.4.2 For all soil/waste samples, add 50% sulfuric acid drop-wise until the pH is < 4 and document it on the prep sheet.

10.1.4.3 Assemble the distillation apparatus, turn on the chiller water, and start the distillation. The distillate is captured in the receiving tube of the distillation rack, which has a 50 mL volumetric mark.

10.1.4.4 When 40 mL of distillate have been collected, turn off the heating mantle and allow to cool.

10.1.4.5 Add 5 to 10 mL of water to the distillation flask and resume distillation until nearly 50 mL have been collected.

10.1.4.6 Bring the distillate to a final volume of 50 mL with deionized water.

10.1.4.7 Turn off the heating mantle and clean out the flask when cool. Do not over-distill the samples as this will lead to interferences in the analysis.

10.1.4.8 Rinse condensers with deionized water several times between samples to ensure no contamination between samples.

10.1.4.9 At the end of the day, turn off the chiller.

10.1.4.10 Refrigerate distillates at 4 ± 2 °C until they are analyzed. Analysis should follow as soon as possible.

NOTE: If the distillate is turbid, filter through pre-washed membrane filter.

10.2 Calibration

10.2.1 Automated Method Initial Calibration (ICAL)

10.2.1.1 The ICAL is performed automatically by the instrument at the beginning of each analytical sequence.

10.2.1.2 Load the standards listed in Section 7.15 into the autosampler so that they are analyzed before any samples. The calibration standards are not distilled.

10.2.1.3 The instrument software processes the calibration data and generates a calibration function that is used to calculate sample

results. The instrument software uses a least squares linear regression to relate the concentration of phenol in each standard and the associated absorbance reading, as follows:

$$y = bx + a$$ **Equation 1**

Where:

y = Absorbance of standard at 505 nm.
x = Phenol concentration of standard, mg/L.
b = Slope of the fitted straight line.
a = Y-intercept of the fitted straight line.

10.2.1.4 The instrument software uses the calibration function to calculate the concentration of each sample that is analyzed. The instrument software prints a calibration report for review, which includes the calibration equation and correlation coefficient. The correlation coefficient of the calibration line must be ≥ 0.995. If this cannot be achieved, then check the calibration standards, correct any problems, and repeat the ICAL.

10.2.1.5 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

10.3 Sample Analysis

10.3.1 Automated Analysis

10.3.1.1 Place all lines in the Dowfax startup solution and place the wash lines into degassed deionized sampler wash water. Allow the system to flow for at least 10 minutes. Ensure that no leaks are present and that the flowcell of the detector is purged of all bubbles. Flow should be stable and free from surging. Ensure that the 505 nm interference filter is in place, and be sure that the lines from the autosampler and to the waste are moved to the appropriate waste container if another analysis was being performed on the instrument.

10.3.1.2 Place the reagents online and allow the system to pump for 10 to 15 minutes. Monitor the baseline at 505 nm and ensure that the baseline noise is low and that the baseline is not drifting up or down. If the baseline is drifting up or down, purge the flowcell of air bubbles before continuing.

10.3.1.3 Set up the sample table in the instrument software and load the standards and the sample and QC sample distillates into the autosampler in the order dictated by the sample table. If any sample is diluted, then make sure to enter the correct dilution factor into the instrument software. When loading, take care to ensure that the sample poured into each cup position is verified against the sample table to ensure accuracy. Following is a typical analytical sequence:

ICAL standards
ICV
ICB
Method Blank
LCS
7 samples (can include the MS and/or MSD)
CCV
CCB
10 samples (can include the MS and/or MSD)
CCV
CCB

10.3.1.4 Start the analytical sequence. Monitor the run carefully to ensure that QC and continuing calibration verification results meet the established acceptance criteria (see Sections 9). Any sample that displays a dip or a split peak should have its pH checked and adjusted to neutral before analysis.

10.3.1.5 Any sample that has a concentration that exceeds the highest standard must be diluted and reanalyzed. Samples analyzed directly after a highly concentrated sample may be adversely affected by carryover.

10.3.1.6 After the analytical sequence is done, flush the system thoroughly with deionized water and turn off the instrument. Be sure to release the pressure on the pump tubes.

10.3.1.7 Clean all glassware, apparatus and the work area.

11.0 Calculations / Data Reduction

11.1 Calculations

11.1.1 All required calculations are performed by the auto analyzer software, provided that dilutions and other information have been correctly entered.

11.1.2 The concentration in an unknown sample distillate is calculated by the instrument software by solving the calibration equation (Equation 1) for concentration (x) and using the measured absorbance of the sample, as follows:

$$x = \frac{y - a}{b}$$ **Equation 3**

Where:

x = Phenol concentration in sample distillate (mg/L).
y = Absorbance of the sample distillate at 505 nm, corrected for the blank.
b = Slope of the fitted calibration line.

a = Y-intercept of the fitted calibration line.

11.1.3 If a sample distillate was diluted, then the instrument software calculates a final result for an aqueous sample as follows (assuming that the analyst entered the correct dilution factor into the instrument software program):

$$C_s = x \times DF \qquad \textbf{Equation 4}$$

Where:

C_s = Phenol concentration in the original sample (mg/L).
x = Phenol concentration in sample distillate at the instrument (mg/L).
DF = Dilution factor.

11.1.4 For soil samples, the phenol concentration in the original sample is calculated as follows:

$$C_s = x \times \frac{V_d}{M_s} \times DF \qquad \textbf{Equation 5}$$

Where:

C_s = Phenol concentration in the original sample (mg/kg).
x = Phenol concentration in sample distillate at the instrument (mg/L).
V_d = Volume of distillate (L). For the automated method, this is usually 0.050 L (50 mL).
M_s = Mass of original sample aliquot (kg). For the automated method, this is typically 0.0005 kg (0.5 g).
DF = Dilution factor, if applicable.

11.1.5 Additional calibration information can be found in the Corporate TestAmeica's Calibration Curve document CA-Q-S-005.

11.2 Reporting

11.2.1 Reporting units are mg/L for water samples and mg/kg for soil samples.

11.2.2 The reporting limit for the automated method is 0.02 mg/L (2 mg/kg).

11.2.3 The reporting limit for the manual method is 0.005 mg/L (0.5 mg/kg).

11.2.4 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor, and the data may require flagging.

11.2.5 All associated data are entered into the LIMS as required.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in TestAmerica Denver's Policy DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal

laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

An initial demonstration of capability for each method must be performed prior to analyzing samples.

- For the standard analyte list, the initial demonstration consists of the preparation and analysis of a QC check sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study.
- Four aliquots of the QC check sample (independent source from the calibration) are analyzed with the same procedures used to analyze samples, including sample preparation.
- The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples.
- For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is successful analysis of an extracted standard at the reporting limit and a single point calibration.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

Standards and reagents, in this SOP, are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention," of the Corporate Environmental Health and Safety Manual, and DV-HS-001, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Distilled client sample – Aqueous Alkaline - Waste Stream E
- Contents of autosampler cups - Aqueous Alkaline - Waste Stream E
- Chloroform extracted sample waste - Aqueous Alkaline – Waste Stream E
- Chloroform extract waste – Waste Stream C

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

- Method 9066, Phenolics (Colorimetric, Automated 4-AAP With Distillation), Revision 0, September 1986.

15.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition; Clesceri, L.S.; Greenberg, A.E.; Eaton, A.D.; Editors; American Public Health Association, American Water Works Association, and Water Environment Federation, 1998.

15.3 Method 5530, Phenols

15.4 EPA Method 420.2, Phenolics (Colorimeteric, Automated 4-AAP With Distillation), Approved for NPDES, 1974.

15.5 EPA Method 420.4, Determination of Total Recoverable Phenolics by Semi-Automated Colorimetry, Approved for NPDES, 2007

16.0 Method Modifications:

Item	Method	Modification
1	SW 9066	There is a discrepancy between the preservation methods and holding times given in the SW-846 methods and those given in Chapter Two of SW-846. The laboratory has chosen to use sulfuric acid to adjust the sample pH to 2.
2	SW 9066, EPA 420.2, and EPA 420.4	The size of the distillation apparatus and volumes of sample and reagent were reduced to conserve space and speed up the analysis. Reduced volume versions of this method that use the same reagents

Item	Method	Modification
		and molar ratios are acceptable provided they meet the quality control and performance requirements stated in the method. It is not possible to use this method to differentiate between different types of phenols.
3	SW 9066, EPA 420.2, and EPA 420.4	Provisions have been made for dilution of the chloroform extracts up to 5x. This is sometimes necessary for samples that are over-range and cannot be reprepared due to limited sample volume or other reasons.
4	SW 9066, EPA 420.2, and EPA 420.4	Standard Methods Method 5530B distills samples by adjusting the pH to "approximately" 4.0 with H_3PO_4, while SW-846 methods distill by adjusting to a pH "<" 4 with H_2SO_4 (Method 9066) and to a pH of "approximately" 4 with H_2SO4 (Method 9065). This SOP distills the samples by adjusting the pH to less than 4 with H_2SO_4.
5	SW 9066, EPA 420.2, and EPA 420.4	The source methods state to place phenol standards in sampler "in order of decreasing concentration." The standards are placed in the sampler in increasing concentration per the instrument manufacturer's specifications.

17.0 Attachments

Attachment 1: Example Wet Chemistry Prep Bench Sheet
Attachment 2: Example Data Review Checklist

18.0 Revision History

- Revision 0, dated 28 March 2010
  - Annual Review
  - Added section 6.3

- Revision 0, dated 15 March 2009

Attachment 1.

Example Wet Chemistry Prep Bench Sheet

TestAmerica Denver

PHENOL SAMPLE PREPARATION

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

	ICV Information	ICAL/CCV/LCS/MS
Analyst: ________	Source: ________	Source: ________
	Verification/Lot #: ________	Verification/Lot #: ________
Date: ________	Prep Date: ________	Prep Date: ________
	Made By: ________	Made By: ________
Prep Time: ________	Concentration: ________	Concentration: ________
	Expiration Date: ________	Expiration Date: ________
Batch #: ________		

SOP Information
SOP #: DEN-WC-0013
Rev #: ________

Lot Number	Work Order #	pH	Initial Vol (mL)*	Default Final Vol (mL)*	Prep Dil.	Comments
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
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			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		
			50 / 200	50 / 200		

** When performing the manual method circle the 200mL aliquot. When performing the automated method circle the 50mL aliquot. If a different final volume is used, cross out "50 or 200", write in correct volume, and explain in* Comments *column.*

Attachment 2.

Example Data Review Checklist

TESTAMERICA Denver

Wet Chemistry Data Review Checklist
For Tests with Calibration Curves

Test Name/ Method #: _____ **SOP #** _____

Instrument: _____ **Analyst:** _____ **Analysis Date:** _____

Client	Lot / Sample Numbers	Matrix	Batch Number	Special Instructions
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD
				No B J G s DCS MSQC RD

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Minimum of five standards in ICAL or as specified in method?				
2. Correlation coefficient ≥ 0.995 ?				
3. Second-source ICV analyzed, and recovery 90-110%?				
4. ICB analyzed immediately after the ICV & results < the RL?				
5. CCV analyzed after every ten samples & recovery +/- 10% of true value?				
6. CCB analyzed after every CCV & results < RL?				
B. Sample Results				
1. All samples greater than highest calibration standard diluted and reanalyzed?				
2. Do associated RLs/MDLs reflect dilutions or limited sample volume?				
3. All reported results bracketed by in control CCV results?				
4. Sample analyses done within holding time?				
5. Initial pH check documented for all samples?				
6. Preparation benchsheet completed and included in package?				
7. Special client requirements met?				
8. Were data manually transcribed from instrument printouts into QuanTIMS verified 100% including significant figures?				
9. Do the prep and analysis dates in QuanTIMS reflect the actual dates?				
10. Are all data being reported highlighted on the benchsheet?				
11. Raw data copies prepared and scanned?				
12. Manual integrations done properly?				
C. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 20x blank have NCM?				
2. LCS run for batch and within QC limits ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within 10%?				

Analyst: _____ **Date:** _____

2nd Level Reviewer : _____ **Date:** _____

2/11/09 Version
QA\Edit\Forms\Wet Chemistry\Calib Curve Checklist

MEASUREMENT OF ANALYTES USING THE KONELAB ANALYZER

(Methods: Various)

Approvals (Signature/Date):

Andrea Teal February 03, 2014
Andrea Teal Date
Quality Assurance Manager

February 05, 2014
Jon Ross Date
Technical Manager – Inorganics

February 05, 2014
Whitney Palefsky Date
Environmental Health and Safety Coordinator

1.0 Scope and Application

This SOP gives the procedures for the determination of various analytes in water and soil samples using the Konelab Autoanalyzer. The following analytes may be determined:

Analyte	Reference Method	SOP Summary
Ammonia	EPA 350.1 SM4500-NH3 B (AQ prep) COE 3-154 (SO Prep) SM4500-NH3 G (analysis)	Attachment 1a
Chloride	EPA 325.2 EPA 9251 SM4500-Cl^- E	Attachment 1b
Total Chlorine	EPA 5050 / EPA 9251	Attachment 1b
Ferrous iron	SM3500-Fe D (SM 21st Edition) SM3500-Fe B (SM On-Line Edition)	Attachment 1c
Ferric Iron	SM3500-Fe B (Calculation)	Attachment 1c
Hexavalent Chromium	EPA 7196A SM3500-Cr D (SM 21st Edition) SM3500-Cr B (SM On-Line Edition)	Attachment 1d
Trivalent Chromium	EPA 7196A SM3500-Cr B (Calculation)	Attachment 1d
Ortho-Phosphate	EPA 365.1 SM4500-P F	Attachment 1e
Sulfate	EPA 375.4 EPA 9038	Attachment 1f
Total Sulfur	EPA 5050 / EPA 9038	Attachment 1f
BTU	ASTM D240-87	Attachment 1f

The reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with each method are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Samples and reagents are dispensed into disposable, acrylic multi-cell cuvettes that hold 12 separate analyses. During the incubation period(s), the sample cuvettes are maintained at 37°C. The measurement system is a single channel interference filter photometer with beam splitting reference. The color wheel can be configured with up to 15 different filters. Fiber optic cabling transfers the light signal from the source to the detector. The lamp is a halogen lamp.

The SOP Attachments contain the method parameters required to perform each analysis, a summary of the "chemistries" (reagents, standards, procedures, wavelengths, etc.) used to measure each target analyte, sample preparation information, as well as any method-specific information, requirements, and/or criteria. Method-specific requirements contained in the Attachments supersede the requirements outlined within the body of this SOP.

This SOP is based on the methods listed in Section 1.0.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Acetone, hydrochloric acid, methanol, nitric acid, and sulfuric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.1.5 Refer to the appropriate SOP Attachment, as listed in Section 1, for any method-specific procedural interference information.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. For this procedure, the only cleanup procedure available is filtration through a 0.45um filter. This filtration step will remove turbidity that is present that may interfere with analysis.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Refer to the appropriate SOP Attachment, as listed in Section 1, for any method-specific matrix interference information.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile gloves, or equivalent, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Mercuric thiocyanate will give off hydrogen cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

Sulfuric acid is a strong oxidizer and is a corrosive. It will react violently when combined with organic compounds, possibly producing fire. Inhalation can cause irritation of the nose, throat, mucus membranes, and upper respiratory tract. Contact with the eyes can cause blurred vision, redness, pain, and even blindness.

Acetic acid is a corrosive. Contact with concentrated acetic acid can cause damage to the skin and eyes. Inhalation of concentrated vapors may cause damage to the lining of the nose, throat, and lungs.

Acetone is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Methanol is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the “MSDS” link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Acetic Acid	Corrosive Poison Flammable	10ppm TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Acetone	Flammable	1000ppm TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Barium Chloride	Poison Irritant	0.5mg/m^3 TWA	May be fatal if swallowed. Harmful if inhaled. Avoid contact with eyes, skin, and clothing. Avoid breathing dust. Keep container closed and when in use adequate ventilation.
Ferric Nitrate	Oxidizer	None	Causes irritation to the respiratory tract. Causes irritation, redness, and pain to the skin and eyes.
Hydrochloric Acid[2]	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Mercuric Thiocyanate	Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely Toxic. Causes irritation to the respiratory tract. **May produce Hydrogen Cyanide gas if combined with strong acids.** Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. **May affect the central nervous system.** Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Methanol	Flammable Poison Irritant	200ppm TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Nitric Acid[2]	Corrosive Oxidizer Poison	2ppm TWA 4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Phenol	Corrosive	5ppm TWA	Breathing vapor, dust or mist results in digestive disturbances. Will irritate and possibly burn respiratory tract. Rapidly absorbed through the skin with systemic poisoning effects to follow. Discoloration and severe burns may occur, but may be disguised by a loss in pain sensation. Eye burns with redness, pain, blurred vision may occur. May cause severe damage and blindness.
Phosphoric Acid	Corrosive	1mg/m^3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Hydroxide	Corrosive Poison	2mg/m^3 TWA	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid[2]	Corrosive Oxidizer Dehydrator Poison Carcinogen	1mg/m^3 TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
[2]Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

All applicable laboratory support equipment and supplies (including balances; thermometers; and volumetric containers, used to determine quantitative measurements, such as mechanical pipettes, disposable graduated pipettes, gas-tight syringes, etc.) must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

6.1 Equipment and Instrumentation

- Top-loading Balance
- Tumbler
- MicroDist Apparatus
- Konelab Model 20 Autoanalyzer, or equivalent
- Data System: The software system is based on the Windows NT operating system. The hardware interface can be either RS232 or an Ethernet connection and can be integrated to both transmit and receive information from a LIMS.

6.2 Lab Supplies

- Volumetric Containers and Dispensing Devices – various sizes; Class A, where applicable. Includes mechanical pipettes, disposable transfer pipettes, and gas-tight syringes.
- pH paper (narrow range)
- Residual Chlorine Check Strips – starch iodide strips
- Detergent – Nochromix, or equivalent, used for washing non-disposable labware.
- Hengar Granules
- Magnetic Stir Bars
- Teflon Chips – used as soil blank matrix
- 125mL plastic screw top bottles

6.3 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination. The routine sample collection containers supplied by the laboratory are purchased with Certificate of Analysis attesting to purity.

Refer to Attachment 2 for a cross-reference of containers to methods.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it; that is, the "children may not outlive the parents".

Unless noted elsewhere in this SOP, the following expiration date requirements and storage conditions apply:

Purchased Unopened = Manufacturer's Expiration Date

Purchased Opened = 5 years from Open Date
Prepared = 3 months from Preparation Date
Storage = Room Temperature

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased reagents, and scanned and filed in the Data Archival Folder on the G-drive or attached to the standard in TALS. Unless otherwise stated in the method-specific attachments, reagents should be stored in a cool, dry, ventilated storage area away from incompatible materials.

Refer to the appropriate SOP Attachment, as listed in Section 1, for the method-specific reagents associated with each analysis.

Acetone, hydrochloric acid, methanol, nitric acid, and sulfuric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Blank Matrix – Ottawa Sand or Teflon Chips (see associated Attachment). Used for the preparation of soil QC samples.

Laboratory Reagent Water – ASTM Type II.

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the G-drive or attached to the standard in TALS.

Refer to the appropriate SOP Attachment, as listed in Section 1, for the method-specific standards associated with each analysis. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

8.0 Sample Collection, Preservation, Shipment, and Storage

Refer to Attachment 2 for the method-specific sample collection, preservation, shipment, and storage information.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data. Refer to the appropriate SOP Attachment, as listed in Section 1, for any method-specific quality control information associated with each analysis.

9.1 Batch QC

A preparation/analysis batch consists of up to 20 environmental samples and the associated QC items analyzed together within a 24 hour period. Refer to the Table in Attachment 3 for a summary of the method-specific batch QC items.

If there is insufficient sample volume to perform the required matrix spike(s), an NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 20mL.

If insufficient sample volume is provided to perform the SD or MS/MSD, the LCS must be prepared in duplicate (LCS/LCSD). An NCM must be initiated on all samples within the batch to denote this situation.

MRL LCS for DW
The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. Therefore, if analyzing drinking water samples, an LCS at the RL must also be included in the required batch QC.

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)
The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 3 standards and a blank. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

The regression coefficient (r^2) of the regression curve must be greater than or equal to 0.995 for the initial calibration curve to be acceptable.

9.2.2 Second Source Initial Calibration Verification (ICV)
The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The ICV must be within 10% to be acceptable.

9.2.3 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)
The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed at the beginning of each analysis run. Continuing calibration blanks are analyzed following each CCV.

Initial and continuing calibration blanks must be <MDL to be acceptable.

9.2.4 Continuing Calibration Verification

Unless noted elsewhere in this SOP, the initial calibration curve must be verified every 10 analyses and at the end of the analysis run with a mid-level standard.

The CCV must be within 10% to be acceptable.

9.2.5 Linear Calibration Range / Linear Dynamic Range

The linear calibration range (LCR), also known as the Linear Dynamic Range (LDR), must be determined initially for the following methods:

SM3500-Fe B
SM3500-Cr B
SM4500-NH_3 G
SM4500-P F
SM4500-Cl^- E
EPA 350.1
EPA 353.2
EPA 365.1

The initial demonstration of linearity uses as many standards as necessary to ensure the curve is linear. The standards should cover the low and high ends of the calibration range. Each standard must be within +/- 10% of the true value.

The LCR/LDR must be verified every 6 months for the following methods:

EPA 350.1
EPA 353.2
EPA 365.1

The continued demonstration of linearity consists of a minimum of 3 standards and a blank. For the linearity to be verified, the standards must be within +/-10% of the initial value to be acceptable. If this criterion is not met, the initial demonstration of linearity must be performed.

Note: This study is not meant to extend the linear range of the calibration. If an analyte is measured at a concentration greater than the highest calibration standard, the sample must be analyzed at a dilution to bring the target analyte into the range of the initial calibration.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedure* and the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

Remove the samples or extracts from the refrigerator and allow them to come to room temperature.

Soil samples must be homogenized prior to preparation in accordance with SOP SA-QA-015: *Compositing, Homogenization, and Segregation of Samples*. Note: If a sample or extract is filtered, the associated QC must also be filtered.

The sample preparation procedures are summarized below. Refer to appropriate SOP Attachment for the method-specific procedures.

Analyte	Aqueous Prep	Soil Prep
Ammonia	Distillation* (EPA 350.1 / SM4500-NH_3 B) Undistilled (No Prep)*	Soil Leachate
Chloride	No Prep	Soil Leachate
Total Chlorine	EPA 5050 (SOP SA-GE-010)	EPA 5050 (SOP SA-GE-010)
Ferrous Iron (Ferric Iron calc)	Filter	
Hexavalent Chromium (Trivalent Chromium calc)	Filter	EPA 7196A (SOP SA-ME-021)
Ortho-Phosphate	No Prep	Soil Leachate
Sulfate	No Prep	Soil Leachate
Total Sulfur	EPA 5050 (SOP SA-GE-010)	EPA 5050 (SOP SA-GE-010)
BTU	ASTM D270-87	ASTM D240-87

*Ammonia samples are distilled upon client request.

10.2 QC Sample Preparation

Refer to appropriate SOP Attachment, as listed in Section 1, for the method-specific QC sample preparation instruction associated with each procedure.

10.3 Analysis

10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

Data System
Programmable instrument parameters include:

Volume of sample (maximum of 125uL)
Wavelength
Type and order of reagent(s) addition
Background absorbance
Time of sample/reagent mixing
Timing of absorbance readings
Dilutions
Reagents lot numbers/expiration dates

QC checks can be programmed into the methods as needed, including initial and continuing control standards, blanks, and upper and lower limits on absorbance. If samples or standards exceed these control limits, the data are flagged.

Operation
The instrument utilizes the following process:

- Sample is dispensed into the cuvettes according to the test method (maximum sample volume is 125uL)
- The reagents are added to the sample and mixed.
- The sample is incubated.
- The sample absorbance is measured (if absorbance exceeds calibration range, sample is diluted as defined in the test method and reanalyzed. If the dilution exceeds the maximum range, the data are flagged and the analyst must prepare dilution for sample).

10.3.2 Initial and Continuing Calibration

The instrument is calibrated using the standards and criteria described in the appropriate SOP Attachment, as listed in Section 1. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.2, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.4 and the appropriate SOP Attachment, as listed in Section 1.

10.3.3 Sample Analysis

Remove the samples or extracts from the refrigerator and allow them to come to room temperature.

Samples are loaded into racks that hold 14 samples each. The autosampler holds up to 6 racks of 14 samples. The racks can be fitted to hold sample containers from 0.5mL up to 10mL. Two separate wash stations are used to minimize sampler carryover. The probe is flow-through and externally rinsed.

The sample/extract must be analyzed using the same volume used for the calibration standards. The default procedure is to include QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples in the analysis.

10.3.4 Example Analytical Sequence

Refer to the appropriate SOP Attachment, as listed in Section 1, for any changes to the sequence listed below.

Description	Comments
Blank	
Initial Calibration	
ICV	Second Source
ICB	
Samples & Batch QC Items	Up to 10 analyses. Not to exceed 2 hours.
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses. Not to exceed 2 hours.
CCV	
CCB	

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-002: *Data Review and Reporting*.

11.1.1 MS/MSD Evaluation

If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data is flagged.

11.1.2 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify LIMS Worksheet Notes and/or use the LIMS Historical Data Tracker Feature to determine if historical data is available for review.

11.1.3 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- Total Results ≥ Dissolved Results
- TKN ≥ Ammonia
- Total Phosphorus ≥ Ortho-phosphate
- TDS ≥ Individual Anions

- Ferrous Iron ≤ Total Iron

11.1.4 Drinking Water Compliance Evaluation

Public water suppliers (PWS) are governed by EPA-specified Maximum Contaminant Levels (MCL) above which indicates noncompliance. The MCLs associated with this procedure are given in Attachment 5. Notify the PM immediately via a Nonconformance Memo if any sample contains a detection above these levels.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
dw = % Solids decimal equivalent
D = Dilution factor

Note: All dry weight corrections are performed automatically in LIMS.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated

(statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS, MS/MSD, SD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four

consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.4 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

- Non-hazardous acidic and alkaline wastewater and samples must be neutralized before disposal into the sewer system.

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-ME-021: *Digestion Procedures for Solids for Hexavalent Chromium*
- SOP SA-QA-002: *Data Review and Reporting*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Standard Methods for the Examination of Water and Wastewater*; American Public Health Association: Washington, DC
  - SM1020: *Quality Assurance*; 20th Edition
  - SM3020: *Quality Assurance/Quality Control*; 2005
  - SM3500-Cr B: *Chromium, Colorimetric Method*; 2001
  - SM3500-Fe B: *Ferrous Iron, Phenanthroline Method*; 1997
  - SM4020: *Quality Assurance/Quality Control*
  - SM4500-Cl^- E: *Chloride, Automated Ferricyanide Method*; (Approved 1997; Editorial Revision 2011)
  - SM4500-NH_3 B: *Nitrogen (Ammonia), Preliminary Distillation Step*; 1997
  - SM4500-P F: *Phosphorous, Automated Ascorbic Acid Reduction Method*; 1999
  - SM4500-NH_3 G: *Nitrogen (Ammonia), Automated Phenate Method*; 1997
  - SM5020: *Quality Assurance/Quality Control*
- *Methods for Chemical Analysis of Water and Wastes*; U.S. EPA Office of Research and Development: Cincinnati, OH, March, 1983
  - EPA 325.2: *Chloride (Colorimetric, Automated Ferricyanide AAII)*; 1978
  - EPA 350.1: *Determination of Ammonia Nitrogen by Semi-Automated Colorimetry*; Revision 2.0, August 1993
  - EPA 365.1: *Determination of Phosphorus by Semi-Automated Colorimetry*; Revision 2.0, August 1993
  - EPA 375.4: *Sulfate (Turbidimetric)*; 1978
- *Test Methods for Evaluating Solid Waste, Third Edition On-line*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC
  - EPA 5050: *Bomb Preparation Method for Solid Waste; Revision 0, September 1994*
  - EPA 7196A: *Chromium, Hexavalent (Colorimetric)*; Revision 1, July 1992
  - EPA 9038: *Sulfate (Turbidimetric)*; Revision 0, September 1986
  - EPA 9251: *Chloride (Colorimetric, Automated Ferricyanide AAII)* ; Revision 0, September 1986
- *Annual Book of ASTM Standards Section 5, Volume 05.01;* American Society of Testing and Materials: Philadelphia, 1992.

- D240-87: *Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter*; May 1991

16.0 Method Modifications and Clarifications

16.1 Many of the reference methods were written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using these procedures. These procedures may be modified to analyze other matrices (e.g., wipe samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project. Wipe, waste, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs and MDLs (converted for initial and final volumes, etc.), and soil QC limits to evaluate wipe, waste, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. Ottawa sand is used as the blank matrix for tissue samples unless a "true" tissue matrix is required by the project.

16.2 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each batch of drinking water samples.

16.3 Refer to appropriate SOP Attachment, as listed in Section 1, for any method-specific modifications and/or clarifications associated with each procedure.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1a: SOP Summary – Ammonia
Attachment 1b: SOP Summary – Chloride and Total Chlorine
Attachment 1c: SOP Summary – Ferrous Iron and Ferric Iron
Attachment 1d: SOP Summary – Hexavalent Chromium and Trivalent Chromium
Attachment 1e: SOP Summary – Ortho-phosphate
Attachment 1f: SOP Summary – Sulfate, Total Sulfur, and BTU
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3a: Batch QC Frequency
Attachment 3b: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Maximum Contaminant Levels (MCL) Table

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Fraction	Matrix	Routine Sample Container	Routine Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Ammonia	Water	250mL plastic	100mL	1mL 1:1 H_2SO_4 pH <2	4℃ [1]	Sodium Thiosulfate	28 Days
Ammonia	Soil	8oz Plastic	20g	None	4℃ [1]	None	28 Days[2]
BTU	Water	4oz glass soil jar	1g	None	4℃ [1]	None	28 Days
BTU	Soil	4oz glass soil jar	1g	None	4℃ [1]	None	28 Days[2]
Chloride	Water	125mL plastic	10mL	None	4℃ [1]	None	28 Days
Chloride	Soil	8oz Plastic	20g	None	4℃ [1]	None	28 Days[2]
Chloride, Total	Water	125mL plastic	1g	None	4℃ [1]	None	28 Days
Chloride, Total	Soil	8oz Plastic	1g	None	4℃ [1]	None	28 Days[2]
Ferrous Iron	Water	125mL plastic	10mL	None	4℃ [1]	None	15 Minutes[3]
Ferric Iron (Calc)	Water	125mL plastic	10mL	None	4℃ [1]	None	15 Minutes[3]
Hexavalent Chromium	Water	125mL plastic	10mL	None	4℃ [1]	None	24 Hours
Hexavalent Chromium	Soil	8oz Plastic	1g	None	4℃ [1]	None	Digestion: 30 days from collection Analysis: 168 hours from digestion

Fraction	Matrix	Routine Sample Container	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Trivalent Chromium (Calc)	Water	125mL plastic	10mL	None	4℃ [1]	None	24 Hours
Trivalent Chromium (Calc)	Soil	8oz Plastic		None	4℃ [1]	None	Digestion: 30 days from collection Analysis: 168 hours from digestion
Ortho-Phosphate	Water	125mL plastic	10mL	None	4℃ [1]	None	48 Hours
Ortho-Phosphate	Soil	8oz Plastic	20g	None	4℃ [1]	None	48 Hours[2]
Sulfate	Water	125mL plastic	10mL	None	4℃ [1]	None	28 Days
Sulfate	Soil	8oz Plastic	20g	None	4℃ [1]	None	28 Days[2]
Sulfur, Total	Water	125mL plastic	1g	None	4℃ [1]	None	28 Days
Sulfur, Total	Soil	8oz Plastic	1g	None	4℃ [1]	None	28 Days[2]

[1]Samples must be maintained at 0-6℃, with no frozen samples.
[2] Inclusive of digestion and analysis.
[3] Due to the nature of this analysis, this test is considered a field test. No definitive holding time is specified in the reference methods; however, the Methods Update Rule of 40CFR Part 136 identifies a 15 minute holding time for this method.

Note: NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives and/or de-chlorination agents.

Residual Chlorine Check (for Ammonia analyses only)

For each sample,

- Place a piece of starch-iodide paper in a disposable medicine cup.
- Pour a few drops of sample into the medicine cup and note the color change of the paper.
- If the paper turns blue or black, residual chlorine is present. Initiate a Nonconformance Memo. Dechlorinate the sample with sodium thiosulfate.

pH Verification (for Ammonia analyses only)

For each sample,

- Place a piece of pH paper in a disposable medicine cup.
- Pour a few drops of sample into the medicine cup and note the color change of the pH paper.
- If the pH is outside the range of less than 2, initiate a Nonconformance Memo. Adjust the sample pH to less than 2 using sulfuric acid. The volume of sulfuric acid must not exceed 1% of the volume of the container or sample; e.g., no more than 2.5mL of sulfuric acid per 250-mL container or sample.

Note: To avoid cross-contamination, use a separate medicine cup, pH strip, and residual chlorine strip per sample. Do not dip the strip into the sample container.

Attachment 1a
SOP Summary
Ammonia

1.0 Summary

Ammonia reacts with hypochlorite and alkaline phenol to form indophenol blue. The blue color produced is intensified with sodium nitroprusside and measured at 660nm.

2.0 Interferences

Extreme sample pH values may interfere. Proper sample preservation and adjustment just prior to analysis should alleviate this problem. Turbidity and color in samples must be filtered and/or diluted prior to analysis. Residual chlorine should be removed at the time of sampling by the addition of sodium thiosulfate.

3.0 Reagents and Standards

3.1 Purchased Reagents

Sodium Thiosulfate ($Na_2S_2O_3 \bullet 5H_2O$) Dechlorination Reagent – reagent grade

Sodium Thiosulfate, 0.025N – Purchased pre-prepared
Expiration: 1 year from opening

Sulfuric Acid (H_2SO_4) – concentrated, reagent grade
Storage: Area with acid resistant floors. Store away from direct sunlight, heat, and water.

Sodium Hydroxide (NaOH) – pellets, reagent grade
Storage: Isolate from moisture and heat sources.

Potassium Chloride (KCl) – reagent grade

Phenol – solution must be ≥89% pure
Storage: Isolate from heat or ignition sources.

Sodium Tetraborate ($Na_2B_4O_7$) – reagent grade

Bleach/Sodium Hypochlorite – must contain 6.15% sodium hypochlorite (NaOCl)
Storage: Protect from heat and light.
Expiration: Unopened: 1 year; Opened: 6 months

Disodium Ethylenediamine Tetraacetate (EDTA) – reagent grade

Sodium Nitroprusside ($Na_2(NO)Fe(CN)_5 \bullet 2H_2O$) – reagent grade
Storage: Away from moisture and incompatibles

3.2 Prepared Reagents

Sodium Phenate Solution
Dissolve 9.3mL liquid (≥89%) phenol in 50mL ammonia free water, slowly add 3.2g NaOH and

mix thoroughly and dilute to 100mL with ammonia free water. **CAUTION**: Wear gloves and eye protection when preparing this solution. Expiration: Daily

Sodium Hypochlorite solution
Dilute 25mL of 6.15% NaOCl with 25mL ammonia free water. Expiration: Daily

EDTA buffer
Dissolve 50g of disodium ethylenediamine tetraacetate and 5.5g of NaOH in 1000mL of ammonia free water. Expiration: 1 year

Sodium Nitroprusside solution
Dissolve 0.5g of $Na_2(NO)FE(CN)_5.2H_2O$ in 1000mL of ammonia free water. Expiration: 1 year

Sulfuric Acid Solution (0.01M) – NH3_Diluent
Add 2.10mL of concentrated sulfuric acid to 1.0gallon of water (0.56ml of concentration H2SO4 per liter of water). The pH of this solution is 2.0. Verify the pH before use. Expiration: 1 year

Sodium Hydroxide Solution (6M)
Add 500mL DI water to a 2-L beaker. Add a stir bar and place on stir plate. In small increments and with constant stirring, slowly add 240g NaOH to the beaker. Allow to cool and dilute to 1.0L with DI water. Transfer to a labeled storage container. Expiration: 1 year

Potassium Chloride (2M)
Dilute 296g of potassium chloride to with1800ml ammonia free water. Add 1.12ml concentrated sulfuric acid. Dilute to 2000ml and mix thoroughly. Expiration: 1 year

MicroDist Trapping Solution
Add 3.3mL of concentrated sulfuric acid to 1.0L of water. Expiration: 1 year

Borate Buffer (MicroDist)
Dilute 5.0g of sodium borate decahydrate and 22g sodium hydroxide to 1.0K with DI water.
Expiration: 1 year

3.3 Standards

Ammonia Stock Standard (1000mg/L)
Dissolve 3.819g of dried Ammonium chloride [NH_4Cl] in 2000mL of ammonia free water.
Expiration: 1 year

Ammonia Intermediate Standard (100mg/L)
Dilute 10.0mL of the stock standard to a final volume of 100mL with DI water.
Expiration: 1 month

Ammonia Calibration Standards
Prepare calibration standards in 0.01M sulfuric acid (NH3 Diluent) according to the following table.

Standard Level	Parent Standard	Volume Intermediate (mL)	Final Volume (mL)	Final Concentration (mg/L)
1	Ammonia Intermediate Standard	2.0	100	2.0
2 (LCS)	Ammonia Intermediate Standard	1.0	1000	1.0
3	Ammonia Intermediate Standard	0.50	100	0.50
4	Ammonia Intermediate Standard	0.25	100	0.25
5	Ammonia Intermediate Standard	0.10	100	0.10
6	Cal 2	5.0	100	0.050
7	Cal 2	2.5	100	0.025

Expiration: 3 months from preparation

Initial Calibration Verification (ICV) – 1.0mg/L standard from NCL Labs. Adjust the to pH 2 with a weak solution of H2SO4. Expiration: manufacturer's date (1 month if no date given)

4.0 Sample Preparation

4.1 Aqueous Samples

Aqueous samples are routinely analyzed with no sample preparation. The samples can be distilled upon request. Note: The ammonia standards are not distilled.

Prepare QC samples in the same manner as field samples as follows:

- Laboratory Control Sample – Prepared in same manner as Cal Level 2.
- Low-Level Laboratory Control Sample (Drinking Water only) – Lowest level standard of the initial calibration
- MS/MSD – Add 0.1mL of the 100mg/L Ammonia Intermediate Standard to 10mL of the sample selected for MS/MSD. Note: SM4500-NH3 G recommends the MS/MSD spike concentration to be varied periodically.

4.1.1 Ammonia Micro-Distillation Preparation

Set controller to 120^{o}C and turn on the heater block to warm up (~40 min).

Determine the number of collector tubes needed and align with "M" end in the up position by placing them into a collector tube rack.

Label and fill each collector tube with 1 mL of trapping solution (0.016M sulfuric acid). At the end of the distillation, once diluted to 6 mL final volume, the final concentration will be 0.003M sulfuric acid.

Place the same number of sample tubes into the sample tube rack.

Label each tube and place 6 mL of sample into each sample tube.

Add 1mL of 0.55M borate buffer to each tube.

Immediately push the “D” end of the collector tube over the open end of the sample tube to start the seal.

Place the tube assembly in the press by putting the sample tube through the hole in the base.

Before pressing, place one hand at the breakaway point located on the collector tube to stabilize the assembly.

Apply pressure to the assembly with a smooth motion until the stop ring on the sample tube hits the D end of the collector tube.

Once complete, put on heat resistant gloves and add the tube assemblies to the pre-heated block by placing the sample tube and D end of the collector tube into the block.

Set the timer for 30 minutes.

Once time is up remove the assemblies from the block one at a time and immediately remove the sample tube from the collector tube and invert so the D side is facing up. Note: this should be done within 4 seconds to prevent “suck-back” of the sample.

Once all of the tubes have been removed and disassembled, Parafilm the M end of the tube and allow the tubes to cool for 10 minutes.

Hold the tube horizontally and roll the tube with your fingers to gather the droplets that are stuck to the walls. The tube can be flicked with your finger as well.

With the D end still up, grasp the tube and break it at the breakaway point by pulling the D end towards your body and twisting the tube. Discard the D end of the tube.

Dilute the distillate up to the 6 mL mark with distilled water and cap the tube until ready for analysis.

4.2 Soil Samples

- Weigh approximately 20g of sample into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of 2M potassium chloride to each sample container and place in the tumbler.
- Rotate for 30-45 minutes. Remove the containers from the extractor and allow the leachates to settle.
- Filter the extract using a syringe filter with a 0.45um pore size filter and analyze the extracts as liquid samples.

Prepare QC samples in the same manner as field samples as follows:

- Method Blank – Weigh approximately 20g of Teflon chips into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g.
- Laboratory Control Sample – Weigh approximately 20g of Teflon chips into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 1mL of 100mg/L Ammonia

Intermediate Standard to the extraction bottle.

- MS/MSD – Weigh additional 20g aliquots of one of the samples for the MS/MSD. Add 1mL of 100mg/L Ammonia Intermediate Standard Ammonia Matrix Spike Solution to the sample.

5.0 Method-Specific QC Requirements

5.1 Linear Calibration Range
EPA 350.1 – Performed initially; verified every 6 months.
SM4500-NH_3 G – Performed initially.

5.2 Method Detection Limit Studies
EPA 350.1 – Requires MDL Studies every 6 months.
SM4500-NH_3 G – Requires MDL Study to be analyzed over 3-5 days.

6.0 Method-Specific Modifications and/or Clarifications

The ammonia distillation sample volumes have been adjusted to compensate for current lab restrictions of glassware and volume of sample submitted. All reagent volumes have been adjusted accordingly.

The reference methods state to adjust sample pH with 1N NaOH. The sample is preserved with H_2SO_4 to a pH of <2. To adjust the pH with such a weak solution would require adding >5% volume and therefore diluting the sample. As such, the laboratory uses 6N NaOH to adjust the sample pH.

Attachment 1b
SOP Summary
Chloride & Total Chlorine

1.0 Summary

Chloride reacts with mercuric thiocyanate forming a mercuric chloride complex. Released thiocyanate reacts with iron (III) forming a red ferric thiocyanate complex the color produced, measured at 480nm, is proportional to the chloride concentration.

Refer to SOP SA-GE-010 for information on the preparation procedures for Total Chlorine. The attached Work Instruction provides the procedural and calculation information.

2.0 Interferences

Highly colored samples can positively interfere. Turbid samples should be filtered.

3.0 Reagents and Standards

3.1 Purchased Reagents

Mercuric Thiocyanate [$Hg(SCN)_2$] – reagent grade
Storage: Protected from light.

Methanol – HPLC or P&T grade
Storage: Protect from heat, sparks, and flame in a flammable storage area.

Mercuric Thiocyanate Solution (Saturated) – Dissolve 4.17g $Hg(SCN)_2$ into 500mL of methanol. Dilute to 1L with methanol, mix, and filter. Dispose if reagent becomes discolored or cloudy.
Storage: Protected from light.
Expiration: 1 year

Ferric Nitrate [$Fe(NO_3)_3.9H_2O$] – reagent grade
Storage: Away from sources of heat and moisture. Avoid storage on wooden floors.
Expiration: 1 year

Nitric Acid, concentrated – A.C.S. grade
Storage: acid resistant floors and good drainage; away from sunlight, heat, and water.

3.2 Prepared Reagents

Ferric Nitrate Solution (20.2%) – Dissolve 202g $Fe(NO_3)_3.9H_2O$ into 500mL reagent water. Carefully add 44.4mL of concentrated nitric acid. Mix and dilute to 1L with reagent water. Store in an amber bottle away from light.
Expiration: 1 Year

Color Reagent – Add 75mL of Mercuric Thiocyanate Solution to 75mL Ferric Nitrate Solution. Mix and dilute to 500mL with reagent water. Dispose if reagent becomes discolored or cloudy.
Storage: Tightly closed-light resistant container. Protect from light.
Expiration: 3 months

3.3 Standards

Sodium Chloride (NaCl) – reagent grade

Chloride Calibration Stock (1000mg/L) – Dissolve 1.6484g dried NaCl into 1000mL reagent water.

Chloride Calibration Standards

Standard Level	LIMS Reagent Name	Parent Standard	Aliquot (mL)	Final Volume (mL)	Final Concentration (mg/L)
1	Cl CAL10	Cl Cal Stk	0.1	100	1
2	Cl CAL9	Cl Cal Stk	0.3	100	3
3	CL LOW CCV	Cl Cal Stk	0.5	100	5
4	Cl CAL7	Cl Cal Stk	0.7	100	7
5	Cl CAL6	Cl Cal Stk	1.0	100	10
6	Cl CAL5	Cl Cal Stk	2.0	100	20
7	Cl CAL4	Cl Cal Stk	4.0	100	40
8	Cl CAL3	Cl Cal Stk	6.0	100	60
9	Cl CAL2	Cl Cal Stk	8.0	100	80
10	Cl CAL1	Cl Cal Stk	10	100	100

Dilute to volume in reagent water.

Chloride Laboratory Control Sample (50mg/L) – Add 5mL Chloride Calibration Stock to a 100mL volumetric. Dilute to volume with reagent water.

Chloride Low Level CCV (5.0mg/L) – Add 0.5mL Chloride Calibration Stock to a 100mL volumetric. Dilute to volume with reagent water.

QCI-710 – ICV purchased from QCI. The concentration is updated to match the COA each time it is ordered
Storage: This stock solution is stored at <6^{o}C but not frozen.
(Opened: may be kept until the manufacturer's expiration date or one year, whichever is shorter)

Chloride Low Level Initial Calibration Verification (5.0mg/L) – Add 20mL of QCI-710 to a 100mL volumetric. Dilute to volume with reagent water. Note: Volumes may need adjusted to obtain 5.0mg/L concentration.

4.0 Sample Preparation

4.1 Aqueous Samples

Aqueous samples are routinely analyzed with no sample preparation.

Prepare QC samples in the same manner as field samples as follows:

- Laboratory Control Sample – prepared as described above
- Low-Level Laboratory Control Sample (Drinking Water only) – Lowest level standard of the initial calibration

- MS/MSD – Add 0.50mL of Chloride Calibration Stock to 10mL of the sample selected for MS/MSD. Note: SM4500-Cl^- E recommends the MS/MSD spike concentration to be varied periodically.
- Sample Duplicate – Prepare an additional aliquot of the chosen sample.

4.2 Soil Samples

- Weigh approximately 5g of sample into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of reagent water to each sample container and place in the tumbler.
- Rotate for 2 hours +/- 15minutes.
- Remove the containers from the extractor and allow the leachates to settle. Filter the extract using a syringe filter with a 0.450um pore size filter and analyze the extracts as liquid samples.

Prepare QC samples in the same manner as field samples as follows:

- Method Blank – Weigh approximately 5g of Ottawa sand into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g.
- Laboratory Control Sample – Weigh approximately 5g of Ottawa sand into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of Chloride Laboratory Control Sample to the extraction bottle. Note: Reagent water will not be added to the LCS.
- MS/MSD – Weigh 2 additional 5g aliquots of one of the samples for the MS/MSD. Add 100mL of Chloride Laboratory Control Sample to the extraction bottle. The concentration of the MS/MSD must be varied periodically for SM4500-Cl^- E. Note: Reagent water will not be added to the MS/MSD.

5.0 Method-Specific QC Requirements

5.1 Linear Calibration Range
SM4500-Cl^- E – Performed initially.

5.2 Method Detection Limit Studies
SM4500-Cl^- E – Requires MDL Study to be analyzed over 3-5 days.

6.0 Method-Specific Modifications and/or Clarifications

6.1 The SM4500-Cl^- E reference method specifies to use 21mL nitric acid to prepare the ferric nitrate solution. The EPA 325.2 and EPA 9251 reference methods specify 31.5mL. The laboratory utilizes 44.4mL as outlined in the Konelab application notes.

6.2 The SM4500-Cl^- E reference method includes the addition of polyoxyethylene 23 lauryl ether to the color reagent. This is not required by the other methods and has been omitted from the laboratory's procedure.

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Calculation of Total Chlorine / Total Halogen

Method: SW-846 5050 in conjunction with SW-846 9056A or 9251

Summary of Procedure:

Total chlorine / total halogens (as total chlorine) in a sample is determined by oxygen bomb combustion using SOP SA-GE-010: *Bomb Preparation*. The sample is oxidized by electrical ignition in a bomb containing oxygen under pressure. Under these conditions, chlorine is converted to inorganic chloride and recovered in the DI water bomb washings. The chloride is quantitated using either ion chromatography (SOP SA-GE-115: *Ion Chromatography*) or Konelab (SOP SA-GE-001: *Measurement of Analytes using the Konelab Analyzer*).

Two fundamental conditions must apply before a sample can be considered to have combusted:

1. Some part of the sample must be heated to its ignition temperature to start the combustion. Then in burning, the sample must liberate sufficient heat to support its own combustion.
2. The combustion must produce enough turbulence within the bomb to bring oxygen into the fuel cup for burning the sample completely.

Calculation:

The calculation of Total Chlorine is performed by the LIMS system.

Total Chlorine / Total Halogens (as Total Chlorine)

$$Total\ Halogen, mg/kg, dw = \frac{A \times B \times C}{D \times E}$$

where

A = chloride concentration determined by IC or Konelab in mg/L
B = final volume of bomb washings in liters (0.10L)
C = 1000g/kg conversion factor.
D = sample weight in grams
E = decimal fraction of the percent solids

**NOTE-- Oil matrices will not have a percent solids, therefore the E variable is dropped from the calculation, and the total halogen is reported as mg/kg as is.

Quality Control:

This procedure is a calculation only; therefore, there are no QC items associated. Formal detection limit studies, as described in 40CFR Part 136B and SOP SA-QA-07: *Determination of Detection Limits,* are not required.

Formal demonstrations of capability (DOCs) are described in section 12.2 of SOP SA-GE-010.

Attachment 1c
SOP Summary
Ferrous Iron and Ferric Iron

1.0 Summary

Ferrous iron is determined by reaction with phenanthroline in acid solution producing an orange color with a maximum absorbance of 510nm.

Ferric iron is performed via the calculation below. The Total Iron result is obtained via ICP and/or ICP/MS analysis in accordance with SOP SA-ME-070 or SA-ME-074..

Ferric Iron = Total Iron – Ferrous Iron

2.0 Interferences

The reaction of ferrous iron with 1,10-phenanthroline is free from common interferences. Long storage times or exposure of samples to light must be avoided. Protect from light and analyze as soon as possible.

3.0 Standards and Reagents

3.1 Purchased Reagents

Hydrochloric Acid (HCl) – concentrated, A.C.S. grade
Storage: Acid resistant floors and good drainage. Store away from sunlight, heat and water.

Glacial Acetic Acid (CH_3COOH) – concentrated, A.C.S. grade
Storage: Acid resistant floors and good drainage. Protect from freezing. Store away from sunlight, heat, and ignition sources.

Ammonium Acetate ($NH_4C_2H_3O_2$) – reagent grade

1,10-phenanthroline monohydrate ($C_{12}H_8N_2 \bullet H_2O$) – reagent grade

3.2 Prepared Reagents

Hydrochloric Acid (1:1) – Slowly add 500mL concentrated HCl to about 400mL DI water in a 1-L volumetric flask, cool and dilute to volume with DI water. This solution must be prepared in a hood. Expiration: parent's expiration date or within 1 year of preparation, whichever comes first

Ammonium Acetate Buffer Solution – Dissolve 250g $NH_4C_2H_3O_2$ into 150mL reagent water. Add 700mL concentrated glacial acetic acid. Note: Because $NH_4C_2H_3O_2$ contains a significant amount of iron, new reference standards must be made with each buffer preparation.
Expiration: 5 years (not to exceed expiration of parent reagents).

Phenanthroline Solution (Color Reagent) – Dissolve 100mg $C_{12}H_8N_2 \bullet H_2O$ into 100mL of reagent water. Add 2 drops of concentrated HCl. Discard this solution if it darkens.
Expiration 1 year.

3.3 Standards

Ferrous Ammonium Sulfate [$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$] – reagent grade; Note: The laboratory utilizes two independent lots of ferrous ammonium sulfate. One lot (Mallinckrodt) is utilized as the primary source for calibration standards, laboratory control samples, and matrix spikes. The second lot (Hach) is utilized as the secondary source for the initial calibration verification (ICV).

Ferrous Iron Solution (1000mg/L) – Dissolve 0.7022g $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ (primary source) into 50mL of reagent water in a 100mL volumetric flask. Acidify to pH <2 with 1:1 HCl and dilute to volume with reagent water.

Ferrous Iron Calibration Standards

Standard Level	LIMS Reagent Name	Parent Standard	Aliquot (mL)	Final Volume (mL)	Final Concentration (mg/L)
1	FeCal7	1000mg/L	0.0050	100	0.050
2	FeCal6	1000mg/L	0.010	100	0.10
3	FeCal5	1000mg/L	0.030	100	0.30
4	FeCal4	1000mg/L	0.050	100	0.50
5	FeCal3	1000mg/L	0.10	100	1.0
6	FeCal2	1000mg/L	0.30	100	3.0
7	FeCal1	1000mg/L	0.50	100	5.0

Dilute to volume in reagent water.
Expiration: Prepare fresh daily

Ferrous Iron Matrix Spike Solution (40 mg/L) – Dilute 4mL of the 1000mg/L Fe+2 Cal Stk to a final volume of 100mL with DI water. Expiration: Prepare fresh daily

Ferrous Iron Laboratory Control Sample (2.0 mg/L) – Dilute 2.0 mL of the 40mg/L matrix spike solution to a final volume of 100mL with DI water. Expiration: Prepare fresh daily

Ferrous Iron ICV Stock Solution (1000mg/L) – Dissolve 0.7022g $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ (secondary source) into 50mL of reagent water in a 100mL volumetric flask. Acidify to pH <2 with 1:1 HCl and dilute to volume with reagent water.

Ferrous Iron Initial Calibration Verification (3.0 mg/L) – Dilute 0.30mL of the 1000mg/L Fe+2 ICVSTK to a final volume of 100mL with DI water. Expiration: Prepare fresh daily

4.0 Sample Preparation

4.1 Aqueous Samples

Filter through a 0.45 micron filter prior to analysis. Acidify to pH <2.0 with 1:1 HCl.

Prepare QC samples in the same manner as field samples as follows:

- Laboratory Control Sample – prepared as described above
- Low-Level Laboratory Control Sample (Drinking Water only) – Lowest level standard of the initial calibration
- Matrix Spike/Matrix Spike Duplicate – Add 0.50mL of Ferrous Iron Matrix Spike Solution to 10mL of the chosen sample.

4.2 Soil samples are not performed for this analysis.

5.0 Method-Specific QC Requirements

5.1 Linear Calibration Range required to be performed initially.

5.2 Reporting limit verification required for every batch. An RL check standard must be analyzed at the beginning of each sequence.

5.3 Analyze CCV immediately after ICAL (in addition to the ICV). Recovery must be within 5% of true value.

5.4 Any sample containing a detection greater than 90% of the determined LDR must be diluted to until the raw result is less than 90% of the determined LDR.

6.0 Method-specific Modifications and/or Clarifications

6.1 The preparation of the Ferrous Iron Solution (1000mg/L) has been revised from the reference method to correspond to that outlined in the Konelab Application Notes.

Attachment 1d
SOP Summary
Hexavalent Chromium and Trivalent Chromium

1.0 Summary

The sample is acidified to pH 2 (+/-0.5) and 1,5-diphenylcarbazide (a color reagent) is added. The intensity of the purple color, measured at 540nm, is proportional to the concentration of hexavalent chromium present in the sample.

Trivalent Chromium is performed via the calculation below. The Total Chromium result is obtained via ICP and/or ICP/MS analysis in accordance with SOP SA-ME-070 or SA-ME-074..

Trivalent Chromium = Total Chromium – Hexavalent Chromium

2.0 Interferences

Iron in concentrations of greater than 1mg/L may produce a yellow color, but this is usually not significant when measured at 540nm. Hexavalent molybdenum and mercury salts react to form colored complexes with the color reagent, but the intensities of the colors produced are much lower than for chromium at the specified pH. Mercury and molybdenum concentrations of up to 200mg/L can be tolerated. Vanadium interferes more strongly, but concentrations of up to 10 times that of chromium can be tolerated.

All samples analyzed must be adjusted to a pH range of 1.5-2.5 with sulfuric acid prior to analysis to minimize interference and allow proper color development.

After acidification, soil digestates should be purged with helium to eliminate excess carbon dioxide gas. Bubble formation in the cuvettes can interfere with readings.

The recovery of soluble forms of hexavalent chromium may be low where the sample matrix contains substances that are readily oxidized by hexavalent chromium (a reducing matrix). The measurement pH and oxidation-reduction potential may provide information about the ability of hexavalent chromium to persist in the sample.

3.0 Standards and Reagents

3.1 Purchased Reagents

Sulfuric Acid (H_2SO_4) – concentrated, A.C.S. grade
Storage: Acid resistant floors and good drainage; away from sunlight, heat, and water.

Acetone – concentrated, A.C.S. grade
Storage: Protect from heat, sparks, and flame in a flammable storage area.

1,5-diphenylcarbazide – reagent grade
Storage: Protect from light.

3.2 Prepared Reagents

Sulfuric Acid (50% v/v) – Slowly add 50mL concentrated H_2SO_4 to about 40mL reagent water in a 100mL volumetric flask, cool and dilute to volume with reagent water. This solution must be prepared in a hood.

Color Reagent – Transfer 0.1250g of 1,5-diphenylcarbazide to 25mL volumetric flask. Add 10mL acetone; swirl to dissolve. Dilute to volume with acetone. Discard if reagent becomes discolored.
Storage: Store in the dark in a tightly capped.
Expiration: 1 Week

3.3 Standards

Hexavalent Chromium Solution (10mg/L) – Purchased from Precision Labs.
Storage: Opened: 1 year or the manufacturer's expiration date, whichever is shorter)

Hexavalent Chromium Laboratory Control Sample (0.20mg/L) – Dilute 2mL of the 10mg/L Hexavalent Chromium Solution to 100mL with DI water; Expiration: 1 month from preparation

Hexavalent Chromium Calibration Standards

Standard Level	LIMS Reagent Name	Parent Standard	Aliquot (mL)	Final Volume (mL)	Final Concentration (mg/L)
1	CR6 CAL 7	Cr6 LCS	1.0	100	0.0020
2	CR6 CAL 6	10mg/L Cr6 Solution	0.10	100	0.010
3	CR6 CAL 5	10mg/L Cr6 Solution	0.25	100	0.025
4	CR6 CAL 4	10mg/L Cr6 Solution	0.50	100	0.050
5	CR6 CAL 3	10mg/L Cr6 Solution	1.0	100	0.10
6	CR6 CAL 2	10mg/L Cr6 Solution	3.0	100	0.30
7	CR6 CAL 1	10mg/L Cr6 Solution	5.0	100	0.50

Dilute to volume in reagent water. Expiration: 1 month from preparation

Hexavalent Chromium Initial Calibration Verification Stock (1000mg/L) – Purchased from Hach.
Expiration: Opened: 1 year or the manufacturer's expiration date, whichever is shorter

Hexavalent Chromium ICV Intermediate Standard (10mg/L) – Dilute 1mL of the 1000mg/L CR6 ICV Stock standard to 100 mL with DI water. Expiration: 1 month from preparation

Hexavalent Chromium Initial Calibration Verification (0.30mg/L) – Dilute 3mL of the 10mg/L CR6 ICV Int standard to 100 mL with DI water. Expiration: 1 month from preparation

4.0 Sample Preparation

4.1 Aqueous Samples

Aqueous samples are filtered through a 0.45 micron filter prior to analysis. Adjust the pH of 100mL of sample to pH 1.5–2.5 with 50% H_2SO_4. Use narrow range pH paper during adjustment to verify the proper pH is achieved.

Prepare QC samples in the same manner as field samples as follows:

- Method Blank – reagent water
- Laboratory Control Sample – prepared as described above.
- Low-Level Laboratory Control Sample (drinking water) – Lowest level standard of the initial calibration
- Matrix Spike/Matrix Spike Duplicate – Add 0.20mL of Hexavalent Chromium Solution (10mg/L) Hexavalent Chromium Matrix Spike Solution to 10mL of the chosen sample.

4.1 Soil Samples

Refer to SOP SA-ME-021: *Digestion Procedures for Solids for Hexavalent Chromium* for preparation procedures. Samples are filtered through a 0.45 micron filter prior to analysis. Adjust the pH of 100mL of sample to pH 1.5–2.5 with 50% H_2SO_4. Use narrow range pH paper during adjustment to verify the proper pH is achieved.

Prepare QC samples in the same manner as field samples in accordance with SOP SA-ME-021.

5.0 Method-Specific QC Requirements

SM3500-Cr B – Linear Dynamic Range (LDR) study initially. Reporting limit verification per batch. Any sample containing a detection greater than 90% of the determined LDR must be diluted to until the raw result is less than 90% of the determined LDR.

A CCV must be analyzed immediately following the initial calibration (in addition to the ICV). This CCV must recover within 5% of the true value.

An RL check sample must be analyzed at the beginning of each sequence.

6.0 Method-Specific Modifications and Clarifications

6.1 The SM3500-Cr B reference method states to adjust sample pH to 2 +/-0.5 with 0.2N sulfuric acid. The EPA 7196A reference method states to adjust sample pH to 2 +/-0.5 with 10%sulfuric acid. Adjusting the pH with such a weak solution requires adding >5% volume and therefore diluting the sample. As such, the laboratory has chosen to use 50% sulfuric to adjust the sample pH.

6.2 The EPA 7196A reference method indicates the reductive nature of soil samples must be documented if the pre-digestion matrix spike recovery is not within the recovery limits. The laboratory routinely analyzes a matrix spike duplicate, which helps eliminate technical error as the reason for the failure. For this reason, the laboratory does not perform the tests to determine the reductive nature of the sample unless requested by the client.

6.3 The EPA 7196A reference method states to add the color reagent, then adjust the sample pH to 1.5-2.5. The Konelab instrument automatically adds the color reagent; therefore, the order of these steps is reversed (i.e., the laboratory adjusts the pH then adds the color reagent). Correspondence with EPA indicates this is an acceptable modification to the method.

Attachment 1e
SOP Summary
Ortho-phosphate

1.0 Summary

Orthophosphate reacts with ammonium molybdate and antimony potassium tartrate under acidic conditions to form a complex which, when reduced with ascorbic acid, produces an intense blue color. The color is measured photometrically at 880nm and the absorbance is proportionate to the amount of orthophosphate in the sample.

2.0 Interferences

Samples that are blue in color may interfere. Samples with high concentrations of arsenic may interfere.

3.0 Reagents and Standards

3.1 Purchased Reagents

Antimony Potassium Tartrate [$K(SbO)C_4H_4O_6 \bullet ½\ H_2O$] – reagent grade

Ammonium Molybdate [$(NH_4)_6Mo_7O_{24} \bullet 4H_2O$] – reagent grade. Storage: Isolated from sources of heat and moisture.

Sulfuric Acid (H_2SO_4) – concentrated, A.C.S. grade
Storage: Acid resistant floors and good drainage. Away from sunlight, heat, and water.

Ascorbic Acid – reagent grade
Storage: Isolate from sources of heat and fire.

3.2 Prepared Reagents

Antimony Potassium Tartrate Solution – Dissolve 3.0g $K(SbO)C_4H_4O_6 \bullet ½\ H_2O$ in approximately 500mL of reagent water. Dilute to 1000mL with reagent water. Storage: Store at 4°C in a dark, glass-stoppered bottle.

Ammonium Molybdate Solution – Dissolve 40g $(NH_4)_6Mo_7O_{24} \bullet 4H_2O$ in approximately 800mL of reagent water. Dilute to 1L with reagent water. Storage: Store at 4°C in a plastic bottle.

Sulfuric Acid (10N) – Slowly add 140mL concentrated H_2SO_4 to about 300mL reagent water in a 500-mL volumetric flask, cool and dilute to volume with reagent water. This solution must be prepared in a hood. This will produce heat.

Sulfuric Acid (5N) – Slowly add 70mL concentrated H_2SO_4 to about 400mL reagent water in a 500-mL volumetric flask, cool and dilute to volume with reagent water. This solution must be prepared in a hood. This will produce heat.

Color Reagent 2 – Ascorbic Acid Solution (0.1M) – Dissolve 1.76g ascorbic acid in approximately 50mL of reagent water. Dilute to 100mL with reagent water. Prepare fresh daily.

Color Reagent 1 – Add 75mL Ammonium Molybdate Solution to 250mL 5N sulfuric acid. Mix well. Add 25mL Antimony Potassium Tartrate Solution to this solution and mix well. Expiration: 6 months

Combined Color Reagent – Add 28mL of Color Reagent 1 to 12mL of Color Reagent 2. Mix well. Expiration: Solution is only good for 4 hours.

3.2 Standards

Potassium Phosphate Monobasic (KH_2PO_4) – reagent grade. Dry at 105^{o}C for one hour. (LIMS – K2HPO4). Storage: Store tightly closed in a cool, dry, ventilated area.

Ortho-phosphate Solution (1000mg/L) – Dissolve 5.6235g pre-dried K_2HPO_4 into approximately 800mL of reagent water in a 1L volumetric flask. Dilute to volume with reagent water. (LIMS – PO4 Stock)

Ortho-phosphate Calibration Standards

Standard Level	LIMS Reagent Name	Parent Standard	Volume of Parent Standard (mL)	Final Volume (mL)	Final Concentration (mg/L)
6	PO4 CAL 6	PO4 100ppm	2	100	2.0
5	PO4 CAL 5	PO4 100ppm	1.6	100	1.6
4	PO4 CAL 4	PO4 100ppm	0.8	100	0.80
3	PO4 CAL 3	PO4 100ppm	0.4	100	0.40
2	PO4 CAL 2	PO4 100ppm	0.2	100	0.20
1	PO4 CAL 1	PO4 CAL6	1.0	100	0.020

Dilute to volume in reagent water.
Expiration: 1 month

Ortho-phosphate Laboratory Control Sample (1.2mg/L) – Dilute 0.12mL of the 1000mg/L PO4 Stock solution to a final volume of 100mL with reagent water. Expiration: 1 month

Ortho-phosphate Initial Calibration Verification (1.0mg/L) – This is a purchased standard. Expiration: (Unopened: may be kept until the manufacturer's expiration date), (Opened: may be kept until the manufacturer's expiration date or a maximum of 1 year from opening)

Ortho-phosphate Matrix Spike Solution (100mg/L) – Dilute 10mL of the 1000mg/L PO4 Stock solution to a final volume of 100mL with reagent water. Expiration: 1 month

4.0 Sample Preparation

4.1 Aqueous Samples

Aqueous samples are routinely analyzed with no sample preparation.

Add one drop of phenolphthalein indicator solution to approximately 50mL of sample. If a red color develops add 10N sulfuric acid dropwise until the color dissipates.

Note: Acidic samples must be neutralized with 1N NaOH prior to analysis.

4.1.1 QC Sample Preparation
Method Blank – reagent water

Laboratory Control Sample – Prepared as outlined above

Low-Level Laboratory Control Sample (Drinking Water only) – Lowest level standard of the initial calibration

Matrix Spike/Matrix Spike Duplicate – Add 0.10mL of 100mg/L Matrix Spike Solution to 10mL of the chosen sample. The concentration of the MS/MSD must equal the concentration of the LCS.

Sample Duplicate – Prepare an additional aliquot of the chosen sample.

Prepare all QC in the same manner as samples.

4.2 Soil Samples
Weigh approximately 5g of sample into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of reagent water to each sample container and place in the tumbler.

Rotate for 2 hours +/- 30 minutes.

Remove the containers from the extractor and allow the leachates to settle. Filter the extract using a syringe filter with a 0.45um pore size filter and analyze the extracts as liquid samples.

Add one drop of phenolphthalein indicator solution to approximately 50mL of leachate. If a red color develops add 10N sulfuric acid dropwise until the color dissipates.

Note: Acidic samples must be neutralized with 1N NaOH prior to analysis.

4.2.1 QC Sample Preparation
Method Blank – Weigh approximately 20g of Teflon chips into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g.

Laboratory Control Sample – Weigh approximately 5g of Teflon chips into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 1.0mL of 100mg/L Matrix Spike Solution to the extraction bottle.

Matrix Spike – Weigh an additional 5g aliquot of one of the samples for the MS. Add 1.0mL of 100mg/L Matrix Spike Solution to the extraction bottle. One sample must be chosen from samples 1-10 and one sample from samples 11-20. The concentration of the MS must equal the concentration of the LCS.
Sample Duplicate – If precision is required, weigh one additional 20g aliquot of one of the samples for the SD.

Prepare all QC in the same manner as samples.

5.0 Method-Specific QC Requirements

SM4500-P F requires the MDL study to be analyzed over 3-5 days.

SM4500-P F recommends the matrix spike concentration be rotated.

Attachment 1f
SOP Summary
Sulfate, Total Sulfur, and Heat of Combustion (BTU)

1.0 Summary

Sulfate ion is precipitated in a strongly acidic medium with Barium Chloride. The resulting turbidity is measured photometrically at 405nm and compared with appropriate standard solutions.

Refer to SOP SA-GE-010 for information on the preparation procedures for Total Sulfur and Heat of Combustion (BTU). The attached Work Instruction provides the procedural and calculation information.

2.0 Interferences

Silica at a concentration above 500mg/L in the sample will interfere. Color and turbidity due to the sample matrix can cause positive interferences that must be accounted for by the analysis of sample blanks (i.e. samples analyzed without adding reagents). The absorbance or turbidity of the sample blank is subtracted from the absorbance or turbidity of the treated sample, and the concentration is determined from the corrected response.

3.0 Reagents and Standards

3.1 Purchased Reagents

Barium Chloride ($BaCl_2$) – reagent grade
Storage: Store in a cool, dry, ventilated area. Isolate from heat, moisture, and incompatibles.

Sodium Chloride (NaCl) – reagent grade
Storage: Store in a cool, dry, ventilated area.

Gelatin – (LIMS – Gelatin)

Hydrochloric Acid (HCl) – concentrated, A.C.S. grade
Storage: Store in a cool, dry, ventilated area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.

3.2 Prepared Reagents

Precipitating Solution – In a 1L volumetric flask, dissolve 10g $BaCl_2$, 20g NaCl, and 0.5g of Gelatin in approximately 300mL reagent water. Stir until completely dissolved. Carefully add 5mL concentrated HCl and dilute to volume with reagent water.
Expiration: 3 months

3.3 Purchased Standards

Sodium Sulfate (Na_2SO_4) – anhydrous, reagent grade (LIMS – Na2SO4Salt)
Storage: Store in a tightly closed container in a cool, dry, ventilated area.

Sulfate Calibration Stock Solution (1000mg/L) – In a 1-L volumetric flask, dissolve 1.479g Na_2SO_4 in approximately 800mL reagent water. Dilute to volume with reagent water. Expiration: 1 year

Sulfate Calibration Standards

Standard Level	LIMS Reagent Name	Parent Standard	Volume of Parent Standard (mL)	Final Volume (mL)	Final Concentration (mg/L)
6	SO4 CAL 6	1000mg/L	3.5	100	35
5	SO4 CAL 5	1000mg/L	3.0	100	30
4	SO4 CAL 4	1000mg/L	2.5	100	25
3	SO4 CAL 3	1000mg/L	1.5	100	15
2	SO4 CAL 2	1000mg/L	1.0	100	10
1	SO4 CAL 1	1000mg/L	0.5	100	5

Dilute to volume in reagent water.
Expiration: 3 months

Sulfate Laboratory Control Sample (20mg/L) – Dilute 5mL of the 1000mg/L SO4 Cal stock to 250mL final volume.
Expiration: 3 months

Sulfate Initial Calibration Verification (24.14mg/L – this concentration may change due to changes in the lot number)
Expiration: 3 months

Sulfate Matrix Spike Solution (1000mg/L) – This is the same as the SO4 Calibration Stock Solution
Expiration: 3 months

4.0 Sample Preparation

4.1 Aqueous Samples

Aqueous samples are routinely analyzed with no sample preparation.

4.1.1 QC Sample Preparation

Method Blank – reagent water

Laboratory Control Sample – Prepared as outlined above.

Low-Level Laboratory Control Sample (drinking water) – Lowest level standard of the initial calibration

Matrix Spike/Matrix Spike Duplicate – Add 0.2mL of Sulfate Matrix Spike Solution to 10mL of the chosen sample.

Prepare all QC in the same manner as samples

4.2 Soil Samples

Weigh approximately 5g of sample into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of reagent water to each sample container and place in the tumbler.

Rotate for 2 hours +/- 15 minutes.

Remove the containers from the extractor and allow the leachates to settle. Filter the extract using a syringe filter with a 0.450-um pore size filter and analyze the extracts as liquid samples.

4.2.1 QC Sample Preparation
Method Blank – Weigh approximately 5g of Ottawa sand into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g.

Laboratory Control Sample – Weigh approximately 5g of Ottawa sand into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of Sulfate Laboratory Control Sample to the extraction bottle. Note: Reagent water will not be added to the LCS.

Matrix Spike/Matrix Spike Duplicate – Weigh two additional 5g aliquots of one of the samples for the MS and MSD. Add 100mL of Sulfate Laboratory Control Sample to the extraction bottle. Note: Reagent water will not be added to the MS.

Prepare all QC in the same manner as samples.

5.0 Method-Specific QC Requirements

A CCV must be analyzed every 3-4 sample analyses (EPA 375.4 Section 6.3.4 and EPA 9038 Section 7.3.4).

6.0 Method-Specific Modifications and Clarifications

6.1 The EPA 375.4 reference method states to measure turbidity every 30 seconds for 1-5 minutes after the addition of barium chloride, with the maximum turbidity reading used for determining the Sulfate concentration. The Konelab instrument performs parallel analyses of each sample - one for performing turbidity measurements and one for sulfate determination. The turbidity measurements are made 200 seconds after the color reagent is added and every 28 seconds after that for 240 seconds. The turbidity absorbances are presented on the raw data to indicate if the sample is settling out but do not affect how the sulfate result is determined. The sulfate determination is made 300 seconds after the color reagent is added. Although the Konelab instrument will allow multiple sulfate readings to be made, the instrument cannot select the result associated with the highest turbidity; rather, it will perform an average of the results obtained. As this instrument configuration cannot be changed to use the sulfate reading taken from the highest turbidity replicate.

6.2 The Sulfate Precipitating Solution is prepared in accordance with the Konelab Application Notes which differs from the information provided in the reference method.

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Work Instruction
FGE302:05.24.10:0
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Calculation of Total Sulfur

Method: SW-846 5050 in conjunction with SW-846 9038

Summary of Procedure:

Total sulfur in a sample is determined by oxygen bomb combustion using SOP SA-GE-010: *Bomb Preparation.* The sample is oxidized by electrical ignition in a bomb containing oxygen under pressure. Under these conditions, sulfur is converted to inorganic sulfate and recovered in the DI water bomb washings. Sulfate content is quantitated by using the Konelab (SOP SA-GE-001: *Measurement of Analytes using the Konelab Analyzer).*

Two fundamental conditions must apply before a sample can be considered to have combusted:

1. Some part of the sample must be heated to its ignition temperature to start the combustion. Then in burning, the sample must liberate sufficient heat to support its own combustion.
2. The combustion must produce enough turbulence within the bomb to bring oxygen into the fuel cup for burning the sample completely.

Calculation:

The calculation of Total Chlorine is performed by the LIMS system.

Total Sulfur

$$Total\ Sulfur, mg/kg, dw = \frac{A \times B \times C}{D \times E}$$

where

A = mg S/L (mg SO4/L determined by Konelab divided by 3)
B = final volume of bomb washings in liters (0.10L)
C = 1000g/kg conversion factor.
D = sample weight in grams
E = decimal fraction of the percent solids

**NOTE-- Oil matrices will not have a percent solids, therefore the E variable is dropped from the calculation, and the total halogen is reported as mg/kg as is.

Quality Control:

This procedure is a calculation only; therefore, there are no QC items associated. Formal detection limit studies, as described in 40CFR Part 136B and SOP SA-QA-07: *Determination of Detection Limits,* are not required.

Formal demonstrations of capability (DOCs) are described in section 12.2 of SOP SA-GE-010.

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Work Instruction
FGE301:05.24.10:0
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Calculation of Heat of Combustion

Method: ASTM D240-87

Summary of Procedure:

The gross heat of combustion in a sample is determined by oxygen bomb combustion using SOP SA-GE-010: *Bomb Preparation*. The sample is oxidized by electrical ignition in a bomb containing oxygen under pressure. Accurate time and temperatures are measured throughout the test period. Under these conditions, sulfur is converted to inorganic sulfate and recovered in the DI water bomb washings. The oxygen bomb washings are titrated for nitric acid formation then analyzed by Konelab (SOP SA-GE-001: *Measurement of Analytes using the Konelab Analyzer)* for sulfate content. Variables from the test period and analytical constants are used to calculate the gross heat of combustion (BTU/lb).

Two fundamental conditions must apply before a sample can be considered to have a heat of combustion:

1. Some part of the sample must be heated to its ignition temperature to start the combustion. Then in burning, the sample must liberate sufficient heat to support its own combustion.
2. The combustion must produce enough turbulence within the bomb to bring oxygen into the fuel cup for burning the sample completely.

Calculation:

A spreadsheet is used to determine the final heat of combustion value.

Energy (Water) Equivalent, W

$$W = \frac{[(H * m) + e1 + e3]}{t}$$

where:

W = energy (water) equivalent of the calorimeter in cal/°C
H = heat of combustion of standard benzoic acid. (6318 cal/g)
m = weight (mass) of the benzoic acid standard in grams
e1 = correction for heat of formation of nitric acid in cal
e3 = correction for heat of combustion of firing wire in cal

Temperature Rise, t

$$t = tc - ta$$

where:

t = temperature rise in °C
tc = temperature at beginning of period in which the rate of temperature change with time has become constant in °C
ta = temperature at time of firing in °C

Gross Heat of Combustion (BTU/lb), H

$$H = \frac{[(t * W) - e1 - e2 - e3]}{m} * 1.8$$

where:

H = gross heat of combustion in BTU/lb
t = temperature rise in °C
W = energy equivalent in cal/°C
e1 = correction for heat of formation of nitric acid in cal
e2 = correction for heat of formation of sulfuric acid in cal
e3 = correction for heat of combustion of firing wire in cal
m = weight (mass) of sample in grams
1.8 = conversion factor from cal/g to BTU/lb

Quality Control:

This procedure is a calculation only; therefore, there are no QC items associated. Formal detection limit studies, as described in 40CFR Part 136B and SOP SA-QA-07: *Determination of Detection Limits*, are not required.

Formal demonstrations of capability (DOCs) are described in section 12.2 of SOP SA-GE-010.

The spreadsheet used to determine the heat of combustion result has been verified and protected.

Attachment 3a: Batch QC Frequency

Fraction	Method	MB Frequency[1]	LCS Frequency[1]	LCSD Frequency[1]	LLCS Frequency[1]	MS Frequency[1]	MSD Frequency[1]	SD Frequency[1]
Ammonia	EPA 350.1	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	10% of samples	1 per batch	None
Ammonia	SM4500-NH3 G	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	1 per batch	1 per batch	1 per batch
BTU	ASTM D240-87	None	1 per batch	None	None	None	None	1 per 10
Chloride	EPA 325.2	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	1 per batch	1 per batch	None
Chloride	EPA 9251	1 per batch	1 per batch	1 per batch[2]	None	10% of samples	10% of samples	None
Chloride	SM4500-Cl E	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	1 per batch	1 per batch	1 per batch
Chlorine	EPA 5050/EPA 9251	1 per batch	1 per batch	None	None	None	None	1 per 10
Ferrous Iron	SM3500-Fe B	1 per batch	1 per batch	1 per batch[2]	None	1 per batch	1 per batch	None
Ferric Iron (Calc)	SM3500-Fe B	1 per batch	1 per batch	1 per batch[2]	None	1 per batch	1 per batch	None
Hexavalent Chromium	SM3500-Cr B	1 per batch	1 per batch	1 per batch[2]	None	1 per batch	1 per batch	None
Hexavalent Chromium	SM3500-Cr D	1 per batch	1 per batch	1 per batch[2]	None	1 per batch	1 per batch	None
Hexavalent Chromium	EPA 7196A	1 per batch	1 per batch	1 per batch[2]	None	10% of samples	1 per batch	None
Ortho-phosphate	EPA 365.1	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	10% of samples	1 per batch	None
Ortho-phosphate	SM4500-P F	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	1 per batch	1 per batch	1 per batch
Sulfate	EPA 375.4	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	1 per batch	1 per batch	None
Sulfate	EPA 9038	1 per batch	1 per batch	1 per batch[2]	None	10% of samples	10% of samples	None
Total Sulfur	EPA 5050/EPA 9038	1 per batch	1 per batch	None	None	None	None	1 per 10
Trivalent Chromium (Calc)	SM3500-Cr B	1 per batch	1 per batch	1 per batch[2]	None	1 per batch	1 per batch	None
Trivalent Chromium (Calc)	SM3500-Cr D	1 per batch	1 per batch	1 per batch[2]	None	1 per batch	1 per batch	None
Trivalent Chromium (Calc)	EPA 7196A	1 per batch	1 per batch	1 per batch[2]	None	10% of samples	1 per batch	None

[1]Batch Definition = Up to 20 field samples prepared and/or analyzed together within 24 hours
[2]If insufficient sample provided for MS/MSD.
[3]For Drinking Water analyses only.

Attachment 3b: QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL) Multi-point: Minimum 3 standards and 1 blank	Daily	Correlation ≥0.995	Recalibrate Note: SM3500-Fe B and SM3500-Cr B do not allow quadratic curves
Second Source Initial Calibration Verification (ICV)	Immediately following the initial calibration; at a minimum, quarterly	Within ±10% of the true value	Recalibrate
Continuing Calibration Verification (CCV)	SM3500-Fe B, SM3500-Cr B: Immediately following ICAL Sulfate: At the beginning and end of the analysis, and after every 3-4 samples All Others: At the beginning and end of the analysis, and after every 10 samples	SM3500-Fe B, SM3500-Cr B: Within ±5% of the true value All Others:	Terminate the analysis, fix the problem and reanalyze the previous samples.
Initial Calibration Blank (ICB)	After ICV	|result| <MDL	Terminate the analysis, correct problem and reanalyze the previous 10 samples.
Continuing Calibration Blank (CCB)	After every CCV	|result| <MDL	Terminate the analysis, correct problem and reanalyze the previous 10 samples.
Batch Frequency	<=20 field samples or 24 hours	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	|result| <MDL	Evaluate according to SOP SA-QA-017

QC Item	Frequency	Criteria	Corrective Action
Laboratory Control Sample (LCS)	One per batch	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient sample provided for MS/MSD/SD	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Low-Level Laboratory Control Sample (LLCS)	Drinking Water Only: One per batch	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Reporting Limit Verification (RLV)	SM3500-Fe B, SM3500-Cr B: One per batch	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Matrix Spike (MS)	Refer to QC Frequency Table	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	Refer to QC Frequency Table	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Sample Duplicate (SD)	Refer to QC Frequency Table	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Linear Calibration Range (LCR) (Also called Linear Dynamic Range, LDR)	Upon method/instrument set-up, and when LCR verification fails acceptance criteria.	Within ±10% of the true value	This determines the range at which the instrument is considered linear. The highest concentration of the calibration curve can not exceed the highest concentration of the LCR study.
Linear Calibration Range Verification (LCRV) (Also called Linear Dynamic Range Verification, LDRV)	Every 6 months	Within ±10% of the initial value	Re-perform the LCR study

QC Item	Frequency	Criteria	Corrective Action
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 (Unsupervised work cannot begin until successful IDOC is obtained.)
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination and annually thereafter EPA 365.1: Upon method/instrument set-up, per analyte/method/matrix combination and every 6 months thereafter	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4: Instrument Maintenance and Troubleshooting

Instrument Labeling
Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log
A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance
Refer to the instrument manufacturer's guides for trouble-shooting items.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Sample Probe	X							Clean and inspect daily
Dispenser Rods			X					Clean and lube monthly
Fetcher Rods			X					Clean and lube monthly
Incubator Rods			X					Clean and lube monthly
Water Lines			X					Clean with isopropyl alcohol monthly

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Contingency Plan
Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Attachment 5: Maximum Contaminant Levels (MCL) Table

Primary Drinking Water Regulations	
Contaminant	**MCL (mg/L)**
Nitrate (as N)	10
Nitrite (as N)	1

Secondary Drinking Water Regulations	
Contaminant	**MCL (mg/L)**
Chloride	250
Sulfate	250

18.0 Revision History

Summary of Changes from Previous Revision:

- Updated to new TestAmerica SOP template. Significant formatting and content changes made. Complete re-work/re-structuring of the SOP text and Attachments.
- Removed all references to Cyanide, TKN, Total Phosphorus, Free Cyanide, Tannins & Lignins, and Hydrazine analyses. Cyanide, TKN, and TP are now analyzed using the Latchet Autoanalyzer. Free Cyanide and Hydrazine are no longer performed.
- Added references/procedures for trivalent chromium, ferric iron, Total Chlorine, Total Sulfur, and Heat of Combustion (BTU).
- Revised standard and reagent information to reflect current laboratory practice.
- Incorporated information previously listed as alpha-character revisions.
- Incorporated preparation SOPs GE15 (Ammonia). These SOPs are now obsolete.
- Added requirement to perform LFB at MRL for SDWA.
- In response to FLDOH audit finding a pH range of 1.5-2.5 will be used for Hexavalent Chromium samples analyzed by EPA 7196 and SM3500Cr.
- Revised Ortho-phosphate soil holding time from 28 days to 48 hours to coincide with the holding time set up in LIMS.
- Revised Standard Methods references for Hexavalent Chromium and Ferrous Iron. The letter version changed with the newest revision of these methods.
- Updated batch QC requirements to match referenced methods
- Created default QC requirements for the methods that do not have batch QC requirements.
- Updated soil preparation procedures to include the use of a solid matrix for the method blank and laboratory control sample.
- Removed the reference to CoE Method 3-154 for Ammonia soil prep. The lab performs a modified leach procedure.
- Revised method blank criteria to be <MDL.
- Revised Sodium Hydroxide TWA criteria. Added safety information for formation of mercuric thiocyanate.
- Added requirement that if a sample or extract is filtered, the associated QC must also be filtered.
- Added reference to Historical Data and TALS HDT module.
- Added note that the requirements in the Attachments supersede the requirements listed in the body of the SOP.
- Removed requirement to utilize second source for IDOC. Attestation form cover page is no longer required.
- Removed reference to pH meter. Narrow range pH paper is used instead.

Summary of Changes from Previous Revision:
SA-GE-001 Rev. 2 (Eff. Date 06/23/2013) to SA-GE-001 Rev. 2A (Eff. Date 09/05/2013)
Minor corrections were made as outlined below. This revision is administrative in nature, does constitute a change to existing laboratory procedures, and does not require analyst re-training.

- Revised method reference to reflect 2011 revision of the methods in the 22nd Edition of Standards Methods. This change stems from a SC DHEC Technical Review Deficiency. The following method reference was revised:

  Standard Methods for the Examination of Water and Wastewater; American Public Health Association; Washington, D.C.

  - SM4500-Cl^- E: *Chloride, Automated Ferricyanide Method*; (Approved 1997; Editorial Revision 2011)

Summary of Changes from Previous Revision:
SA-GE-001 Rev. 2A (Eff. Date 09/05/2013) to SA-GE-001 Rev. 2B (Eff. Date 02/05/2014)
A minor clarification was made as outlined below. This revision is administrative in nature, does constitute a change to existing laboratory procedures, and does not require analyst re-training.

- Re-worded Method Modification Section for Hexavalent Chromium to clarify the laboratory's procedure and the actual modification being made (i.e., the order of addition of the color reagent and the pH adjustment has been reversed from that listed in the reference method). This clarification stems from the 12/2013 FL DOH Audit Finding.
- Included reference to Safety Data Sheets (SDS).

Appendix C

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York
FA8903-10-R-9999-R044

ID	WBS	WBS Codes	Task Name	Duration	Start	Finish	Predecessors
1	**1**	**PM001**	**Kick Off Meeting at Griffiss AFB**	**1 day**	**Mon 1/10/11**	**Mon 1/10/11**	
2	**2**	**PM002**	**PMP**	**58 days**	**Tue 1/11/11**	**Thu 3/31/11**	**1**
3	2.1	2.1	Draft PMP Submittal	20 days	Tue 1/11/11	Mon 2/7/11	
4	2.2	2.2	Air Force Review of Draft PMP	20 days	Tue 2/8/11	Mon 3/7/11	3
5	2.3	2.3	Final PMP	18 days	Tue 3/8/11	Thu 3/31/11	4
6	**3**	**PM003**	**Monthly Progress Meetings**	**1261 days**	**Mon 2/28/11**	**Mon 12/28/15**	
7	3.1	3.1	Monthly Progress Meetings 1	1 day	Mon 2/28/11	Mon 2/28/11	
8	3.2	3.2	Monthly Progress Meetings 2	1 day	Mon 3/28/11	Mon 3/28/11	
9	3.3	3.3	Monthly Progress Meetings 3	1 day	Mon 4/25/11	Mon 4/25/11	
10	3.4	3.4	Monthly Progress Meetings 4	1 day	Tue 5/31/11	Tue 5/31/11	
11	3.5	3.5	Monthly Progress Meetings 5	1 day	Mon 6/27/11	Mon 6/27/11	
12	3.6	3.6	Monthly Progress Meetings 6	1 day	Mon 7/25/11	Mon 7/25/11	
13	3.7	3.7	Monthly Progress Meetings 7	1 day	Mon 8/29/11	Mon 8/29/11	
14	3.8	3.8	Monthly Progress Meetings 8	1 day	Mon 9/26/11	Mon 9/26/11	
15	3.9	3.9	Monthly Progress Meetings 9	1 day	Mon 10/31/11	Mon 10/31/11	
16	3.10	3.10	Monthly Progress Meetings 10	1 day	Mon 11/28/11	Mon 11/28/11	
17	3.11	3.11	Monthly Progress Meetings 11	1 day	Tue 12/27/11	Tue 12/27/11	
18	3.12	3.12	Monthly Progress Meetings 12	1 day	Mon 1/30/12	Mon 1/30/12	
19	3.13	3.13	Monthly Progress Meetings 13	1 day	Mon 2/27/12	Mon 2/27/12	
20	3.14	3.14	Monthly Progress Meetings 14	1 day	Mon 3/26/12	Mon 3/26/12	
21	3.15	3.15	Monthly Progress Meetings 15	1 day	Mon 4/30/12	Mon 4/30/12	
22	3.16	3.16	Monthly Progress Meetings 16	1 day	Tue 5/29/12	Tue 5/29/12	
23	3.17	3.17	Monthly Progress Meetings 17	1 day	Mon 6/25/12	Mon 6/25/12	
24	3.18	3.18	Monthly Progress Meetings 18	1 day	Mon 7/30/12	Mon 7/30/12	
25	3.19	3.19	Monthly Progress Meetings 19	1 day	Mon 8/27/12	Mon 8/27/12	
26	3.20	3.20	Monthly Progress Meetings 20	1 day	Mon 9/24/12	Mon 9/24/12	
27	3.21	3.21	Monthly Progress Meetings 21	1 day	Mon 10/29/12	Mon 10/29/12	
28	3.22	3.22	Monthly Progress Meetings 22	1 day	Mon 11/26/12	Mon 11/26/12	
29	3.23	3.23	Monthly Progress Meetings 23	1 day	Mon 12/31/12	Mon 12/31/12	
30	3.24	3.24	Monthly Progress Meetings 24	1 day	Mon 1/28/13	Mon 1/28/13	
31	3.25	3.25	Monthly Progress Meetings 25	1 day	Mon 2/25/13	Mon 2/25/13	
32	3.26	3.26	Monthly Progress Meetings 26	1 day	Tue 6/25/13	Tue 6/25/13	
33	3.27	3.27	Monthly Progress Meetings 27	1 day	Mon 4/29/13	Mon 4/29/13	
34	3.28	3.28	Monthly Progress Meetings 28	1 day	Tue 5/28/13	Tue 5/28/13	
35	3.29	3.29	Monthly Progress Meetings 29	1 day	Mon 6/24/13	Mon 6/24/13	
36	3.30	3.30	Monthly Progress Meetings 30	1 day	Mon 7/29/13	Mon 7/29/13	
37	3.31	3.31	Monthly Progress Meetings 31	1 day	Mon 8/26/13	Mon 8/26/13	
38	3.32	3.32	Monthly Progress Meetings 32	1 day	Mon 9/30/13	Mon 9/30/13	
39	3.33	3.33	Monthly Progress Meetings 33	1 day	Mon 10/28/13	Mon 10/28/13	
40	3.34	3.34	Monthly Progress Meetings 34	1 day	Mon 11/25/13	Mon 11/25/13	
41	3.35	3.35	Monthly Progress Meetings 35	1 day	Mon 12/30/13	Mon 12/30/13	
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43	3.37	3.37	Monthly Progress Meetings 37	1 day	Mon 2/24/14	Mon 2/24/14	
44	3.38	3.38	Monthly Progress Meetings 38	1 day	Mon 3/31/14	Mon 3/31/14	
45	3.39	3.39	Monthly Progress Meetings 39	1 day	Mon 4/28/14	Mon 4/28/14	
46	3.40	3.40	Monthly Progress Meetings 40	1 day	Tue 5/27/14	Tue 5/27/14	
47	3.41	3.41	Monthly Progress Meetings 41	1 day	Mon 6/30/14	Mon 6/30/14	
48	3.42	3.42	Monthly Progress Meetings 42	1 day	Mon 7/28/14	Mon 7/28/14	
49	3.43	3.43	Monthly Progress Meetings 43	1 day	Mon 8/25/14	Mon 8/25/14	
50	3.44	3.44	Monthly Progress Meetings 44	1 day	Mon 9/29/14	Mon 9/29/14	
51	3.45	3.45	Monthly Progress Meetings 45	1 day	Mon 10/27/14	Mon 10/27/14	
52	3.46	3.46	Monthly Progress Meetings 46	1 day	Mon 11/24/14	Mon 11/24/14	
53	3.47	3.47	Monthly Progress Meetings 47	1 day	Mon 12/29/14	Mon 12/29/14	
54	3.48	3.48	Monthly Progress Meetings 48	1 day	Mon 1/26/15	Mon 1/26/15	

Project: Project1
Date: Thu 3/24/11

Task
Split
Progress
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Summary
Project Summary
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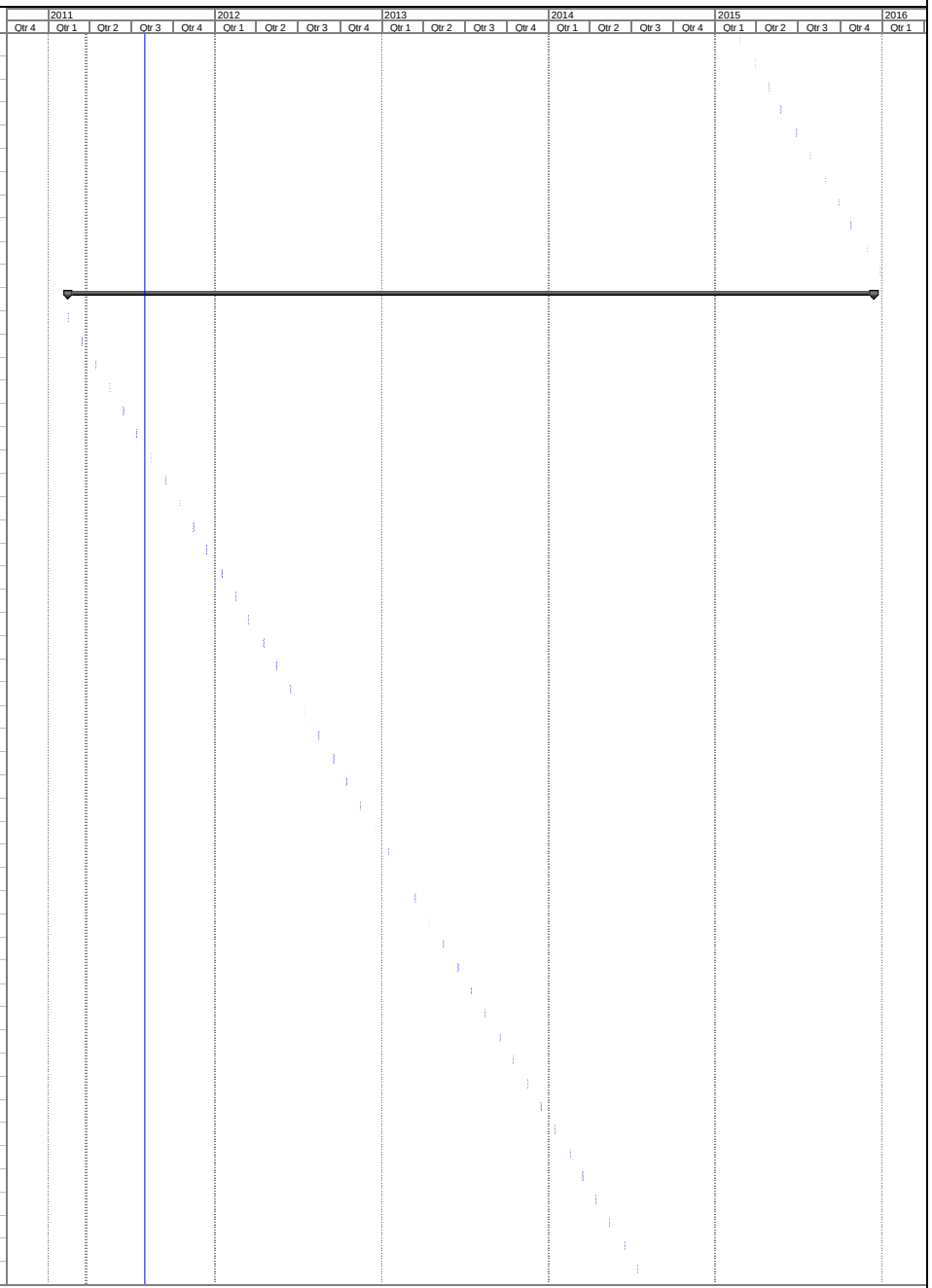
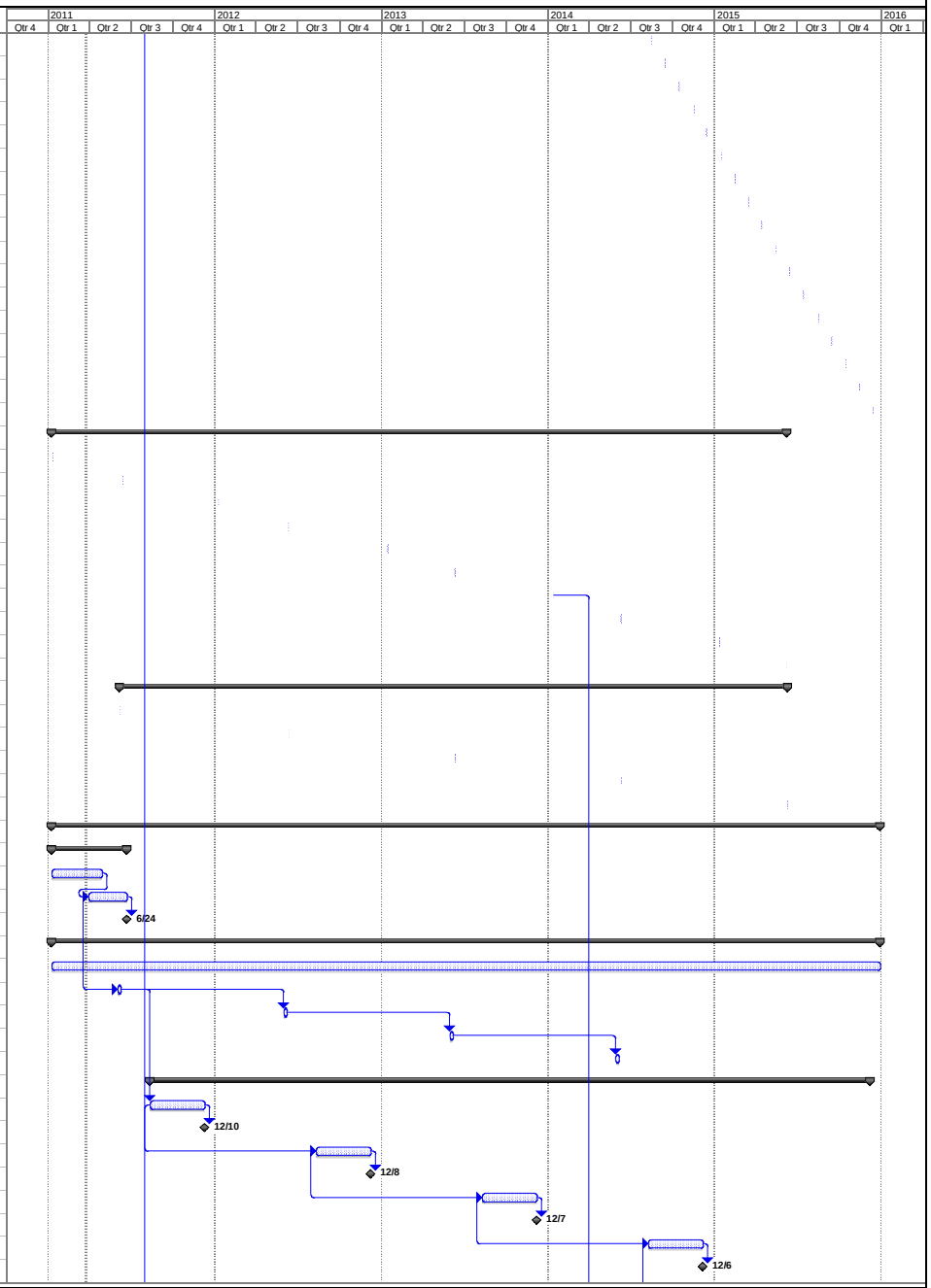
ID	WBS	WBS Codes	Task Name	Duration	Start	Finish	Predecessors
55	3.49	3.49	Monthly Progress Meetings 49	1 day	Mon 2/23/15	Mon 2/23/15	
56	3.50	3.50	Monthly Progress Meetings 50	1 day	Mon 3/30/15	Mon 3/30/15	
57	3.51	3.51	Monthly Progress Meetings 51	1 day	Mon 4/27/15	Mon 4/27/15	
58	3.52	3.52	Monthly Progress Meetings 52	1 day	Tue 5/26/15	Tue 5/26/15	
59	3.53	3.53	Monthly Progress Meetings 53	1 day	Mon 6/29/15	Mon 6/29/15	
60	3.54	3.54	Monthly Progress Meetings 54	1 day	Mon 7/27/15	Mon 7/27/15	
61	3.55	3.55	Monthly Progress Meetings 55	1 day	Mon 8/31/15	Mon 8/31/15	
62	3.56	3.56	Monthly Progress Meetings 56	1 day	Mon 9/28/15	Mon 9/28/15	
63	3.57	3.57	Monthly Progress Meetings 57	1 day	Mon 10/26/15	Mon 10/26/15	
64	3.58	3.58	Monthly Progress Meetings 58	1 day	Mon 11/30/15	Mon 11/30/15	
65	3.59	3.59	Monthly Progress Meetings 59	1 day	Mon 12/28/15	Mon 12/28/15	
66	**4**	**PM004**	**CPSMR Monthly Progress Reports**	**1261 days**	**Tue 2/15/11**	**Tue 12/15/15**	
67	4.1	4.1	CPSMR Monthly Progress Reports 1	1 day	Tue 2/15/11	Tue 2/15/11	
68	4.2	4.2	CPSMR Monthly Progress Reports 2	1 day	Tue 3/15/11	Tue 3/15/11	
69	4.3	4.3	CPSMR Monthly Progress Reports 3	1 day	Fri 4/15/11	Fri 4/15/11	
70	4.4	4.4	CPSMR Monthly Progress Reports 4	1 day	Mon 5/16/11	Mon 5/16/11	
71	4.5	4.5	CPSMR Monthly Progress Reports 5	1 day	Wed 6/15/11	Wed 6/15/11	
72	4.6	4.6	CPSMR Monthly Progress Reports 6	1 day	Fri 7/15/11	Fri 7/15/11	
73	4.7	4.7	CPSMR Monthly Progress Reports 7	1 day	Mon 8/15/11	Mon 8/15/11	
74	4.8	4.8	CPSMR Monthly Progress Reports 8	1 day	Thu 9/15/11	Thu 9/15/11	
75	4.9	4.9	CPSMR Monthly Progress Reports 9	1 day	Mon 10/17/11	Mon 10/17/11	
76	4.10	4.10	CPSMR Monthly Progress Reports 10	1 day	Tue 11/15/11	Tue 11/15/11	
77	4.11	4.11	CPSMR Monthly Progress Reports 11	1 day	Thu 12/15/11	Thu 12/15/11	
78	4.12	4.12	CPSMR Monthly Progress Reports 12	1 day	Mon 1/16/12	Mon 1/16/12	
79	4.13	4.13	CPSMR Monthly Progress Reports 13	1 day	Wed 2/15/12	Wed 2/15/12	
80	4.14	4.14	CPSMR Monthly Progress Reports 14	1 day	Thu 3/15/12	Thu 3/15/12	
81	4.15	4.15	CPSMR Monthly Progress Reports 15	1 day	Mon 4/16/12	Mon 4/16/12	
82	4.16	4.16	CPSMR Monthly Progress Reports 16	1 day	Tue 5/15/12	Tue 5/15/12	
83	4.17	4.17	CPSMR Monthly Progress Reports 17	1 day	Fri 6/15/12	Fri 6/15/12	
84	4.18	4.18	CPSMR Monthly Progress Reports 18	1 day	Mon 7/16/12	Mon 7/16/12	
85	4.19	4.19	CPSMR Monthly Progress Reports 19	1 day	Wed 8/15/12	Wed 8/15/12	
86	4.20	4.20	CPSMR Monthly Progress Reports 20	1 day	Mon 9/17/12	Mon 9/17/12	
87	4.21	4.21	CPSMR Monthly Progress Reports 21	1 day	Mon 10/15/12	Mon 10/15/12	
88	4.22	4.22	CPSMR Monthly Progress Reports 22	1 day	Thu 11/15/12	Thu 11/15/12	
89	4.23	4.23	CPSMR Monthly Progress Reports 23	1 day	Mon 12/17/12	Mon 12/17/12	
90	4.24	4.24	CPSMR Monthly Progress Reports 24	1 day	Tue 1/15/13	Tue 1/15/13	
91	4.25	4.25	CPSMR Monthly Progress Reports 25	1 day	Fri 2/15/13	Fri 2/15/13	
92	4.26	4.26	CPSMR Monthly Progress Reports 26	1 day	Fri 3/15/13	Fri 3/15/13	
93	4.27	4.27	CPSMR Monthly Progress Reports 27	1 day	Mon 4/15/13	Mon 4/15/13	
94	4.28	4.28	CPSMR Monthly Progress Reports 28	1 day	Wed 5/15/13	Wed 5/15/13	
95	4.29	4.29	CPSMR Monthly Progress Reports 29	1 day	Mon 6/17/13	Mon 6/17/13	
96	4.30	4.30	CPSMR Monthly Progress Reports 30	1 day	Mon 7/15/13	Mon 7/15/13	
97	4.31	4.31	CPSMR Monthly Progress Reports 31	1 day	Thu 8/15/13	Thu 8/15/13	
98	4.32	4.32	CPSMR Monthly Progress Reports 32	1 day	Mon 9/16/13	Mon 9/16/13	
99	4.33	4.33	CPSMR Monthly Progress Reports 33	1 day	Tue 10/15/13	Tue 10/15/13	
100	4.34	4.34	CPSMR Monthly Progress Reports 34	1 day	Fri 11/15/13	Fri 11/15/13	
101	4.35	4.35	CPSMR Monthly Progress Reports 35	1 day	Mon 12/16/13	Mon 12/16/13	
102	4.36	4.36	CPSMR Monthly Progress Reports 36	1 day	Wed 1/15/14	Wed 1/15/14	
103	4.37	4.37	CPSMR Monthly Progress Reports 37	1 day	Mon 2/17/14	Mon 2/17/14	
104	4.38	4.38	CPSMR Monthly Progress Reports 38	1 day	Mon 3/17/14	Mon 3/17/14	
105	4.39	4.39	CPSMR Monthly Progress Reports 39	1 day	Tue 4/15/14	Tue 4/15/14	
106	4.40	4.40	CPSMR Monthly Progress Reports 40	1 day	Thu 5/15/14	Thu 5/15/14	
107	4.41	4.41	CPSMR Monthly Progress Reports 41	1 day	Mon 6/16/14	Mon 6/16/14	
108	4.42	4.42	CPSMR Monthly Progress Reports 42	1 day	Tue 7/15/14	Tue 7/15/14	



Project: Project1
Date: Thu 3/24/11

Task | Progress | Summary | External Tasks | Split
Split | Milestone | Project Summary | External MileTask

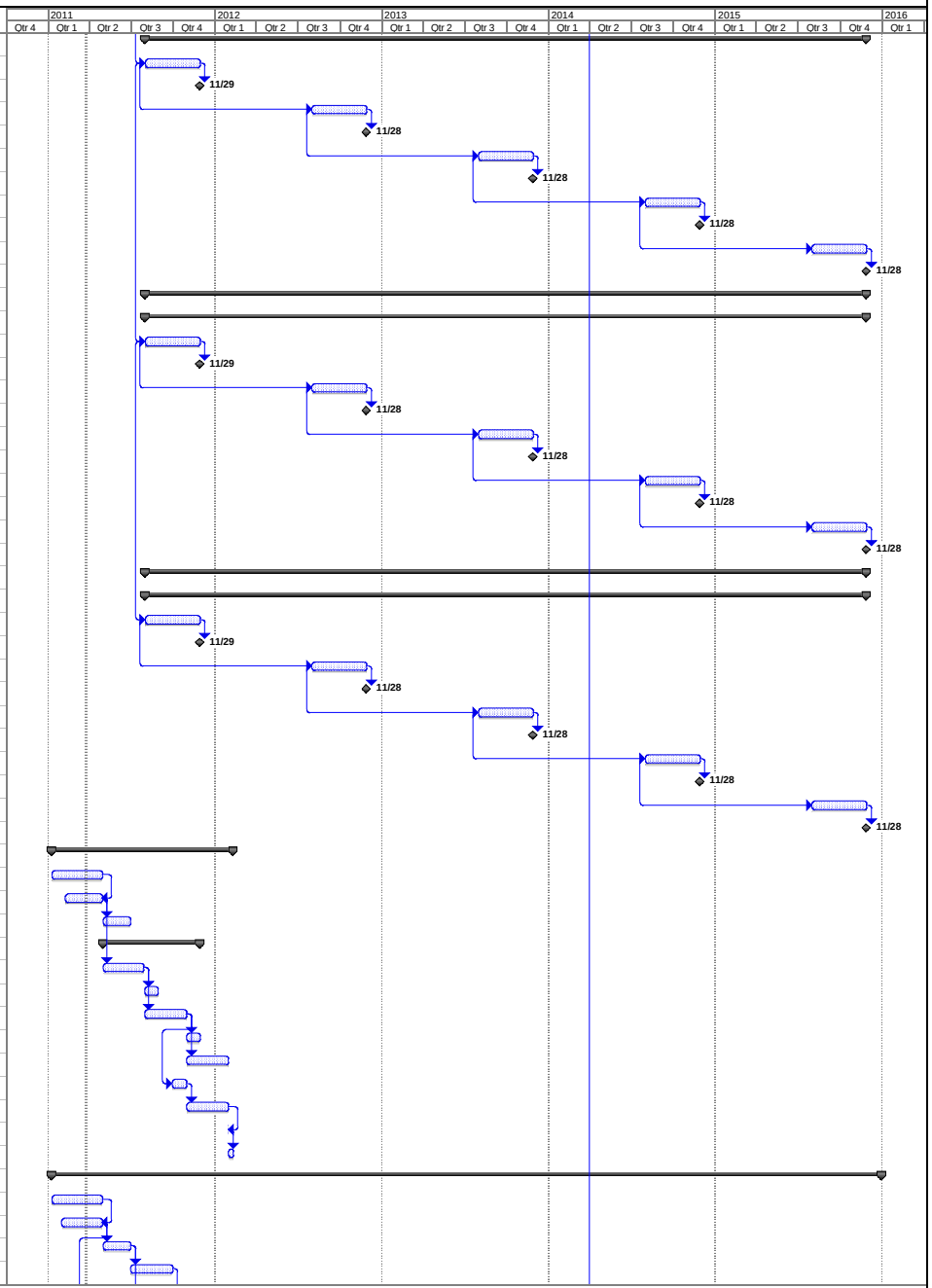
ID	WBS	WBS Codes	Task Name	Duration	Start	Finish	Predecessors
109	4.43	4.43	CPSMR Monthly Progress Reports 43	1 day	Fri 8/15/14	Fri 8/15/14	
110	4.44	4.44	CPSMR Monthly Progress Reports 44	1 day	Mon 9/15/14	Mon 9/15/14	
111	4.45	4.45	CPSMR Monthly Progress Reports 45	1 day	Wed 10/15/14	Wed 10/15/14	
112	4.46	4.46	CPSMR Monthly Progress Reports 46	1 day	Mon 11/17/14	Mon 11/17/14	
113	4.47	4.47	CPSMR Monthly Progress Reports 47	1 day	Mon 12/15/14	Mon 12/15/14	
114	4.48	4.48	CPSMR Monthly Progress Reports 48	1 day	Thu 1/15/15	Thu 1/15/15	
115	4.49	4.49	CPSMR Monthly Progress Reports 49	1 day	Mon 2/16/15	Mon 2/16/15	
116	4.50	4.50	CPSMR Monthly Progress Reports 50	1 day	Mon 3/16/15	Mon 3/16/15	
117	4.51	4.51	CPSMR Monthly Progress Reports 51	1 day	Wed 4/15/15	Wed 4/15/15	
118	4.52	4.52	CPSMR Monthly Progress Reports 52	1 day	Fri 5/15/15	Fri 5/15/15	
119	4.53	4.53	CPSMR Monthly Progress Reports 53	1 day	Mon 6/15/15	Mon 6/15/15	
120	4.54	4.54	CPSMR Monthly Progress Reports 54	1 day	Wed 7/15/15	Wed 7/15/15	
121	4.55	4.55	CPSMR Monthly Progress Reports 55	1 day	Mon 8/17/15	Mon 8/17/15	
122	4.56	4.56	CPSMR Monthly Progress Reports 56	1 day	Tue 9/15/15	Tue 9/15/15	
123	4.57	4.57	CPSMR Monthly Progress Reports 57	1 day	Thu 10/15/15	Thu 10/15/15	
124	4.58	4.58	CPSMR Monthly Progress Reports 58	1 day	Mon 11/16/15	Mon 11/16/15	
125	4.59	4.59	CPSMR Monthly Progress Reports 59	1 day	Tue 12/15/15	Tue 12/15/15	
126	**5**	**PM005**	**Semi Annual on Site Meetings**	**1151 days**	**Mon 1/10/11**	**Mon 6/8/15**	
127	5.1	5.1	Semi Annual Meeting 1	1 day	Mon 1/10/11	Mon 1/10/11	
128	5.2	5.2	Semi Annual Meeting 2	1 day	Mon 6/13/11	Mon 6/13/11	
129	5.3	5.3	Semi Annual Meeting 3	1 day	Mon 1/9/12	Mon 1/9/12	
130	5.4	5.4	Semi Annual Meeting 4	1 day	Mon 6/11/12	Mon 6/11/12	
131	5.5	5.5	Semi Annual Meeting 5	1 day	Mon 1/14/13	Mon 1/14/13	
132	5.6	5.6	Semi Annual Meeting 6	1 day	Mon 6/10/13	Mon 6/10/13	
133	5.7	5.7	Semi Annual Meeting 7	1 day	Mon 1/13/14	Mon 1/13/14	
134	5.8	5.8	Semi Annual Meeting 8	1 day	Mon 6/9/14	Mon 6/9/14	
135	5.9	5.9	Semi Annual Meeting 9	1 day	Mon 1/12/15	Mon 1/12/15	
136	5.10	5.10	Semi Annual Meeting 10	1 day	Mon 6/8/15	Mon 6/8/15	
137	**6**	**PM006**	**RAB/BCT Meeting**	**1046 days**	**Wed 6/8/11**	**Wed 6/10/15**	
138	6.1	6.1	RAB/BCT Meeting 1	1 day	Wed 6/8/11	Wed 6/8/11	
139	6.2	6.2	RAB/BCT Meeting 2	1 day	Wed 6/13/12	Wed 6/13/12	
140	6.3	6.3	RAB/BCT Meeting 3	1 day	Wed 6/12/13	Wed 6/12/13	
141	6.4	6.4	RAB/BCT Meeting 4	1 day	Wed 6/11/14	Wed 6/11/14	
142	6.5	6.5	RAB/BCT Meeting 5	1 day	Wed 6/10/15	Wed 6/10/15	
143	**7**	**LF001**	**LF001 - Landfill 1 (Optimized Exit Strategy)**	**1298 days**	**Mon 1/10/11**	**Thu 12/31/15**	
144	**7.1**	**LF001.1**	**LF001 - Planning Documents**	**119 days**	**Mon 1/10/11**	**Fri 6/24/11**	
145	7.1.1	LF001.1.1	LF001 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	Mon 1/10/11	Fri 4/29/11	
146	7.1.2	LF001.1.2	LF001 - Design and Remedy Optimization Documents	85 edays	Thu 3/31/11	Fri 6/24/11	145FS-22 days
147	7.1.3	LF001.1.3	LF001 - Acceptance of Final Design and Remedy Optimization Documents	0 days	Fri 6/24/11	Fri 6/24/11	146
148	**7.2**	**LF001.2**	**LF001 - Field Work**	**1298 days**	**Mon 1/10/11**	**Thu 12/31/15**	
149	7.2.1	LF001.2.1	LF001 - Landfill O&M	1816 edays	Mon 1/10/11	Thu 12/31/15	
150	7.2.2	LF001.2.2	LF001 - Monitoring Year 1	5 days	Fri 6/3/11	Thu 6/9/11	146SS+46 days
151	7.2.3	LF001.2.3	LF001 - Monitoring Year 2	5 days	Fri 6/1/12	Thu 6/7/12	150FS+255 days
152	7.2.4	LF001.2.4	LF001 - Monitoring Year 3	5 days	Fri 5/31/13	Thu 6/6/13	151FS+255 days
153	7.2.5	LF001.2.5	LF001 - Monitoring Year 4	5 days	Fri 5/30/14	Thu 6/5/14	152FS+255 days
154	**7.3**	**LF001.3**	**LF001 - Reporting**	**1127 days**	**Fri 8/12/11**	**Tue 12/8/15**	
155	7.3.1	LF001.3.1	LF001 - Long-Term Monitoring Report - Year 1	120 edays	Fri 8/12/11	Sat 12/10/11	150FS+46 days
156	7.3.2	LF001.3.2	LF001 - Acceptance of Long-Term Monitoring Report - Year 1	0 days	Sat 12/10/11	Sat 12/10/11	155
157	7.3.3	LF001.3.3	LF001 - Long-Term Monitoring Report - Year 2	120 edays	Fri 8/10/12	Sat 12/8/12	155SS+260 days
158	7.3.4	LF001.3.4	LF001 - Acceptance of Long-Term Monitoring Report - Year 2	0 days	Sat 12/8/12	Sat 12/8/12	157
159	7.3.5	LF001.3.5	LF001 - Long-Term Monitoring Report - Year 3	120 edays	Fri 8/9/13	Sat 12/7/13	157SS+260 days
160	7.3.6	LF001.3.6	LF001 - Acceptance of Long-Term Monitoring Report - Year 3	0 days	Sat 12/7/13	Sat 12/7/13	159
161	7.3.7	LF001.3.7	LF001 - Long-Term Monitoring Report - Year 4	120 edays	Fri 8/8/14	Sat 12/6/14	159SS+260 days
162	7.3.8	LF001.3.8	LF001 - Acceptance of Long-Term Monitoring Report - Year 4	0 days	Sat 12/6/14	Sat 12/6/14	161



Project: Project1
Date: Thu 3/24/11

Task | Progress | Summary | External Tasks | Split
Split | Milestone | Project Summary | External MileTask

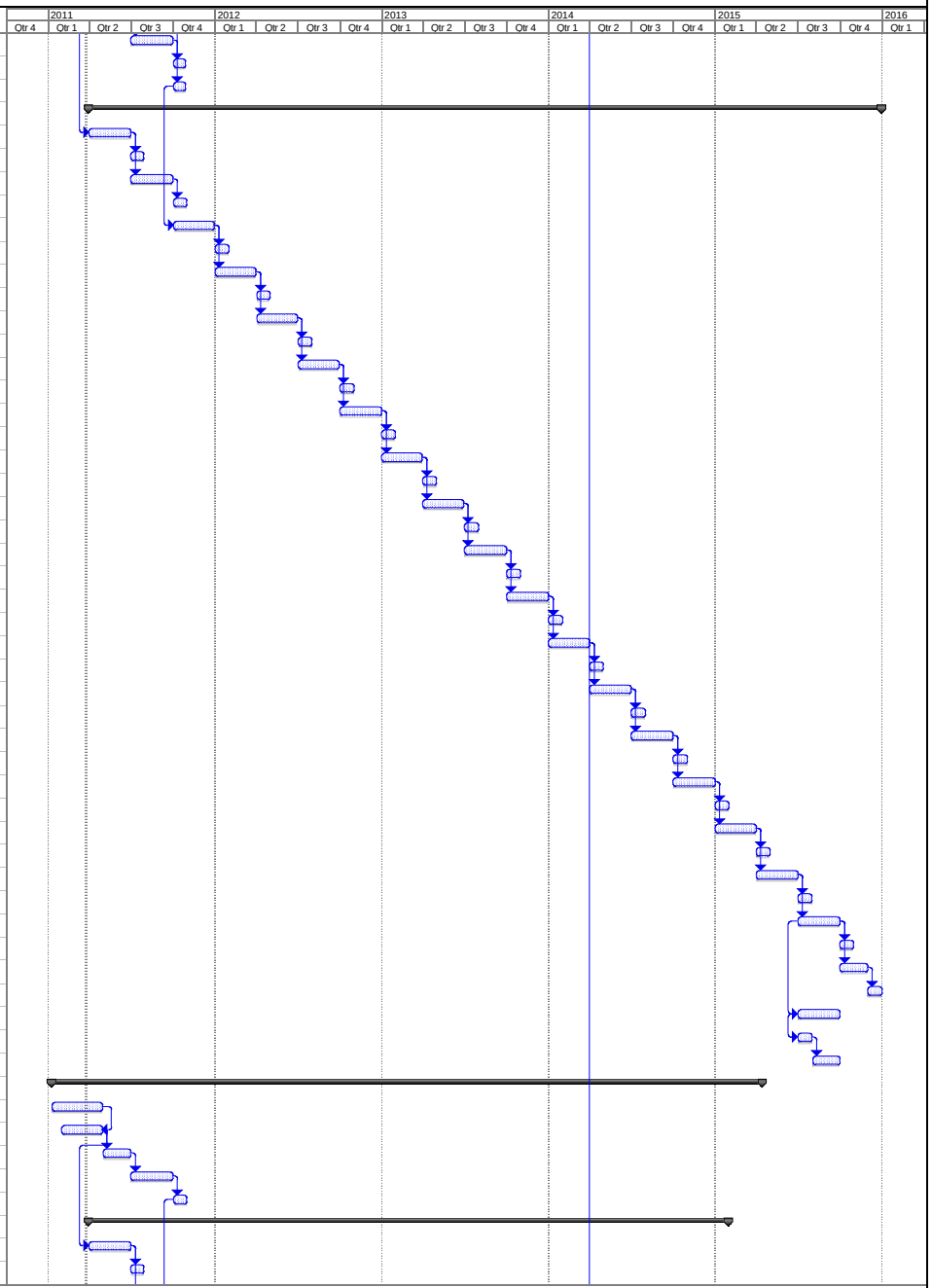
ID	WBS	WBS Codes	Task Name	Duration	Start	Finish	Predecessors
595	**34.1**	**SD52-04.1**	**SD52-04 - LUCIC Inspection**	**1129 days**	**Mon 8/1/11**	**Sat 11/28/15**	
596	34.1.1	SD52-04.1.1	SD52-04 - LUCIC Inspection Report - Year 1	120 edays	Mon 8/1/11	Tue 11/29/11	584SS
597	34.1.2	SD52-04.1.2	SD52-04 - Acceptance of LUCIC Inspection Report - Year 1	0 days	Tue 11/29/11	Tue 11/29/11	596
598	34.1.3	SD52-04.1.3	SD52-04 - LUCIC Inspection Report - Year 2	120 edays	Tue 7/31/12	Wed 11/28/12	596SS+261 days
599	34.1.4	SD52-04.1.4	SD52-04 - Acceptance of LUCIC Inspection Report - Year 2	0 days	Wed 11/28/12	Wed 11/28/12	598
600	34.1.5	SD52-04.1.5	SD52-04 - LUCIC Inspection Report - Year 3	120 edays	Wed 7/31/13	Thu 11/28/13	598SS+261 days
601	34.1.6	SD52-04.1.6	SD52-04 - Acceptance of LUCIC Inspection Report - Year 3	0 days	Thu 11/28/13	Thu 11/28/13	600
602	34.1.7	SD52-04.1.7	SD52-04 - LUCIC Inspection Report - Year 4	120 edays	Thu 7/31/14	Fri 11/28/14	600SS+261 days
603	34.1.8	SD52-04.1.8	SD52-04 - Acceptance of LUCIC Inspection Report - Year 4	0 days	Fri 11/28/14	Fri 11/28/14	602
604	34.1.9	SD52-04.1.9	SD52-04 - LUCIC Inspection Report - Year 5	120 edays	Fri 7/31/15	Sat 11/28/15	602SS+261 days
605	34.1.10	SD52-04.1.10	SD52-04 - Acceptance of LUCIC Inspection Report - Year 5	0 days	Sat 11/28/15	Sat 11/28/15	604
606	**35**	**SD52-05**	**SD52-05 - Building 817 Chlorinated Plume (Status Quo)**	**1129 days**	**Mon 8/1/11**	**Sat 11/28/15**	
607	**35.1**	**SD52-05.1**	**SD52-05 - LUCIC Inspection**	**1129 days**	**Mon 8/1/11**	**Sat 11/28/15**	
608	35.1.1	SD52-05.1.1	SD52-05 - LUCIC Inspection Report - Year 1	120 edays	Mon 8/1/11	Tue 11/29/11	596SS
609	35.1.2	SD52-05.1.2	SD52-05 - Acceptance of LUCIC Inspection Report - Year 1	0 days	Tue 11/29/11	Tue 11/29/11	608
610	35.1.3	SD52-05.1.3	SD52-05 - LUCIC Inspection Report - Year 2	120 edays	Tue 7/31/12	Wed 11/28/12	608SS+261 days
611	35.1.4	SD52-05.1.4	SD52-05 - Acceptance of LUCIC Inspection Report - Year 2	0 days	Wed 11/28/12	Wed 11/28/12	610
612	35.1.5	SD52-05.1.5	SD52-05 - LUCIC Inspection Report - Year 3	120 edays	Wed 7/31/13	Thu 11/28/13	610SS+261 days
613	35.1.6	SD52-05.1.6	SD52-05 - Acceptance of LUCIC Inspection Report - Year 3	0 days	Thu 11/28/13	Thu 11/28/13	612
614	35.1.7	SD52-05.1.7	SD52-05 - LUCIC Inspection Report - Year 4	120 edays	Thu 7/31/14	Fri 11/28/14	612SS+261 days
615	35.1.8	SD52-05.1.8	SD52-05 - Acceptance of LUCIC Inspection Report - Year 4	0 days	Fri 11/28/14	Fri 11/28/14	614
616	35.1.9	SD52-05.1.9	SD52-05 - LUCIC Inspection Report - Year 5	120 edays	Fri 7/31/15	Sat 11/28/15	614SS+261 days
617	35.1.10	SD52-05.1.10	SD52-05 - Acceptance of LUCIC Inspection Report - Year 5	0 days	Sat 11/28/15	Sat 11/28/15	616
618	**36**	**SS062**	**SS062 - AOC-9 (Status Quo)**	**1129 days**	**Mon 8/1/11**	**Sat 11/28/15**	
619	**36.1**	**SS062.1**	**SS062 - LUCIC Inspection**	**1129 days**	**Mon 8/1/11**	**Sat 11/28/15**	
620	36.1.1	SS062.1.1	SS062 - LUCIC Inspection Report - Year 1	120 edays	Mon 8/1/11	Tue 11/29/11	608SS
621	36.1.2	SS062.1.2	SS062 - Acceptance of LUCIC Inspection Report - Year 1	0 days	Tue 11/29/11	Tue 11/29/11	620
622	36.1.3	SS062.1.3	SS062 - LUCIC Inspection Report - Year 2	120 edays	Tue 7/31/12	Wed 11/28/12	620SS+261 days
623	36.1.4	SS062.1.4	SS062 - Acceptance of LUCIC Inspection Report - Year 2	0 days	Wed 11/28/12	Wed 11/28/12	622
624	36.1.5	SS062.1.5	SS062 - LUCIC Inspection Report - Year 3	120 edays	Wed 7/31/13	Thu 11/28/13	622SS+261 days
625	36.1.6	SS062.1.6	SS062 - Acceptance of LUCIC Inspection Report - Year 3	0 days	Thu 11/28/13	Thu 11/28/13	624
626	36.1.7	SS062.1.7	SS062 - LUCIC Inspection Report - Year 4	120 edays	Thu 7/31/14	Fri 11/28/14	624SS+261 days
627	36.1.8	SS062.1.8	SS062 - Acceptance of LUCIC Inspection Report - Year 4	0 days	Fri 11/28/14	Fri 11/28/14	626
628	36.1.9	SS062.1.9	SS062 - LUCIC Inspection Report - Year 5	120 edays	Fri 7/31/15	Sat 11/28/15	626SS+261 days
629	36.1.10	SS062.1.10	SS062 - Acceptance of LUCIC Inspection Report - Year 5	0 days	Sat 11/28/15	Sat 11/28/15	628
630	**37**	**SD041**	**SD041 - Nose Doc #1 (Closure Unrestricted Reuse)**	**285 days**	**Mon 1/10/11**	**Fri 2/10/12**	
631	37.1	SD041.1	SD041 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	Mon 1/10/11	Fri 4/29/11	
632	37.2	SD041.2	SD041 -Prepare and Submit Design/plan remedy optimization	59 days	Tue 2/8/11	Fri 4/29/11	631FF
633	37.3	SD041.3	SD041 - AFCEE Review and Acceptance of Design/Plan Remedy Optimization	44 days	Mon 5/2/11	Thu 6/30/11	632
634	**37.4**	**SD041.4**	**SD041 - Conduct Monitoring, Operations & Maintenance**	**152 days**	**Mon 5/2/11**	**Tue 11/29/11**	
635	37.4.1	SD041.4.1	SD041 - Conduct Monitoring, Prep and Submit CY11 Q2 LTO/LTM Report	65 days	Mon 5/2/11	Fri 7/29/11	632
636	37.4.2	SD041.4.2	SD041 - AFCEE Review and Acceptance of CY11 Q2 LTO/LTM Report	21 days	Mon 8/1/11	Mon 8/29/11	635
637	37.4.3	SD041.4.3	SD041 - Conduct Monitoring, Prep and Submit CY11 Q3 LTO/LTM Report	66 days	Mon 8/1/11	Mon 10/31/11	635
638	37.4.4	SD041.4.4	SD041 - AFCEE Review and Acceptance of CY11 Q3 LTO/LTM Report	21 days	Tue 11/1/11	Tue 11/29/11	637
639	37.5	SD041.5	SD041 - Achieve Site Closure	65 days	Tue 11/1/11	Mon 1/30/12	637
640	37.6	SD041.6	SD041 - Prepare and Submit Closure Report	22 days	Fri 9/30/11	Mon 10/31/11	637FS-22 days
641	37.7	SD041.7	SD041 - AFCEE Review and Acceptance of Closure Report	65 days	Tue 11/1/11	Mon 1/30/12	640
642	37.8	SD041.8	SD041 - NYSDEC Closure of Spill Number	1 day	Mon 1/30/12	Mon 1/30/12	641FF
643	37.9	SD041.9	SD041 - Wells Decommissioned	9 days	Tue 1/31/12	Fri 2/10/12	642
644	**38**	**SS054**	**SS054 - BLDG 781 Pumphouse (Optimized Exit Strategy)**	**1299 days**	**Mon 1/10/11**	**Thu 12/31/15**	
645	38.1	SS054.1	SS054 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	Mon 1/10/11	Fri 4/29/11	
646	38.2	SS054.2	SS054 - Design/Plan Remedy Optimation	65 days	Mon 1/31/11	Fri 4/29/11	645FF
647	38.3	SS054.3	SS054 - AFCEE Review and Acceptance of Design/Plan Remedy Optimization	44 days	Mon 5/2/11	Thu 6/30/11	646
648	38.4	SS054.4	SS054 - Complete Supplemental Investigation, submit Investigation Report	66 days	Fri 7/1/11	Fri 9/30/11	647



Project: Project1
Date: Thu 3/24/11

Task | Progress | Summary | External Tasks | Split
Split | Milestone | Project Summary | External MileTask

ID	WBS	WBS Codes	Task Name	Duration	Start	Finish	Predecessors
649	38.5	SS054.5	SS054 - Implement Remedy Optimization Systems and Submit Report	66 days	Fri 7/1/11	Fri 9/30/11	647
650	38.6	SS054.6	SS054 - AFCEE Review and Acceptance of Investigation Report	20 days	Mon 10/3/11	Fri 10/28/11	648
651	38.7	SS054.7	SS054 - AFCEE Review and Acceptance of Optimization System and Report	20 days	Mon 10/3/11	Fri 10/28/11	649
652	**38.8**	**SS054.8**	**SS054 - Conduct Monitoring, Operations & Maintenance**	**1240 days**	**Fri 4/1/11**	**Thu 12/31/15**	
653	38.8.1	SS054.8.1	SS054 - Conduct Monitoring, Prep and Submit CY11 Q2 LTO/LTM Report	65 days	Fri 4/1/11	Thu 6/30/11	646FS-21 days
654	38.8.2	SS054.8.2	SS054 - AFCEE Review and Acceptance of CY11 Q2 LTO/LTM Report	21 days	Fri 7/1/11	Fri 7/29/11	653
655	38.8.3	SS054.8.3	SS054 - Conduct Monitoring, Prep and Submit CY11 Q3 LTO/LTM Report	66 days	Fri 7/1/11	Fri 9/30/11	653
656	38.8.4	SS054.8.4	SS054 - AFCEE Review and Acceptance of CY11 Q3 LTO/LTM Report	21 days	Mon 10/3/11	Mon 10/31/11	655
657	38.8.5	SS054.8.5	SS054 - Conduct Monitoring, Prep and Submit CY11 Q4 LTO/LTM Report	65 days	Mon 10/3/11	Fri 12/30/11	651SS
658	38.8.6	SS054.8.6	SS054 - AFCEE Review and Acceptance of CY11 Q4 LTO/LTM Report	22 days	Mon 1/2/12	Tue 1/31/12	657
659	38.8.7	SS054.8.7	SS054 - Conduct Monitoring, Prep and Submit CY12 Q1 LTO/LTM Report	65 days	Mon 1/2/12	Fri 3/30/12	657
660	38.8.8	SS054.8.8	SS054 - AFCEE Review and Acceptance of CY12 Q1 LTO/LTM Report	21 days	Mon 4/2/12	Mon 4/30/12	659
661	38.8.9	SS054.8.9	SS054 - Conduct Monitoring, Prep and Submit CY12 Q2 LTO/LTM Report	65 days	Mon 4/2/12	Fri 6/29/12	659
662	38.8.10	SS054.8.10	SS054 - AFCEE Review and Acceptance of CY12 Q2 LTO/LTM Report	22 days	Mon 7/2/12	Tue 7/31/12	661
663	38.8.11	SS054.8.11	SS054 - Conduct Monitoring, Prep and Submit CY12 Q3 LTO/LTM Report	65 days	Mon 7/2/12	Fri 9/28/12	661
664	38.8.12	SS054.8.12	SS054 - AFCEE Review and Acceptance of CY12 Q3 LTO/LTM Report	23 days	Mon 10/1/12	Wed 10/31/12	663
665	38.8.13	SS054.8.13	SS054 - Conduct Monitoring, Prep and Submit CY12 Q4 LTO/LTM Report	66 days	Mon 10/1/12	Mon 12/31/12	663
666	38.8.14	SS054.8.14	SS054 - AFCEE Review and Acceptance of CY12 Q4 LTO/LTM Report	22 days	Tue 1/1/13	Wed 1/30/13	665
667	38.8.15	SS054.8.15	SS054 - Conduct Monitoring, Prep and Submit CY13 Q1 LTO/LTM Report	64 days	Tue 1/1/13	Fri 3/29/13	665
668	38.8.16	SS054.8.16	SS054 - AFCEE Review and Acceptance of CY13 Q1 LTO/LTM Report	22 days	Mon 4/1/13	Tue 4/30/13	667
669	38.8.17	SS054.8.17	SS054 - Conduct Monitoring, Prep and Submit CY13 Q2 LTO/LTM Report	65 days	Mon 4/1/13	Fri 6/28/13	667
670	38.8.18	SS054.8.18	SS054 - AFCEE Review and Acceptance of CY13 Q2 LTO/LTM Report	23 days	Mon 7/1/13	Wed 7/31/13	669
671	38.8.19	SS054.8.19	SS054 - Conduct Monitoring, Prep and Submit CY13 Q3 LTO/LTM Report	66 days	Mon 7/1/13	Mon 9/30/13	669
672	38.8.20	SS054.8.20	SS054 - AFCEE Review and Acceptance of CY13 Q3 LTO/LTM Report	23 days	Tue 10/1/13	Thu 10/31/13	671
673	38.8.21	SS054.8.21	SS054 - Conduct Monitoring, Prep and Submit CY13 Q4 LTO/LTM Report	66 days	Tue 10/1/13	Tue 12/31/13	671
674	38.8.22	SS054.8.22	SS054 - AFCEE Review and Acceptance of CY13 Q4 LTO/LTM Report	23 days	Wed 1/1/14	Fri 1/31/14	673
675	38.8.23	SS054.8.23	SS054 - Conduct Monitoring, Prep and Submit CY14 Q1 LTO/LTM Report	64 days	Wed 1/1/14	Mon 3/31/14	673
676	38.8.24	SS054.8.24	SS054 - AFCEE Review and Acceptance of CY14 Q1 LTO/LTM Report	22 days	Tue 4/1/14	Wed 4/30/14	675
677	38.8.25	SS054.8.25	SS054 - Conduct Monitoring, Prep and Submit CY14 Q2 LTO/LTM Report	65 days	Tue 4/1/14	Mon 6/30/14	675
678	38.8.26	SS054.8.26	SS054 - AFCEE Review and Acceptance of CY14 Q2 LTO/LTM Report	23 days	Tue 7/1/14	Thu 7/31/14	677
679	38.8.27	SS054.8.27	SS054 - Conduct Monitoring, Prep and Submit CY14 Q3 LTO/LTM Report	66 days	Tue 7/1/14	Tue 9/30/14	677
680	38.8.28	SS054.8.28	SS054 - AFCEE Review and Acceptance of CY14 Q3 LTO/LTM Report	23 days	Wed 10/1/14	Fri 10/31/14	679
681	38.8.29	SS054.8.29	SS054 - Conduct Monitoring, Prep and Submit CY14 Q4 LTO/LTM Report	66 days	Wed 10/1/14	Wed 12/31/14	679
682	38.8.30	SS054.8.30	SS054 - AFCEE Review and Acceptance of CY14 Q4 LTO/LTM Report	22 days	Thu 1/1/15	Fri 1/30/15	681
683	38.8.31	SS054.8.31	SS054 - Conduct Monitoring, Prep and Submit CY15 Q1 LTO/LTM Report	64 days	Thu 1/1/15	Tue 3/31/15	681
684	38.8.32	SS054.8.32	SS054 - AFCEE Review and Acceptance of CY15 Q1 LTO/LTM Report	22 days	Wed 4/1/15	Thu 4/30/15	683
685	38.8.33	SS054.8.33	SS054 - Conduct Monitoring, Prep and Submit CY15 Q2 LTO/LTM Report	65 days	Wed 4/1/15	Tue 6/30/15	683
686	38.8.34	SS054.8.34	SS054 - AFCEE Review and Acceptance of CY15 Q2 LTO/LTM Report	23 days	Wed 7/1/15	Fri 7/31/15	685
687	38.8.35	SS054.8.35	SS054 - Conduct Monitoring, Prep and Submit CY15 Q3 LTO/LTM Report	66 days	Wed 7/1/15	Wed 9/30/15	685
688	38.8.36	SS054.8.36	SS054 - AFCEE Review and Acceptance of CY15 Q3 LTO/LTM Report	22 days	Thu 10/1/15	Fri 10/30/15	687
689	38.8.37	SS054.8.37	SS054 - Conduct Monitoring, Prep and Submit CY15 Q4 LTO/LTM Report	43 days	Thu 10/1/15	Mon 11/30/15	687
690	38.8.38	SS054.8.38	SS054 - AFCEE Review and Acceptance of CY15 Q4 LTO/LTM Report	23 days	Tue 12/1/15	Thu 12/31/15	689
691	38.9	SS054.9	SS054 - Optimization Exit Strategy	66 days	Wed 7/1/15	Wed 9/30/15	687SS
692	38.10	SS054.10	SS054 - Prepare and Submit Optimized Exit Strategy Report	23 days	Wed 7/1/15	Fri 7/31/15	691SS
693	38.11	SS054.11	SS054 - AFCEE Review and Acceptance of Optimized Exit Strategy Report	43 days	Mon 8/3/15	Wed 9/30/15	692
694	**39**	**SS020**	**SS020 - Tank Farms 1 & 3 (Closure Unrestricted Reuse)**	**1112 days**	**Mon 1/10/11**	**Tue 4/14/15**	
695	39.1	SS020.1	SS020 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	Mon 1/10/11	Fri 4/29/11	
696	39.2	SS020.2	SS020 -Prepare and Submit Design/plan remedy optimization	65 days	Mon 1/31/11	Fri 4/29/11	695FF
697	39.3	SS020.3	SS020 - AFCEE Review and Acceptance of Design/Plan Remedy Optimization	44 days	Mon 5/2/11	Thu 6/30/11	696
698	39.4	SS020.4	SS020 - Implement Remedy Optimization Systems and Submit Report	66 days	Fri 7/1/11	Fri 9/30/11	697
699	39.5	SS020.5	SS020 - AFCEE Review and Acceptance of Optimization System and Report	21 days	Mon 10/3/11	Mon 10/31/11	698
700	**39.6**	**SS020.6**	**SS020 - Conduct Monitoring, Operations & Maintenance**	**1001 days**	**Fri 4/1/11**	**Fri 1/30/15**	
701	39.6.1	SS020.6.1	SS020 - Conduct Monitoring, Prep and Submit CY11 Q2 LTO/LTM Report	65 days	Fri 4/1/11	Thu 6/30/11	696FS-21 days
702	39.6.2	SS020.6.2	SS020 - AFCEE Review and Acceptance of CY11 Q2 LTO/LTM Report	21 days	Fri 7/1/11	Fri 7/29/11	701



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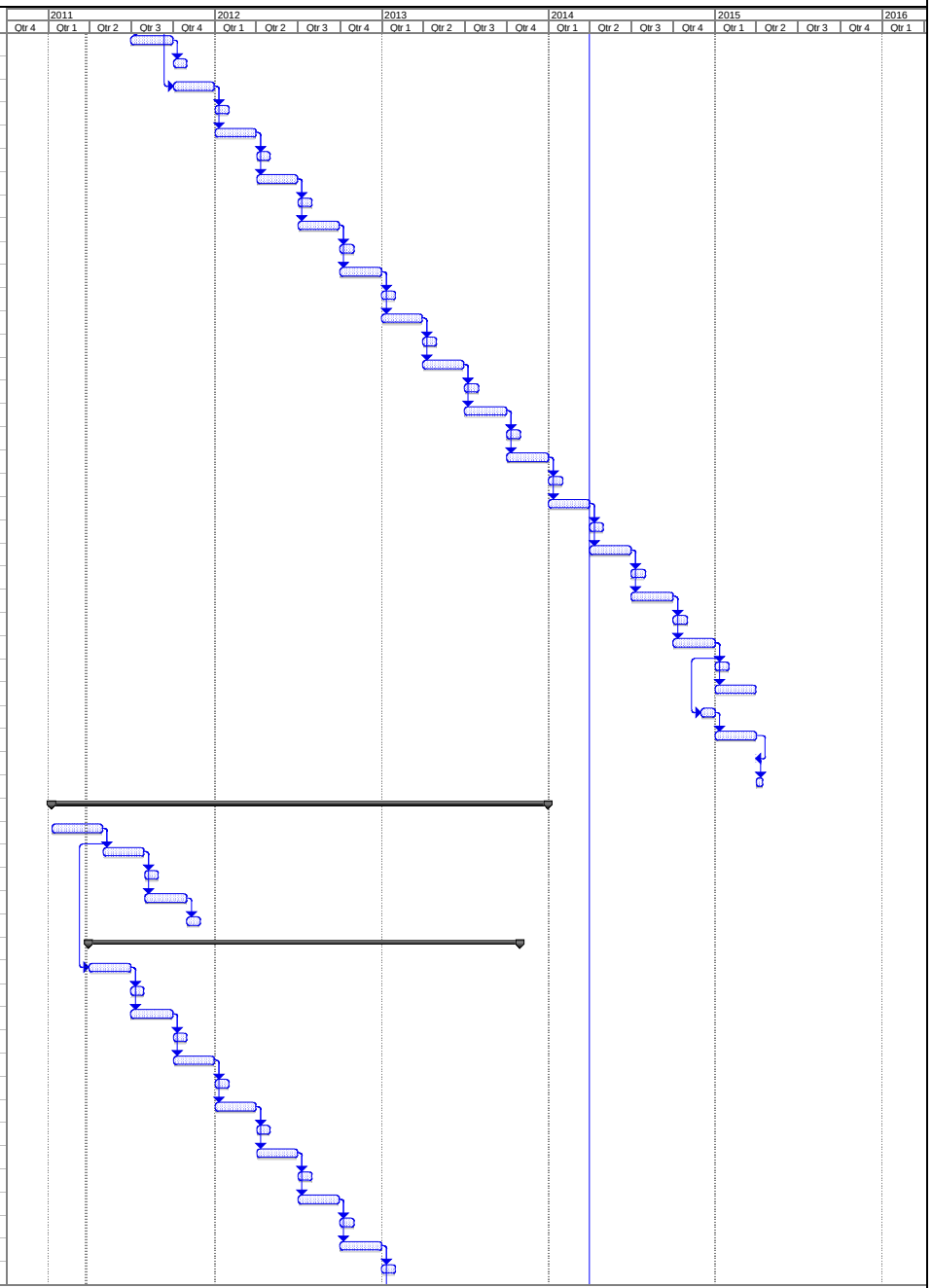
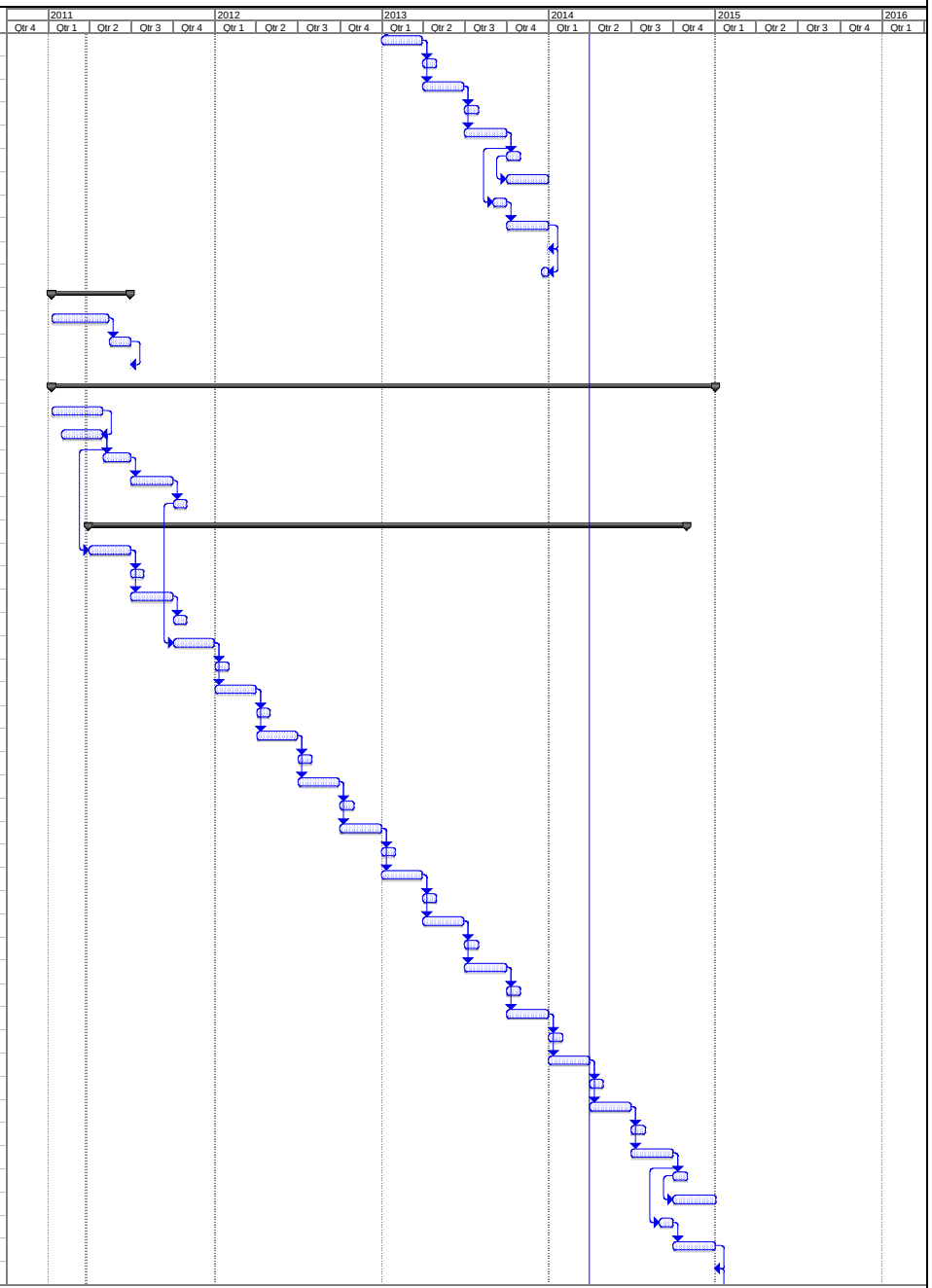
Task | Progress | Summary | External Tasks | Split
Split | Milestone | Project Summary | External MileTask

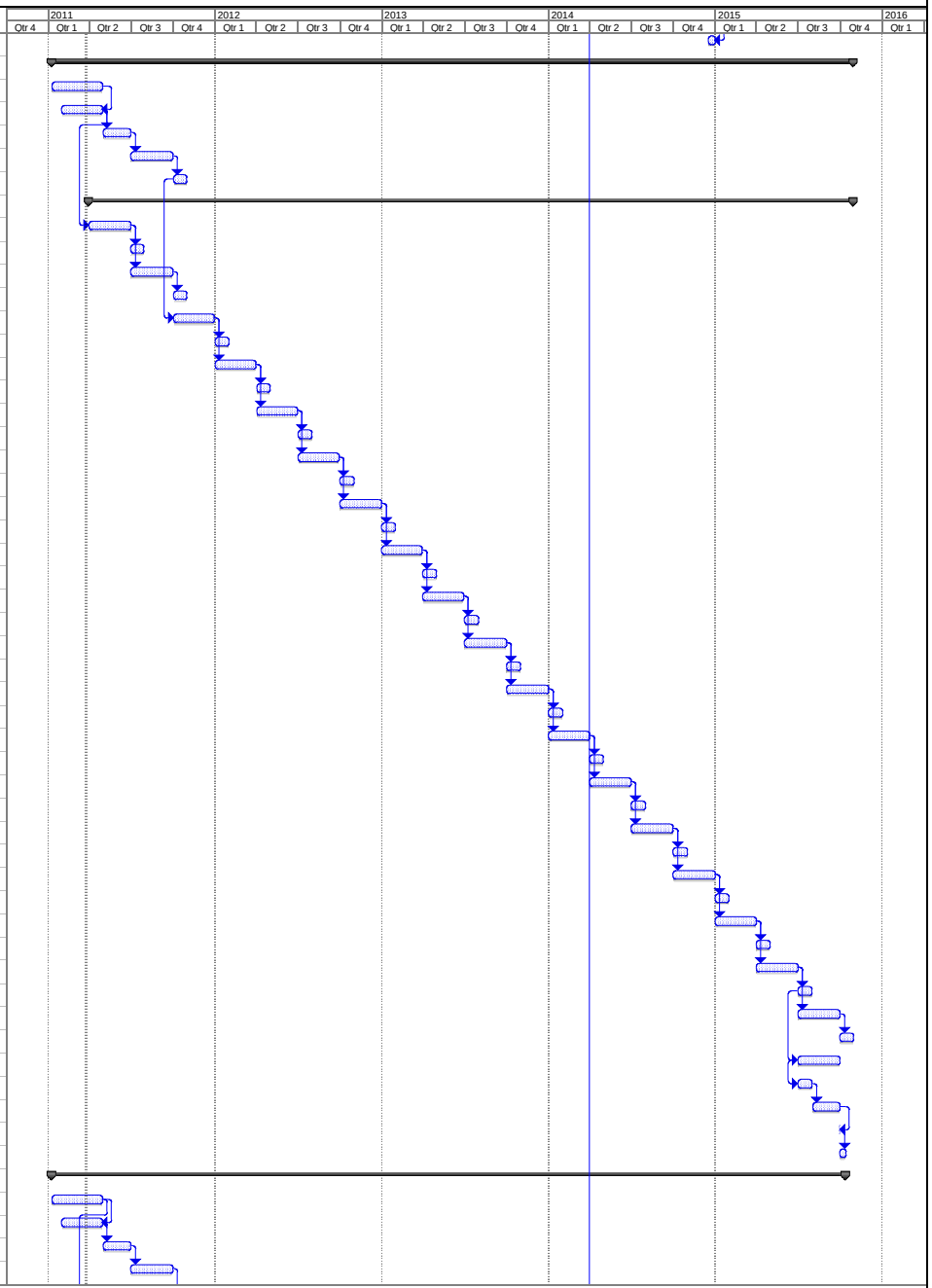
ID	WBS	WBS Codes	Task Name	Duration	Start	Finish	Predecessors
703	39.6.3	SS020.6.3	SS020 - Conduct Monitoring, Prep and Submit CY11 Q3 LTO/LTM Report	66 days	Fri 7/1/11	Fri 9/30/11	701
704	39.6.4	SS020.6.4	SS020 - AFCEE Review and Acceptance of CY11 Q3 LTO/LTM Report	21 days	Mon 10/3/11	Mon 10/31/11	703
705	39.6.5	SS020.6.5	SS020 - Conduct Monitoring, Prep and Submit CY11 Q4 LTO/LTM Report	65 days	Mon 10/3/11	Fri 12/30/11	699SS
706	39.6.6	SS020.6.6	SS020 - AFCEE Review and Acceptance of CY11 Q4 LTO/LTM Report	22 days	Mon 1/2/12	Tue 1/31/12	705
707	39.6.7	SS020.6.7	SS020 - Conduct Monitoring, Prep and Submit CY12 Q1 LTO/LTM Report	65 days	Mon 1/2/12	Fri 3/30/12	705
708	39.6.8	SS020.6.8	SS020 - AFCEE Review and Acceptance of CY12 Q1 LTO/LTM Report	21 days	Mon 4/2/12	Mon 4/30/12	707
709	39.6.9	SS020.6.9	SS020 - Conduct Monitoring, Prep and Submit CY12 Q2 LTO/LTM Report	65 days	Mon 4/2/12	Fri 6/29/12	707
710	39.6.10	SS020.6.10	SS020 - AFCEE Review and Acceptance of CY12 Q2 LTO/LTM Report	22 days	Mon 7/2/12	Tue 7/31/12	709
711	39.6.11	SS020.6.11	SS020 - Conduct Monitoring, Prep and Submit CY12 Q3 LTO/LTM Report	65 days	Mon 7/2/12	Fri 9/28/12	709
712	39.6.12	SS020.6.12	SS020 - AFCEE Review and Acceptance of CY12 Q3 LTO/LTM Report	23 days	Mon 10/1/12	Wed 10/31/12	711
713	39.6.13	SS020.6.13	SS020 - Conduct Monitoring, Prep and Submit CY12 Q4 LTO/LTM Report	66 days	Mon 10/1/12	Mon 12/31/12	711
714	39.6.14	SS020.6.14	SS020 - AFCEE Review and Acceptance of CY12 Q4 LTO/LTM Report	22 days	Tue 1/1/13	Wed 1/30/13	713
715	39.6.15	SS020.6.15	SS020 - Conduct Monitoring, Prep and Submit CY13 Q1 LTO/LTM Report	64 days	Tue 1/1/13	Fri 3/29/13	713
716	39.6.16	SS020.6.16	SS020 - AFCEE Review and Acceptance of CY13 Q1 LTO/LTM Report	22 days	Mon 4/1/13	Tue 4/30/13	715
717	39.6.17	SS020.6.17	SS020 - Conduct Monitoring, Prep and Submit CY13 Q2 LTO/LTM Report	65 days	Mon 4/1/13	Fri 6/28/13	715
718	39.6.18	SS020.6.18	SS020 - AFCEE Review and Acceptance of CY13 Q2 LTO/LTM Report	23 days	Mon 7/1/13	Wed 7/31/13	717
719	39.6.19	SS020.6.19	SS020 - Conduct Monitoring, Prep and Submit CY13 Q3 LTO/LTM Report	66 days	Mon 7/1/13	Mon 9/30/13	717
720	39.6.20	SS020.6.20	SS020 - AFCEE Review and Acceptance of CY13 Q3 LTO/LTM Report	23 days	Tue 10/1/13	Thu 10/31/13	719
721	39.6.21	SS020.6.21	SS020 - Conduct Monitoring, Prep and Submit CY13 Q4 LTO/LTM Report	66 days	Tue 10/1/13	Tue 12/31/13	719
722	39.6.22	SS020.6.22	SS020 - AFCEE Review and Acceptance of CY13 Q4 LTO/LTM Report	23 days	Wed 1/1/14	Fri 1/31/14	721
723	39.6.23	SS020.6.23	SS020 - Conduct Monitoring, Prep and Submit CY14 Q1 LTO/LTM Report	64 days	Wed 1/1/14	Mon 3/31/14	721
724	39.6.24	SS020.6.24	SS020 - AFCEE Review and Acceptance of CY14 Q1 LTO/LTM Report	22 days	Tue 4/1/14	Wed 4/30/14	723
725	39.6.25	SS020.6.25	SS020 - Conduct Monitoring, Prep and Submit CY14 Q2 LTO/LTM Report	65 days	Tue 4/1/14	Mon 6/30/14	723
726	39.6.26	SS020.6.26	SS020 - AFCEE Review and Acceptance of CY14 Q2 LTO/LTM Report	23 days	Tue 7/1/14	Thu 7/31/14	725
727	39.6.27	SS020.6.27	SS020 - Conduct Monitoring, Prep and Submit CY14 Q3 LTO/LTM Report	66 days	Tue 7/1/14	Tue 9/30/14	725
728	39.6.28	SS020.6.28	SS020 - AFCEE Review and Acceptance of CY14 Q3 LTO/LTM Report	23 days	Wed 10/1/14	Fri 10/31/14	727
729	39.6.29	SS020.6.29	SS020 - Conduct Monitoring, Prep and Submit CY14 Q4 LTO/LTM Report	66 days	Wed 10/1/14	Wed 12/31/14	727
730	39.6.30	SS020.6.30	SS020 - AFCEE Review and Acceptance of CY14 Q4 LTO/LTM Report	22 days	Thu 1/1/15	Fri 1/30/15	729
731	39.7	SS020.7	SS020 - Achieve Site Closure	63 days	Thu 1/1/15	Mon 3/30/15	729
732	39.8	SS020.8	SS020 - Prepare and Submit Closure Report	23 days	Mon 12/1/14	Wed 12/31/14	729FS-23 days
733	39.9	SS020.9	SS020 - AFCEE Review and Acceptance of Closure Report	64 days	Thu 1/1/15	Tue 3/31/15	732
734	39.10	SS020.10	SS020 - NYSDEC Closure of Spill Number	1 day	Tue 3/31/15	Tue 3/31/15	733FF
735	39.11	SS020.11	SS020 - Wells Decommissioned	10 days	Wed 4/1/15	Tue 4/14/15	734
736	**40**	**SS063**	**SS063 - Apron 1 (Closure Unrestricted Reuse)**	**777 days**	**Mon 1/10/11**	**Tue 12/31/13**	
737	40.1	SS063.1	SS063 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	Mon 1/10/11	Fri 4/29/11	
738	40.2	SS063.2	SS063 - Prepare and Submit Design/plan remedy optimization	65 days	Mon 5/2/11	Fri 7/29/11	737
739	40.3	SS063.3	SS063 - AFCEE Review and Acceptance of Design/Plan Remedy Optimization	21 days	Mon 8/1/11	Mon 8/29/11	738
740	40.4	SS063.4	SS063 - Implement Remedy Optimization Systems and Submit Report	66 days	Mon 8/1/11	Mon 10/31/11	738
741	40.5	SS063.5	SS063 - AFCEE Review and Acceptance of Optimization System and Report	21 days	Tue 11/1/11	Tue 11/29/11	740
742	**40.6**	**SS063.6**	**SS063 - Conduct Monitoring, Operations & Maintenance**	**675 days**	**Fri 4/1/11**	**Thu 10/31/13**	
743	40.6.1	SS063.6.1	SS063 - Conduct Monitoring, Prep and Submit CY11 Q2 LTO/LTM Report	65 days	Fri 4/1/11	Thu 6/30/11	737FS-21 days
744	40.6.2	SS063.6.2	SS063 - AFCEE Review and Acceptance of CY11 Q2 LTO/LTM Report	21 days	Fri 7/1/11	Fri 7/29/11	743
745	40.6.3	SS063.6.3	SS063 - Conduct Monitoring, Prep and Submit CY11 Q3 LTO/LTM Report	66 days	Fri 7/1/11	Fri 9/30/11	743
746	40.6.4	SS063.6.4	SS063 - AFCEE Review and Acceptance of CY11 Q3 LTO/LTM Report	21 days	Mon 10/3/11	Mon 10/31/11	745
747	40.6.5	SS063.6.5	SS063 - Conduct Monitoring, Prep and Submit CY11 Q4 LTO/LTM Report	65 days	Mon 10/3/11	Fri 12/30/11	745
748	40.6.6	SS063.6.6	SS063 - AFCEE Review and Acceptance of CY11 Q4 LTO/LTM Report	22 days	Mon 1/2/12	Tue 1/31/12	747
749	40.6.7	SS063.6.7	SS063 - Conduct Monitoring, Prep and Submit CY12 Q1 LTO/LTM Report	65 days	Mon 1/2/12	Fri 3/30/12	747
750	40.6.8	SS063.6.8	SS063 - AFCEE Review and Acceptance of CY12 Q1 LTO/LTM Report	21 days	Mon 4/2/12	Mon 4/30/12	749
751	40.6.9	SS063.6.9	SS063 - Conduct Monitoring, Prep and Submit CY12 Q2 LTO/LTM Report	65 days	Mon 4/2/12	Fri 6/29/12	749
752	40.6.10	SS063.6.10	SS063 - AFCEE Review and Acceptance of CY12 Q2 LTO/LTM Report	22 days	Mon 7/2/12	Tue 7/31/12	751
753	40.6.11	SS063.6.11	SS063 - Conduct Monitoring, Prep and Submit CY12 Q3 LTO/LTM Report	65 days	Mon 7/2/12	Fri 9/28/12	751
754	40.6.12	SS063.6.12	SS063 - AFCEE Review and Acceptance of CY12 Q3 LTO/LTM Report	23 days	Mon 10/1/12	Wed 10/31/12	753
755	40.6.13	SS063.6.13	SS063 - Conduct Monitoring, Prep and Submit CY12 Q4 LTO/LTM Report	66 days	Mon 10/1/12	Mon 12/31/12	753
756	40.6.14	SS063.6.14	SS063 - AFCEE Review and Acceptance of CY12 Q4 LTO/LTM Report	22 days	Tue 1/1/13	Wed 1/30/13	755



ID	WBS	WBS Codes	Task Name	Duration	Start	Finish	Predecessors
757	40.6.15	SS063.6.15	SS063 - Conduct Monitoring, Prep and Submit CY13 Q1 LTO/LTM Report	64 days	Tue 1/1/13	Fri 3/29/13	755
758	40.6.16	SS063.6.16	SS063 - AFCEE Review and Acceptance of CY13 Q1 LTO/LTM Report	22 days	Mon 4/1/13	Tue 4/30/13	757
759	40.6.17	SS063.6.17	SS063 - Conduct Monitoring, Prep and Submit CY13 Q2 LTO/LTM Report	65 days	Mon 4/1/13	Fri 6/28/13	757
760	40.6.18	SS063.6.18	SS063 - AFCEE Review and Acceptance of CY13 Q2 LTO/LTM Report	23 days	Mon 7/1/13	Wed 7/31/13	759
761	40.6.19	SS063.6.19	SS063 - Conduct Monitoring, Prep and Submit CY13 Q3 LTO/LTM Report	66 days	Mon 7/1/13	Mon 9/30/13	759
762	40.6.20	SS063.6.20	SS063 - AFCEE Review and Acceptance of CY13 Q3 LTO/LTM Report	23 days	Tue 10/1/13	Thu 10/31/13	761
763	40.7	SS063.7	SS063 - Achieve Site Closure	66 days	Tue 10/1/13	Tue 12/31/13	762SS
764	40.8	SS063.8	SS063 - Prepare and Submit Closure Report	21 days	Mon 9/2/13	Mon 9/30/13	761FS-21 days
765	40.9	SS063.9	SS063 - AFCEE Review and Acceptance of Closure Report	66 days	Tue 10/1/13	Tue 12/31/13	764
766	40.10	SS063.10	SS063 - NYSDEC Closure of Spill Number	1 day	Tue 12/31/13	Tue 12/31/13	765FF
767	40.11	SS063.11	SS063 - Wells Decommissioned	11 days	Tue 12/17/13	Tue 12/31/13	766FF
768	**41**	**ST037**	**ST037 - Bldg 771, Pumphouse 5 (Closure Unrestricted Use)**	**124 days**	**Mon 1/10/11**	**Thu 6/30/11**	
769	41.1	ST037.1	ST037 - Prepare and Submit Closure Report	90 days	Mon 1/10/11	Fri 5/13/11	
770	41.2	ST037.2	ST037 - AFCEE Review and Acceptance of Closure Report	34 days	Mon 5/16/11	Thu 6/30/11	769
771	41.3	ST037.3	ST037 - NYSDEC Closure of Spill Number	1 day	Thu 6/30/11	Thu 6/30/11	770FF
772	**42**	**SS064**	**SS064 - Apron 2 (Closure Unrestricted Reuse)**	**1040 days**	**Mon 1/10/11**	**Fri 1/2/15**	
773	42.1	SS064.1	SS064 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	Mon 1/10/11	Fri 4/29/11	
774	42.2	SS064.2	SS064 - Prepare and Submit Design/plan remedy optimization	65 days	Mon 1/31/11	Fri 4/29/11	773FF
775	42.3	SS064.3	SS064 - AFCEE Review and Acceptance of Design/Plan Remedy Optimization	44 days	Mon 5/2/11	Thu 6/30/11	774
776	42.4	SS064.4	SS064 - Implement Remedy Optimization Systems and Submit Report	66 days	Fri 7/1/11	Fri 9/30/11	775
777	42.5	SS064.5	SS064 - AFCEE Review and Acceptance of Optimization System and Report	21 days	Mon 10/3/11	Mon 10/31/11	776
778	**42.6**	**SS064.6**	**SS064 - Conduct Monitoring, Operations & Maintenance**	**936 days**	**Fri 4/1/11**	**Fri 10/31/14**	
779	42.6.1	SS064.6.1	SS064 - Conduct Monitoring, Prep and Submit CY11 Q2 LTO/LTM Report	65 days	Fri 4/1/11	Thu 6/30/11	774FS-21 days
780	42.6.2	SS064.6.2	SS064 - AFCEE Review and Acceptance of CY11 Q2 LTO/LTM Report	21 days	Fri 7/1/11	Fri 7/29/11	779
781	42.6.3	SS064.6.3	SS064 - Conduct Monitoring, Prep and Submit CY11 Q3 LTO/LTM Report	66 days	Fri 7/1/11	Fri 9/30/11	779
782	42.6.4	SS064.6.4	SS064 - AFCEE Review and Acceptance of CY11 Q3 LTO/LTM Report	21 days	Mon 10/3/11	Mon 10/31/11	781
783	42.6.5	SS064.6.5	SS064 - Conduct Monitoring, Prep and Submit CY11 Q4 LTO/LTM Report	65 days	Mon 10/3/11	Fri 12/30/11	777SS
784	42.6.6	SS064.6.6	SS064 - AFCEE Review and Acceptance of CY11 Q4 LTO/LTM Report	22 days	Mon 1/2/12	Tue 1/31/12	783
785	42.6.7	SS064.6.7	SS064 - Conduct Monitoring, Prep and Submit CY12 Q1 LTO/LTM Report	65 days	Mon 1/2/12	Fri 3/30/12	783
786	42.6.8	SS064.6.8	SS064 - AFCEE Review and Acceptance of CY12 Q1 LTO/LTM Report	21 days	Mon 4/2/12	Mon 4/30/12	785
787	42.6.9	SS064.6.9	SS064 - Conduct Monitoring, Prep and Submit CY12 Q2 LTO/LTM Report	65 days	Mon 4/2/12	Fri 6/29/12	785
788	42.6.10	SS064.6.10	SS064 - AFCEE Review and Acceptance of CY12 Q2 LTO/LTM Report	22 days	Mon 7/2/12	Tue 7/31/12	787
789	42.6.11	SS064.6.11	SS064 - Conduct Monitoring, Prep and Submit CY12 Q3 LTO/LTM Report	65 days	Mon 7/2/12	Fri 9/28/12	787
790	42.6.12	SS064.6.12	SS064 - AFCEE Review and Acceptance of CY12 Q3 LTO/LTM Report	23 days	Mon 10/1/12	Wed 10/31/12	789
791	42.6.13	SS064.6.13	SS064 - Conduct Monitoring, Prep and Submit CY12 Q4 LTO/LTM Report	66 days	Mon 10/1/12	Mon 12/31/12	789
792	42.6.14	SS064.6.14	SS064 - AFCEE Review and Acceptance of CY12 Q4 LTO/LTM Report	22 days	Tue 1/1/13	Wed 1/30/13	791
793	42.6.15	SS064.6.15	SS064 - Conduct Monitoring, Prep and Submit CY13 Q1 LTO/LTM Report	64 days	Tue 1/1/13	Fri 3/29/13	791
794	42.6.16	SS064.6.16	SS064 - AFCEE Review and Acceptance of CY13 Q1 LTO/LTM Report	22 days	Mon 4/1/13	Tue 4/30/13	793
795	42.6.17	SS064.6.17	SS064 - Conduct Monitoring, Prep and Submit CY13 Q2 LTO/LTM Report	65 days	Mon 4/1/13	Fri 6/28/13	793
796	42.6.18	SS064.6.18	SS064 - AFCEE Review and Acceptance of CY13 Q2 LTO/LTM Report	23 days	Mon 7/1/13	Wed 7/31/13	795
797	42.6.19	SS064.6.19	SS064 - Conduct Monitoring, Prep and Submit CY13 Q3 LTO/LTM Report	66 days	Mon 7/1/13	Mon 9/30/13	795
798	42.6.20	SS064.6.20	SS064 - AFCEE Review and Acceptance of CY13 Q3 LTO/LTM Report	23 days	Tue 10/1/13	Thu 10/31/13	797
799	42.6.21	SS064.6.21	SS064 - Conduct Monitoring, Prep and Submit CY13 Q4 LTO/LTM Report	66 days	Tue 10/1/13	Tue 12/31/13	797
800	42.6.22	SS064.6.22	SS064 - AFCEE Review and Acceptance of CY13 Q4 LTO/LTM Report	23 days	Wed 1/1/14	Fri 1/31/14	799
801	42.6.23	SS064.6.23	SS064 - Conduct Monitoring, Prep and Submit CY14 Q1 LTO/LTM Report	64 days	Wed 1/1/14	Mon 3/31/14	799
802	42.6.24	SS064.6.24	SS064 - AFCEE Review and Acceptance of CY14 Q1 LTO/LTM Report	22 days	Tue 4/1/14	Wed 4/30/14	801
803	42.6.25	SS064.6.25	SS064 - Conduct Monitoring, Prep and Submit CY14 Q2 LTO/LTM Report	65 days	Tue 4/1/14	Mon 6/30/14	801
804	42.6.26	SS064.6.26	SS064 - AFCEE Review and Acceptance of CY14 Q2 LTO/LTM Report	23 days	Tue 7/1/14	Thu 7/31/14	803
805	42.6.27	SS064.6.27	SS064 - Conduct Monitoring, Prep and Submit CY14 Q3 LTO/LTM Report	66 days	Tue 7/1/14	Tue 9/30/14	803
806	42.6.28	SS064.6.28	SS064 - AFCEE Review and Acceptance of CY14 Q3 LTO/LTM Report	23 days	Wed 10/1/14	Fri 10/31/14	805
807	42.7	SS064.7	SS064 - Achieve Site Closure	68 days	Wed 10/1/14	Fri 1/2/15	806SS
808	42.8	SS064.8	SS064 - Prepare and Submit Closure Report	22 days	Mon 9/1/14	Tue 9/30/14	805FS-22 days
809	42.9	SS064.9	SS064 - AFCEE Review and Acceptance of Closure Report	66 days	Wed 10/1/14	Wed 12/31/14	808
810	42.10	SS064.10	SS064 - NYSDEC Closure of Spill Number	1 day	Wed 12/31/14	Wed 12/31/14	809FF



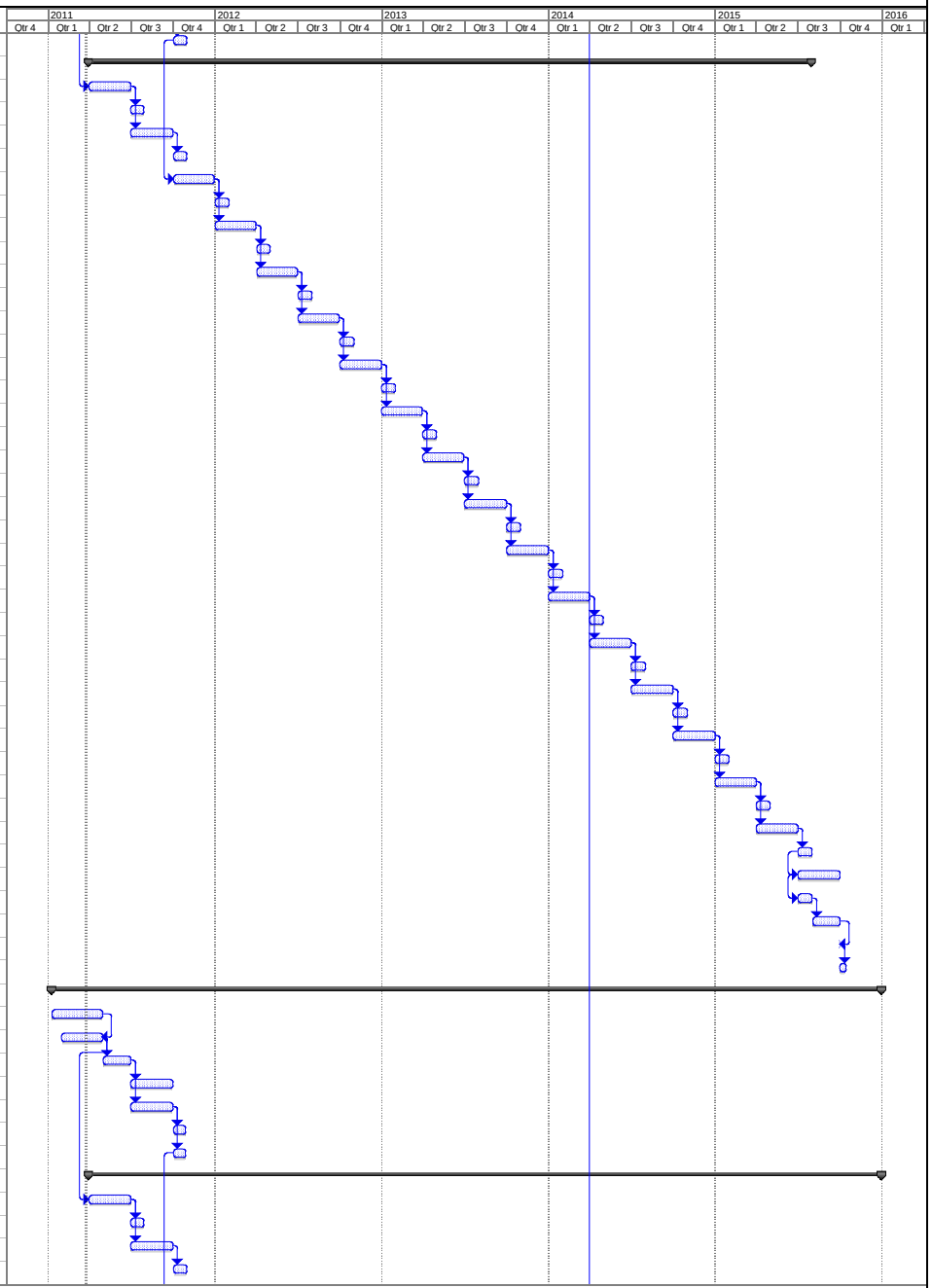
ID	WBS	WBS Codes	Task Name	Duration	Start	Finish	Predecessors
811	42.11	SS064.11	SS064 - Wells Decommissioned	11 days	Wed 12/17/14	Wed 12/31/14	810FF
812	**43**	**SS065**	**SS065 - BLDG 15 (Closure Unrestricted Reuse)**	**1255 days**	**Mon 1/10/11**	**Fri 10/30/15**	
813	43.1	SS065.1	SS065 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	Mon 1/10/11	Fri 4/29/11	
814	43.2	SS065.2	SS065 - Prepare and Submit Design/plan remedy optimization	65 days	Mon 1/31/11	Fri 4/29/11	813FF
815	43.3	SS065.3	SS065 - AFCEE Review and Acceptance of Design/Plan Remedy Optimization	44 days	Mon 5/2/11	Thu 6/30/11	814
816	43.4	SS065.4	SS065 - Implement Remedy Optimization Systems and Submit Report	66 days	Fri 7/1/11	Fri 9/30/11	815
817	43.5	SS065.5	SS065 - AFCEE Review and Acceptance of Optimization System and Report	21 days	Mon 10/3/11	Mon 10/31/11	816
818	**43.6**	**SS065.6**	**SS065 - Conduct Monitoring, Operations & Maintenance**	**1196 days**	**Fri 4/1/11**	**Fri 10/30/15**	
819	43.6.1	SS065.6.1	SS065 - Conduct Monitoring, Prep and Submit CY11 Q2 LTO/LTM Report	65 days	Fri 4/1/11	Thu 6/30/11	814FS-21 days
820	43.6.2	SS065.6.2	SS065 - AFCEE Review and Acceptance of CY11 Q2 LTO/LTM Report	21 days	Fri 7/1/11	Fri 7/29/11	819
821	43.6.3	SS065.6.3	SS065 - Conduct Monitoring, Prep and Submit CY11 Q3 LTO/LTM Report	66 days	Fri 7/1/11	Fri 9/30/11	819
822	43.6.4	SS065.6.4	SS065 - AFCEE Review and Acceptance of CY11 Q3 LTO/LTM Report	21 days	Mon 10/3/11	Mon 10/31/11	821
823	43.6.5	SS065.6.5	SS065 - Conduct Monitoring, Prep and Submit CY11 Q4 LTO/LTM Report	65 days	Mon 10/3/11	Fri 12/30/11	817SS
824	43.6.6	SS065.6.6	SS065 - AFCEE Review and Acceptance of CY11 Q4 LTO/LTM Report	22 days	Mon 1/2/12	Tue 1/31/12	823
825	43.6.7	SS065.6.7	SS065 - Conduct Monitoring, Prep and Submit CY12 Q1 LTO/LTM Report	65 days	Mon 1/2/12	Fri 3/30/12	823
826	43.6.8	SS065.6.8	SS065 - AFCEE Review and Acceptance of CY12 Q1 LTO/LTM Report	21 days	Mon 4/2/12	Mon 4/30/12	825
827	43.6.9	SS065.6.9	SS065 - Conduct Monitoring, Prep and Submit CY12 Q2 LTO/LTM Report	65 days	Mon 4/2/12	Fri 6/29/12	825
828	43.6.10	SS065.6.10	SS065 - AFCEE Review and Acceptance of CY12 Q2 LTO/LTM Report	22 days	Mon 7/2/12	Tue 7/31/12	827
829	43.6.11	SS065.6.11	SS065 - Conduct Monitoring, Prep and Submit CY12 Q3 LTO/LTM Report	65 days	Mon 7/2/12	Fri 9/28/12	827
830	43.6.12	SS065.6.12	SS065 - AFCEE Review and Acceptance of CY12 Q3 LTO/LTM Report	23 days	Mon 10/1/12	Wed 10/31/12	829
831	43.6.13	SS065.6.13	SS065 - Conduct Monitoring, Prep and Submit CY12 Q4 LTO/LTM Report	66 days	Mon 10/1/12	Mon 12/31/12	829
832	43.6.14	SS065.6.14	SS065 - AFCEE Review and Acceptance of CY12 Q4 LTO/LTM Report	22 days	Tue 1/1/13	Wed 1/30/13	831
833	43.6.15	SS065.6.15	SS065 - Conduct Monitoring, Prep and Submit CY13 Q1 LTO/LTM Report	64 days	Tue 1/1/13	Fri 3/29/13	831
834	43.6.16	SS065.6.16	SS065 - AFCEE Review and Acceptance of CY13 Q1 LTO/LTM Report	22 days	Mon 4/1/13	Tue 4/30/13	833
835	43.6.17	SS065.6.17	SS065 - Conduct Monitoring, Prep and Submit CY13 Q2 LTO/LTM Report	65 days	Mon 4/1/13	Fri 6/28/13	833
836	43.6.18	SS065.6.18	SS065 - AFCEE Review and Acceptance of CY13 Q2 LTO/LTM Report	23 days	Mon 7/1/13	Wed 7/31/13	835
837	43.6.19	SS065.6.19	SS065 - Conduct Monitoring, Prep and Submit CY13 Q3 LTO/LTM Report	66 days	Mon 7/1/13	Mon 9/30/13	835
838	43.6.20	SS065.6.20	SS065 - AFCEE Review and Acceptance of CY13 Q3 LTO/LTM Report	23 days	Tue 10/1/13	Thu 10/31/13	837
839	43.6.21	SS065.6.21	SS065 - Conduct Monitoring, Prep and Submit CY13 Q4 LTO/LTM Report	66 days	Tue 10/1/13	Tue 12/31/13	837
840	43.6.22	SS065.6.22	SS065 - AFCEE Review and Acceptance of CY13 Q4 LTO/LTM Report	23 days	Wed 1/1/14	Fri 1/31/14	839
841	43.6.23	SS065.6.23	SS065 - Conduct Monitoring, Prep and Submit CY14 Q1 LTO/LTM Report	64 days	Wed 1/1/14	Mon 3/31/14	839
842	43.6.24	SS065.6.24	SS065 - AFCEE Review and Acceptance of CY14 Q1 LTO/LTM Report	22 days	Tue 4/1/14	Wed 4/30/14	841
843	43.6.25	SS065.6.25	SS065 - Conduct Monitoring, Prep and Submit CY14 Q2 LTO/LTM Report	65 days	Tue 4/1/14	Mon 6/30/14	841
844	43.6.26	SS065.6.26	SS065 - AFCEE Review and Acceptance of CY14 Q2 LTO/LTM Report	23 days	Tue 7/1/14	Thu 7/31/14	843
845	43.6.27	SS065.6.27	SS065 - Conduct Monitoring, Prep and Submit CY14 Q3 LTO/LTM Report	66 days	Tue 7/1/14	Tue 9/30/14	843
846	43.6.28	SS065.6.28	SS065 - AFCEE Review and Acceptance of CY14 Q3 LTO/LTM Report	23 days	Wed 10/1/14	Fri 10/31/14	845
847	43.6.29	SS065.6.29	SS065 - Conduct Monitoring, Prep and Submit CY14 Q4 LTO/LTM Report	66 days	Wed 10/1/14	Wed 12/31/14	845
848	43.6.30	SS065.6.30	SS065 - AFCEE Review and Acceptance of CY14 Q4 LTO/LTM Report	22 days	Thu 1/1/15	Fri 1/30/15	847
849	43.6.31	SS065.6.31	SS065 - Conduct Monitoring, Prep and Submit CY15 Q1 LTO/LTM Report	64 days	Thu 1/1/15	Tue 3/31/15	847
850	43.6.32	SS065.6.32	SS065 - AFCEE Review and Acceptance of CY15 Q1 LTO/LTM Report	22 days	Wed 4/1/15	Thu 4/30/15	849
851	43.6.33	SS065.6.33	SS065 - Conduct Monitoring, Prep and Submit CY15 Q2 LTO/LTM Report	65 days	Wed 4/1/15	Tue 6/30/15	849
852	43.6.34	SS065.6.34	SS065 - AFCEE Review and Acceptance of CY15 Q2 LTO/LTM Report	23 days	Wed 7/1/15	Fri 7/31/15	851
853	43.6.35	SS065.6.35	SS065 - Conduct Monitoring, Prep and Submit CY15 Q3 LTO/LTM Report	66 days	Wed 7/1/15	Wed 9/30/15	851
854	43.6.36	SS065.6.36	SS065 - AFCEE Review and Acceptance of CY15 Q3 LTO/LTM Report	22 days	Thu 10/1/15	Fri 10/30/15	853
855	43.7	SS065.7	SS065 - Achieve Site Closure	66 days	Wed 7/1/15	Wed 9/30/15	852SS
856	43.8	SS065.8	SS065 - Prepare and Submit Closure Report	23 days	Wed 7/1/15	Fri 7/31/15	855SS
857	43.9	SS065.9	SS065 - AFCEE Review and Acceptance of Closure Report	43 days	Mon 8/3/15	Wed 9/30/15	856
858	43.10	SS065.10	SS065 - NYSDEC Closure of Spill Number	1 day	Wed 9/30/15	Wed 9/30/15	857FF
859	43.11	SS065.11	SS065 - Wells Decommissioned	9 days	Thu 10/1/15	Tue 10/13/15	858
860	**44**	**SS066**	**SS066 - BLDG 786 (Closure Unrestricted Reuse)**	**1242 days**	**Mon 1/10/11**	**Tue 10/13/15**	
861	44.1	SS066.1	SS066 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	Mon 1/10/11	Fri 4/29/11	
862	44.2	SS066.2	SS066 - Prepare and Submit Design/plan remedy optimization	65 days	Mon 1/31/11	Fri 4/29/11	861FF
863	44.3	SS066.3	SS066 - AFCEE Review and Acceptance of Design/Plan Remedy Optimization	44 days	Mon 5/2/11	Thu 6/30/11	862
864	44.4	SS066.4	SS066 - Implement Remedy Optimization Systems and Submit Report	66 days	Fri 7/1/11	Fri 9/30/11	863



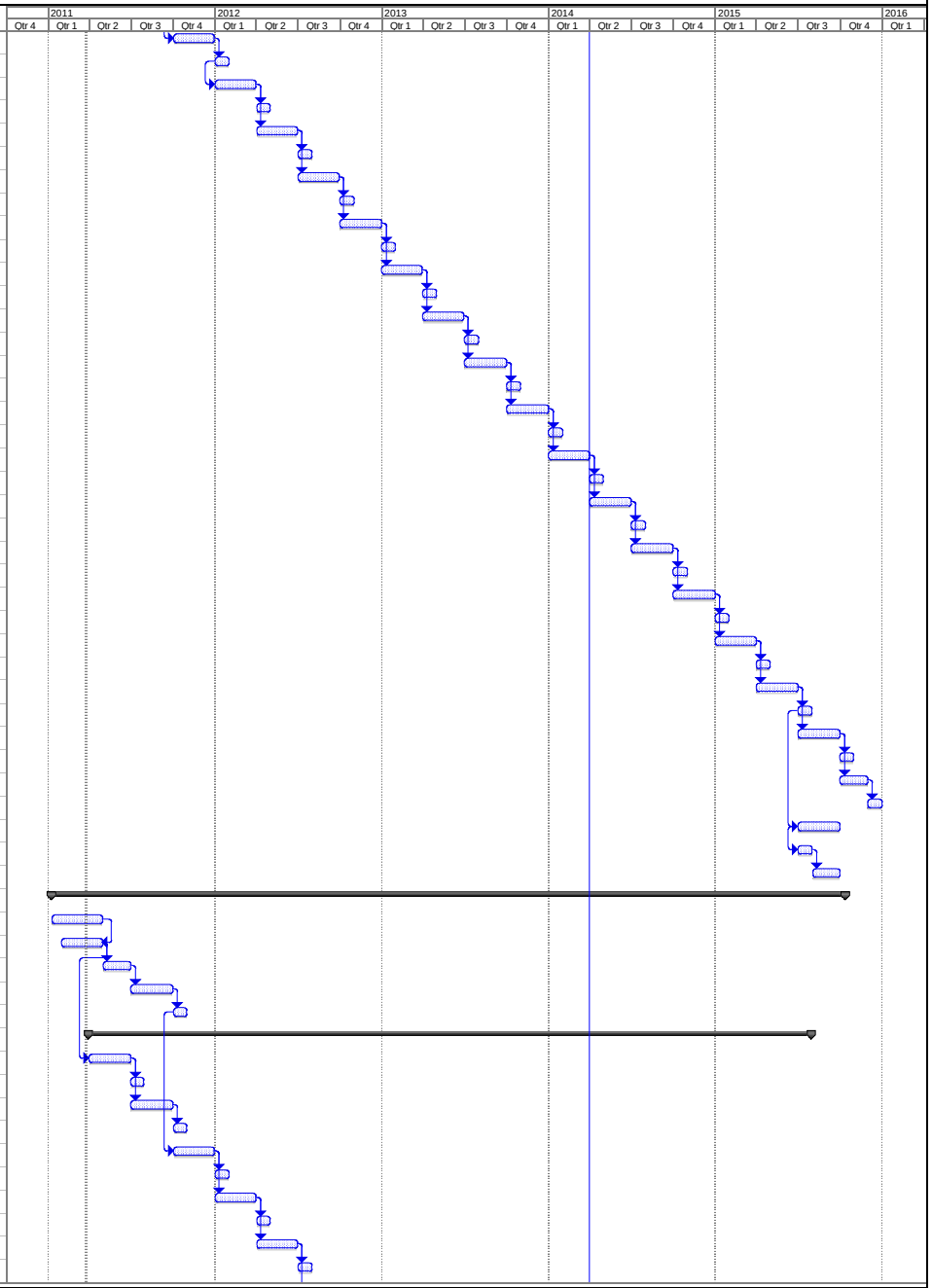
Project: Project1
Date: Thu 3/24/11

Task | Split | Progress | Milestone | Summary | Project Summary | External Tasks | External MileTask | Split

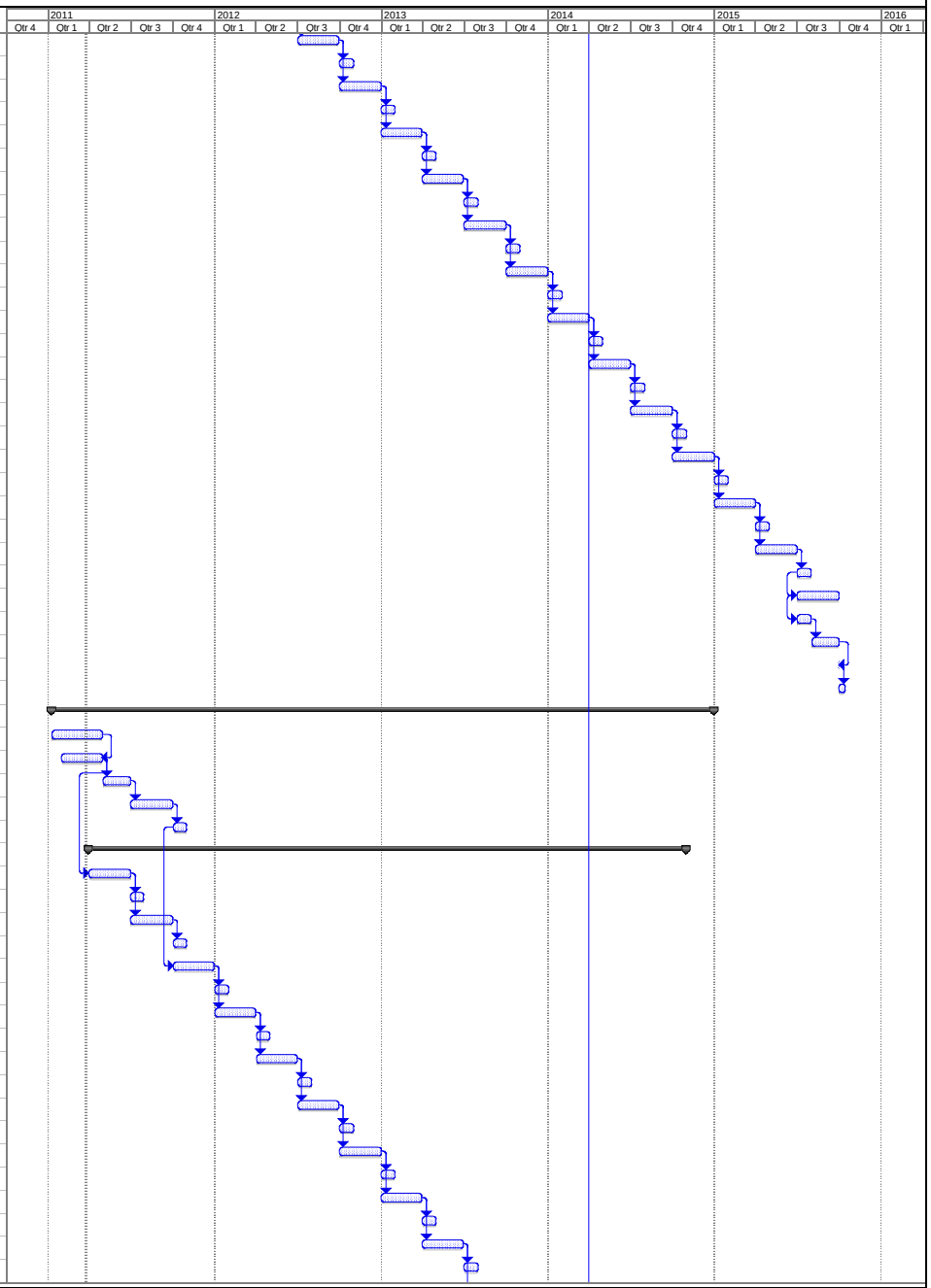
ID	WBS	WBS Codes	Task Name	Duration	Start	Finish	Predecessors
865	44.5	SS066.5	SS066 - AFCEE Review and Acceptance of Optimization System and Report	21 days	Mon 10/3/11	Mon 10/31/11	864
866	**44.6**	**SS066.6**	**SS066 - Conduct Monitoring, Operations & Maintenance**	**1131 days**	**Fri 4/1/11**	**Fri 7/31/15**	
867	44.6.1	SS066.6.1	SS066 - Conduct Monitoring, Prep and Submit CY11 Q2 LTO/LTM Report	65 days	Fri 4/1/11	Thu 6/30/11	861FS-21 days
868	44.6.2	SS066.6.2	SS066 - AFCEE Review and Acceptance of CY11 Q2 LTO/LTM Report	21 days	Fri 7/1/11	Fri 7/29/11	867
869	44.6.3	SS066.6.3	SS066 - Conduct Monitoring, Prep and Submit CY11 Q3 LTO/LTM Report	66 days	Fri 7/1/11	Fri 9/30/11	867
870	44.6.4	SS066.6.4	SS066 - AFCEE Review and Acceptance of CY11 Q3 LTO/LTM Report	21 days	Mon 10/3/11	Mon 10/31/11	869
871	44.6.5	SS066.6.5	SS066 - Conduct Monitoring, Prep and Submit CY11 Q4 LTO/LTM Report	65 days	Mon 10/3/11	Fri 12/30/11	865SS
872	44.6.6	SS066.6.6	SS066 - AFCEE Review and Acceptance of CY11 Q4 LTO/LTM Report	22 days	Mon 1/2/12	Tue 1/31/12	871
873	44.6.7	SS066.6.7	SS066 - Conduct Monitoring, Prep and Submit CY12 Q1 LTO/LTM Report	65 days	Mon 1/2/12	Fri 3/30/12	871
874	44.6.8	SS066.6.8	SS066 - AFCEE Review and Acceptance of CY12 Q1 LTO/LTM Report	21 days	Mon 4/2/12	Mon 4/30/12	873
875	44.6.9	SS066.6.9	SS066 - Conduct Monitoring, Prep and Submit CY12 Q2 LTO/LTM Report	65 days	Mon 4/2/12	Fri 6/29/12	873
876	44.6.10	SS066.6.10	SS066 - AFCEE Review and Acceptance of CY12 Q2 LTO/LTM Report	22 days	Mon 7/2/12	Tue 7/31/12	875
877	44.6.11	SS066.6.11	SS066 - Conduct Monitoring, Prep and Submit CY12 Q3 LTO/LTM Report	65 days	Mon 7/2/12	Fri 9/28/12	875
878	44.6.12	SS066.6.12	SS066 - AFCEE Review and Acceptance of CY12 Q3 LTO/LTM Report	23 days	Mon 10/1/12	Wed 10/31/12	877
879	44.6.13	SS066.6.13	SS066 - Conduct Monitoring, Prep and Submit CY12 Q4 LTO/LTM Report	66 days	Mon 10/1/12	Mon 12/31/12	877
880	44.6.14	SS066.6.14	SS066 - AFCEE Review and Acceptance of CY12 Q4 LTO/LTM Report	22 days	Tue 1/1/13	Wed 1/30/13	879
881	44.6.15	SS066.6.15	SS066 - Conduct Monitoring, Prep and Submit CY13 Q1 LTO/LTM Report	64 days	Tue 1/1/13	Fri 3/29/13	879
882	44.6.16	SS066.6.16	SS066 - AFCEE Review and Acceptance of CY13 Q1 LTO/LTM Report	22 days	Mon 4/1/13	Tue 4/30/13	881
883	44.6.17	SS066.6.17	SS066 - Conduct Monitoring, Prep and Submit CY13 Q2 LTO/LTM Report	65 days	Mon 4/1/13	Fri 6/28/13	881
884	44.6.18	SS066.6.18	SS066 - AFCEE Review and Acceptance of CY13 Q2 LTO/LTM Report	23 days	Mon 7/1/13	Wed 7/31/13	883
885	44.6.19	SS066.6.19	SS066 - Conduct Monitoring, Prep and Submit CY13 Q3 LTO/LTM Report	66 days	Mon 7/1/13	Mon 9/30/13	883
886	44.6.20	SS066.6.20	SS066 - AFCEE Review and Acceptance of CY13 Q3 LTO/LTM Report	23 days	Tue 10/1/13	Thu 10/31/13	885
887	44.6.21	SS066.6.21	SS066 - Conduct Monitoring, Prep and Submit CY13 Q4 LTO/LTM Report	66 days	Tue 10/1/13	Tue 12/31/13	885
888	44.6.22	SS066.6.22	SS066 - AFCEE Review and Acceptance of CY13 Q4 LTO/LTM Report	23 days	Wed 1/1/14	Fri 1/31/14	887
889	44.6.23	SS066.6.23	SS066 - Conduct Monitoring, Prep and Submit CY14 Q1 LTO/LTM Report	64 days	Wed 1/1/14	Mon 3/31/14	887
890	44.6.24	SS066.6.24	SS066 - AFCEE Review and Acceptance of CY14 Q1 LTO/LTM Report	22 days	Tue 4/1/14	Wed 4/30/14	889
891	44.6.25	SS066.6.25	SS066 - Conduct Monitoring, Prep and Submit CY14 Q2 LTO/LTM Report	65 days	Tue 4/1/14	Mon 6/30/14	889
892	44.6.26	SS066.6.26	SS066 - AFCEE Review and Acceptance of CY14 Q2 LTO/LTM Report	23 days	Tue 7/1/14	Thu 7/31/14	891
893	44.6.27	SS066.6.27	SS066 - Conduct Monitoring, Prep and Submit CY14 Q3 LTO/LTM Report	66 days	Tue 7/1/14	Tue 9/30/14	891
894	44.6.28	SS066.6.28	SS066 - AFCEE Review and Acceptance of CY14 Q3 LTO/LTM Report	23 days	Wed 10/1/14	Fri 10/31/14	893
895	44.6.29	SS066.6.29	SS066 - Conduct Monitoring, Prep and Submit CY14 Q4 LTO/LTM Report	66 days	Wed 10/1/14	Wed 12/31/14	893
896	44.6.30	SS066.6.30	SS066 - AFCEE Review and Acceptance of CY14 Q4 LTO/LTM Report	22 days	Thu 1/1/15	Fri 1/30/15	895
897	44.6.31	SS066.6.31	SS066 - Conduct Monitoring, Prep and Submit CY15 Q1 LTO/LTM Report	64 days	Thu 1/1/15	Tue 3/31/15	895
898	44.6.32	SS066.6.32	SS066 - AFCEE Review and Acceptance of CY15 Q1 LTO/LTM Report	22 days	Wed 4/1/15	Thu 4/30/15	897
899	44.6.33	SS066.6.33	SS066 - Conduct Monitoring, Prep and Submit CY15 Q2 LTO/LTM Report	65 days	Wed 4/1/15	Tue 6/30/15	897
900	44.6.34	SS066.6.34	SS066 - AFCEE Review and Acceptance of CY15 Q2 LTO/LTM Report	23 days	Wed 7/1/15	Fri 7/31/15	899
901	44.7	SS066.7	SS066 - Achieve Site Closure	66 days	Wed 7/1/15	Wed 9/30/15	900SS
902	44.8	SS066.8	SS066 - Prepare and Submit Closure Report	23 days	Wed 7/1/15	Fri 7/31/15	901SS
903	44.9	SS066.9	SS066 - AFCEE Review and Acceptance of Closure Report	43 days	Mon 8/3/15	Wed 9/30/15	902
904	44.10	SS066.10	SS066 - NYSDEC Closure of Spill Number	1 day	Wed 9/30/15	Wed 9/30/15	903FF
905	44.11	SS066.11	SS066 - Wells Decommissioned	9 days	Thu 10/1/15	Tue 10/13/15	904
906	**45**	**SS067**	**SS067 - BLDG 789 (Optimized Exit Strategy)**	**1299 days**	**Mon 1/10/11**	**Thu 12/31/15**	
907	45.1	SS067.1	SS067 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	Mon 1/10/11	Fri 4/29/11	
908	45.2	SS067.2	SS067 - Design/Plan Remedy Optimation	65 days	Mon 1/31/11	Fri 4/29/11	907FF
909	45.3	SS067.3	SS067 - AFCEE Review and Acceptance of Design/Plan Remedy Optimization	44 days	Mon 5/2/11	Thu 6/30/11	908
910	45.4	SS067.4	SS067 - Complete Supplemental Investigation, submit Investigation Report	66 days	Fri 7/1/11	Fri 9/30/11	909
911	45.5	SS067.5	SS067 - Implement Remedy Optimization Systems and Submit Report	66 days	Fri 7/1/11	Fri 9/30/11	909
912	45.6	SS067.6	SS067 - AFCEE Review and Acceptance of Investigation Report	20 days	Mon 10/3/11	Fri 10/28/11	911
913	45.7	SS067.7	SS067 - AFCEE Review and Acceptance of Optimization System and Report	20 days	Mon 10/3/11	Fri 10/28/11	911
914	**45.8**	**SS067.8**	**SS067 - Conduct Monitoring, Operations & Maintenance**	**1240 days**	**Fri 4/1/11**	**Thu 12/31/15**	
915	45.8.1	SS067.8.1	SS067 - Conduct Monitoring, Prep and Submit CY11 Q2 LTO/LTM Report	65 days	Fri 4/1/11	Thu 6/30/11	908FS-21 days
916	45.8.2	SS067.8.2	SS067 - AFCEE Review and Acceptance of CY11 Q2 LTO/LTM Report	21 days	Fri 7/1/11	Fri 7/29/11	915
917	45.8.3	SS067.8.3	SS067 - Conduct Monitoring, Prep and Submit CY11 Q3 LTO/LTM Report	66 days	Fri 7/1/11	Fri 9/30/11	915
918	45.8.4	SS067.8.4	SS067 - AFCEE Review and Acceptance of CY11 Q3 LTO/LTM Report	21 days	Mon 10/3/11	Mon 10/31/11	917



ID	WBS	WBS Codes	Task Name	Duration	Start	Finish	Predecessors
919	45.8.5	SS067.8.5	SS067 - Conduct Monitoring, Prep and Submit CY11 Q4 LTO/LTM Report	65 days	Mon 10/3/11	Fri 12/30/11	913SS
920	45.8.6	SS067.8.6	SS067 - AFCEE Review and Acceptance of CY11 Q4 LTO/LTM Report	22 days	Mon 1/2/12	Tue 1/31/12	919
921	45.8.7	SS067.8.7	SS067 - Conduct Monitoring, Prep and Submit CY12 Q1 LTO/LTM Report	65 days	Mon 1/2/12	Fri 3/30/12	920SS
922	45.8.8	SS067.8.8	SS067 - AFCEE Review and Acceptance of CY12 Q1 LTO/LTM Report	21 days	Mon 4/2/12	Mon 4/30/12	921
923	45.8.9	SS067.8.9	SS067 - Conduct Monitoring, Prep and Submit CY12 Q2 LTO/LTM Report	65 days	Mon 4/2/12	Fri 6/29/12	921
924	45.8.10	SS067.8.10	SS067 - AFCEE Review and Acceptance of CY12 Q2 LTO/LTM Report	22 days	Mon 7/2/12	Tue 7/31/12	923
925	45.8.11	SS067.8.11	SS067 - Conduct Monitoring, Prep and Submit CY12 Q3 LTO/LTM Report	65 days	Mon 7/2/12	Fri 9/28/12	923
926	45.8.12	SS067.8.12	SS067 AFCEE Review and Acceptance of CY12 Q3 LTO/LTM Report	23 days	Mon 10/1/12	Wed 10/31/12	925
927	45.8.13	SS067.8.13	SS067 - Conduct Monitoring, Prep and Submit CY12 Q4 LTO/LTM Report	66 days	Mon 10/1/12	Mon 12/31/12	925
928	45.8.14	SS067.8.14	SS067 - AFCEE Review and Acceptance of CY12 Q4 LTO/LTM Report	22 days	Tue 1/1/13	Wed 1/30/13	927
929	45.8.15	SS067.8.15	SS067 - Conduct Monitoring, Prep and Submit CY13 Q1 LTO/LTM Report	64 days	Tue 1/1/13	Fri 3/29/13	927
930	45.8.16	SS067.8.16	SS067 - AFCEE Review and Acceptance of CY13 Q1 LTO/LTM Report	22 days	Mon 4/1/13	Tue 4/30/13	929
931	45.8.17	SS067.8.17	SS067 - Conduct Monitoring, Prep and Submit CY13 Q2 LTO/LTM Report	65 days	Mon 4/1/13	Fri 6/28/13	929
932	45.8.18	SS067.8.18	SS067 - AFCEE Review and Acceptance of CY13 Q2 LTO/LTM Report	23 days	Mon 7/1/13	Wed 7/31/13	931
933	45.8.19	SS067.8.19	SS067 - Conduct Monitoring, Prep and Submit CY13 Q3 LTO/LTM Report	66 days	Mon 7/1/13	Mon 9/30/13	931
934	45.8.20	SS067.8.20	SS067 - AFCEE Review and Acceptance of CY13 Q3 LTO/LTM Report	23 days	Tue 10/1/13	Thu 10/31/13	933
935	45.8.21	SS067.8.21	SS067 - Conduct Monitoring, Prep and Submit CY13 Q4 LTO/LTM Report	66 days	Tue 10/1/13	Tue 12/31/13	933
936	45.8.22	SS067.8.22	SS067 - AFCEE Review and Acceptance of CY13 Q4 LTO/LTM Report	23 days	Wed 1/1/14	Fri 1/31/14	935
937	45.8.23	SS067.8.23	SS067 - Conduct Monitoring, Prep and Submit CY14 Q1 LTO/LTM Report	64 days	Wed 1/1/14	Mon 3/31/14	935
938	45.8.24	SS067.8.24	SS067 - AFCEE Review and Acceptance of CY14 Q1 LTO/LTM Report	22 days	Tue 4/1/14	Wed 4/30/14	937
939	45.8.25	SS067.8.25	SS067 - Conduct Monitoring, Prep and Submit CY14 Q2 LTO/LTM Report	65 days	Tue 4/1/14	Mon 6/30/14	937
940	45.8.26	SS067.8.26	SS067 - AFCEE Review and Acceptance of CY14 Q2 LTO/LTM Report	23 days	Tue 7/1/14	Thu 7/31/14	939
941	45.8.27	SS067.8.27	SS067 - Conduct Monitoring, Prep and Submit CY14 Q3 LTO/LTM Report	66 days	Tue 7/1/14	Tue 9/30/14	939
942	45.8.28	SS067.8.28	SS067 - AFCEE Review and Acceptance of CY14 Q3 LTO/LTM Report	23 days	Wed 10/1/14	Fri 10/31/14	941
943	45.8.29	SS067.8.29	SS067 - Conduct Monitoring, Prep and Submit CY14 Q4 LTO/LTM Report	66 days	Wed 10/1/14	Wed 12/31/14	941
944	45.8.30	SS067.8.30	SS067 - AFCEE Review and Acceptance of CY14 Q4 LTO/LTM Report	22 days	Thu 1/1/15	Fri 1/30/15	943
945	45.8.31	SS067.8.31	SS067 - Conduct Monitoring, Prep and Submit CY15 Q1 LTO/LTM Report	64 days	Thu 1/1/15	Tue 3/31/15	943
946	45.8.32	SS067.8.32	SS067 - AFCEE Review and Acceptance of CY15 Q1 LTO/LTM Report	22 days	Wed 4/1/15	Thu 4/30/15	945
947	45.8.33	SS067.8.33	SS067 - Conduct Monitoring, Prep and Submit CY15 Q2 LTO/LTM Report	65 days	Wed 4/1/15	Tue 6/30/15	945
948	45.8.34	SS067.8.34	SS067 - AFCEE Review and Acceptance of CY15 Q2 LTO/LTM Report	23 days	Wed 7/1/15	Fri 7/31/15	947
949	45.8.35	SS067.8.35	SS067 - Conduct Monitoring, Prep and Submit CY15 Q3 LTO/LTM Report	66 days	Wed 7/1/15	Wed 9/30/15	947
950	45.8.36	SS067.8.36	SS067 - AFCEE Review and Acceptance of CY15 Q3 LTO/LTM Report	22 days	Thu 10/1/15	Fri 10/30/15	949
951	45.8.37	SS067.8.37	SS067 - Conduct Monitoring, Prep and Submit CY15 Q4 LTO/LTM Report	43 days	Thu 10/1/15	Mon 11/30/15	949
952	45.8.38	SS067.8.38	SS067 - AFCEE Review and Acceptance of CY15 Q4 LTO/LTM Report	23 days	Tue 12/1/15	Thu 12/31/15	951
953	45.9	SS067.9	SS067 - Optimization Exit Strategy	66 days	Wed 7/1/15	Wed 9/30/15	948SS
954	45.10	SS067.10	SS067 - Prepare and Submit Optimized Exit Strategy Report	23 days	Wed 7/1/15	Fri 7/31/15	953SS
955	45.11	SS067.11	SS067 - AFCEE Review and Acceptance of Optimized Exit Strategy Report	43 days	Mon 8/3/15	Wed 9/30/15	954
956	**46**	**SS068**	**SS068 - BLDG 7001 (Closure Unrestricted Reuse)**	**1242 days**	**Mon 1/10/11**	**Tue 10/13/15**	
957	46.1	SS068.1	SS068 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	Mon 1/10/11	Fri 4/29/11	
958	46.2	SS068.2	SS068 - Prepare and Submit Design/plan remedy optimization	65 days	Mon 1/31/11	Fri 4/29/11	957FF
959	46.3	SS068.3	SS068 - AFCEE Review and Acceptance of Design/Plan Remedy Optimization	44 days	Mon 5/2/11	Thu 6/30/11	958
960	46.4	SS068.4	SS068 - Implement Remedy Optimization Systems and Submit Report	66 days	Fri 7/1/11	Fri 9/30/11	959
961	46.5	SS068.5	SS068 - AFCEE Review and Acceptance of Optimization System and Report	21 days	Mon 10/3/11	Mon 10/31/11	960
962	**46.6**	**SS068.6**	**SS068 - Conduct Monitoring, Operations & Maintenance**	**1131 days**	**Fri 4/1/11**	**Fri 7/31/15**	
963	46.6.1	SS068.6.1	SS068 - Conduct Monitoring, Prep and Submit CY11 Q2 LTO/LTM Report	65 days	Fri 4/1/11	Thu 6/30/11	958FS-21 days
964	46.6.2	SS068.6.2	SS068 - AFCEE Review and Acceptance of CY11 Q2 LTO/LTM Report	21 days	Fri 7/1/11	Fri 7/29/11	963
965	46.6.3	SS068.6.3	SS068 - Conduct Monitoring, Prep and Submit CY11 Q3 LTO/LTM Report	66 days	Fri 7/1/11	Fri 9/30/11	963
966	46.6.4	SS068.6.4	SS068 - AFCEE Review and Acceptance of CY11 Q3 LTO/LTM Report	21 days	Mon 10/3/11	Mon 10/31/11	965
967	46.6.5	SS068.6.5	SS068 - Conduct Monitoring, Prep and Submit CY11 Q4 LTO/LTM Report	65 days	Mon 10/3/11	Fri 12/30/11	961SS
968	46.6.6	SS068.6.6	SS068 - AFCEE Review and Acceptance of CY11 Q4 LTO/LTM Report	22 days	Mon 1/2/12	Tue 1/31/12	967
969	46.6.7	SS068.6.7	SS068 - Conduct Monitoring, Prep and Submit CY12 Q1 LTO/LTM Report	65 days	Mon 1/2/12	Fri 3/30/12	967
970	46.6.8	SS068.6.8	SS068 - AFCEE Review and Acceptance of CY12 Q1 LTO/LTM Report	21 days	Mon 4/2/12	Mon 4/30/12	969
971	46.6.9	SS068.6.9	SS068 - Conduct Monitoring, Prep and Submit CY12 Q2 LTO/LTM Report	65 days	Mon 4/2/12	Fri 6/29/12	969
972	46.6.10	SS068.6.10	SS068 - AFCEE Review and Acceptance of CY12 Q2 LTO/LTM Report	22 days	Mon 7/2/12	Tue 7/31/12	971



ID	WBS	WBS Codes	Task Name	Duration	Start	Finish	Predecessors
973	46.6.11	SS068.6.11	SS068 - Conduct Monitoring, Prep and Submit CY12 Q3 LTO/LTM Report	65 days	Mon 7/2/12	Fri 9/28/12	971
974	46.6.12	SS068.6.12	SS068 - AFCEE Review and Acceptance of CY12 Q3 LTO/LTM Report	23 days	Mon 10/1/12	Wed 10/31/12	973
975	46.6.13	SS068.6.13	SS068 - Conduct Monitoring, Prep and Submit CY12 Q4 LTO/LTM Report	66 days	Mon 10/1/12	Mon 12/31/12	973
976	46.6.14	SS068.6.14	SS068 - AFCEE Review and Acceptance of CY12 Q4 LTO/LTM Report	22 days	Tue 1/1/13	Wed 1/30/13	975
977	46.6.15	SS068.6.15	SS068 - Conduct Monitoring, Prep and Submit CY13 Q1 LTO/LTM Report	64 days	Tue 1/1/13	Fri 3/29/13	975
978	46.6.16	SS068.6.16	SS068 - AFCEE Review and Acceptance of CY13 Q1 LTO/LTM Report	22 days	Mon 4/1/13	Tue 4/30/13	977
979	46.6.17	SS068.6.17	SS068 - Conduct Monitoring, Prep and Submit CY13 Q2 LTO/LTM Report	65 days	Mon 4/1/13	Fri 6/28/13	977
980	46.6.18	SS068.6.18	SS068 - AFCEE Review and Acceptance of CY13 Q2 LTO/LTM Report	23 days	Mon 7/1/13	Wed 7/31/13	979
981	46.6.19	SS068.6.19	SS068 - Conduct Monitoring, Prep and Submit CY13 Q3 LTO/LTM Report	66 days	Mon 7/1/13	Mon 9/30/13	979
982	46.6.20	SS068.6.20	SS068 - AFCEE Review and Acceptance of CY13 Q3 LTO/LTM Report	23 days	Tue 10/1/13	Thu 10/31/13	981
983	46.6.21	SS068.6.21	SS068 - Conduct Monitoring, Prep and Submit CY13 Q4 LTO/LTM Report	66 days	Tue 10/1/13	Tue 12/31/13	981
984	46.6.22	SS068.6.22	SS068 - AFCEE Review and Acceptance of CY13 Q4 LTO/LTM Report	23 days	Wed 1/1/14	Fri 1/31/14	983
985	46.6.23	SS068.6.23	SS068 - Conduct Monitoring, Prep and Submit CY14 Q1 LTO/LTM Report	64 days	Wed 1/1/14	Mon 3/31/14	983
986	46.6.24	SS068.6.24	SS068 - AFCEE Review and Acceptance of CY14 Q1 LTO/LTM Report	22 days	Tue 4/1/14	Wed 4/30/14	985
987	46.6.25	SS068.6.25	SS068 - Conduct Monitoring, Prep and Submit CY14 Q2 LTO/LTM Report	65 days	Tue 4/1/14	Mon 6/30/14	985
988	46.6.26	SS068.6.26	SS068 - AFCEE Review and Acceptance of CY14 Q2 LTO/LTM Report	23 days	Tue 7/1/14	Thu 7/31/14	987
989	46.6.27	SS068.6.27	SS068 - Conduct Monitoring, Prep and Submit CY14 Q3 LTO/LTM Report	66 days	Tue 7/1/14	Tue 9/30/14	987
990	46.6.28	SS068.6.28	SS068 - AFCEE Review and Acceptance of CY14 Q3 LTO/LTM Report	23 days	Wed 10/1/14	Fri 10/31/14	989
991	46.6.29	SS068.6.29	SS068 - Conduct Monitoring, Prep and Submit CY14 Q4 LTO/LTM Report	66 days	Wed 10/1/14	Wed 12/31/14	989
992	46.6.30	SS068.6.30	SS068 - AFCEE Review and Acceptance of CY14 Q4 LTO/LTM Report	22 days	Thu 1/1/15	Fri 1/30/15	991
993	46.6.31	SS068.6.31	SS068 - Conduct Monitoring, Prep and Submit CY15 Q1 LTO/LTM Report	64 days	Thu 1/1/15	Tue 3/31/15	991
994	46.6.32	SS068.6.32	SS068 - AFCEE Review and Acceptance of CY15 Q1 LTO/LTM Report	22 days	Wed 4/1/15	Thu 4/30/15	993
995	46.6.33	SS068.6.33	SS068 - Conduct Monitoring, Prep and Submit CY15 Q2 LTO/LTM Report	65 days	Wed 4/1/15	Tue 6/30/15	993
996	46.6.34	SS068.6.34	SS068 - AFCEE Review and Acceptance of CY15 Q2 LTO/LTM Report	23 days	Wed 7/1/15	Fri 7/31/15	995
997	46.7	SS068.7	SS068 - Achieve Site Closure	66 days	Wed 7/1/15	Wed 9/30/15	996SS
998	46.8	SS068.8	SS068 - Prepare and Submit Closure Report	23 days	Wed 7/1/15	Fri 7/31/15	997SS
999	46.9	SS068.9	SS068 - AFCEE Review and Acceptance of Closure Report	43 days	Mon 8/3/15	Wed 9/30/15	998
1000	46.10	SS068.10	SS068 - NYSDEC Closure of Spill Number	1 day	Wed 9/30/15	Wed 9/30/15	999FF
1001	46.11	SS068.11	SS068 - Wells Decommissioned	9 days	Thu 10/1/15	Tue 10/13/15	1000
1002	**47**	**SS069**	**SS069 - Bulk Fuel Storage Area (Closure Unrestricted Reuse)**	**1038 days**	**Mon 1/10/11**	**Wed 12/31/14**	
1003	47.1	SS069.1	SS069 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	Mon 1/10/11	Fri 4/29/11	
1004	47.2	SS069.2	SS069 - Prepare and Submit Design/plan remedy optimization	65 days	Mon 1/31/11	Fri 4/29/11	1003FF
1005	47.3	SS069.3	SS069 - AFCEE Review and Acceptance of Design/Plan Remedy Optimization	44 days	Mon 5/2/11	Thu 6/30/11	1004
1006	47.4	SS069.4	SS069 - Implement Remedy Optimization Systems and Submit Report	66 days	Fri 7/1/11	Fri 9/30/11	1005
1007	47.5	SS069.5	SS069 - AFCEE Review and Acceptance of Optimization System and Report	21 days	Mon 10/3/11	Mon 10/31/11	1006
1008	**47.6**	**SS069.6**	**SS069 - Conduct Monitoring, Operations & Maintenance**	**936 days**	**Fri 4/1/11**	**Fri 10/31/14**	
1009	47.6.1	SS069.6.1	SS069 - Conduct Monitoring, Prep and Submit CY11 Q2 LTO/LTM Report	65 days	Fri 4/1/11	Thu 6/30/11	1004FS-21 days
1010	47.6.2	SS069.6.2	SS069 - AFCEE Review and Acceptance of CY11 Q2 LTO/LTM Report	21 days	Fri 7/1/11	Fri 7/29/11	1009
1011	47.6.3	SS069.6.3	SS069 - Conduct Monitoring, Prep and Submit CY11 Q3 LTO/LTM Report	66 days	Fri 7/1/11	Fri 9/30/11	1009
1012	47.6.4	SS069.6.4	SS069 - AFCEE Review and Acceptance of CY11 Q3 LTO/LTM Report	21 days	Mon 10/3/11	Mon 10/31/11	1011
1013	47.6.5	SS069.6.5	SS069 - Conduct Monitoring, Prep and Submit CY11 Q4 LTO/LTM Report	65 days	Mon 10/3/11	Fri 12/30/11	1007SS
1014	47.6.6	SS069.6.6	SS069 - AFCEE Review and Acceptance of CY11 Q4 LTO/LTM Report	22 days	Mon 1/2/12	Tue 1/31/12	1013
1015	47.6.7	SS069.6.7	SS069 - Conduct Monitoring, Prep and Submit CY12 Q1 LTO/LTM Report	65 days	Mon 1/2/12	Fri 3/30/12	1013
1016	47.6.8	SS069.6.8	SS069 - AFCEE Review and Acceptance of CY12 Q1 LTO/LTM Report	21 days	Mon 4/2/12	Mon 4/30/12	1015
1017	47.6.9	SS069.6.9	SS069 - Conduct Monitoring, Prep and Submit CY12 Q2 LTO/LTM Report	65 days	Mon 4/2/12	Fri 6/29/12	1015
1018	47.6.10	SS069.6.10	SS069 - AFCEE Review and Acceptance of CY12 Q2 LTO/LTM Report	22 days	Mon 7/2/12	Tue 7/31/12	1017
1019	47.6.11	SS069.6.11	SS069 - Conduct Monitoring, Prep and Submit CY12 Q3 LTO/LTM Report	65 days	Mon 7/2/12	Fri 9/28/12	1017
1020	47.6.12	SS069.6.12	SS069 - AFCEE Review and Acceptance of CY12 Q3 LTO/LTM Report	23 days	Mon 10/1/12	Wed 10/31/12	1019
1021	47.6.13	SS069.6.13	SS069 - Conduct Monitoring, Prep and Submit CY12 Q4 LTO/LTM Report	66 days	Mon 10/1/12	Mon 12/31/12	1019
1022	47.6.14	SS069.6.14	SS069 - AFCEE Review and Acceptance of CY12 Q4 LTO/LTM Report	22 days	Tue 1/1/13	Wed 1/30/13	1021
1023	47.6.15	SS069.6.15	SS069 - Conduct Monitoring, Prep and Submit CY13 Q1 LTO/LTM Report	64 days	Tue 1/1/13	Fri 3/29/13	1021
1024	47.6.16	SS069.6.16	SS069 - AFCEE Review and Acceptance of CY13 Q1 LTO/LTM Report	22 days	Mon 4/1/13	Tue 4/30/13	1023
1025	47.6.17	SS069.6.17	SS069 - Conduct Monitoring, Prep and Submit CY13 Q2 LTO/LTM Report	65 days	Mon 4/1/13	Fri 6/28/13	1023
1026	47.6.18	SS069.6.18	SS069 - AFCEE Review and Acceptance of CY13 Q2 LTO/LTM Report	23 days	Mon 7/1/13	Wed 7/31/13	1025

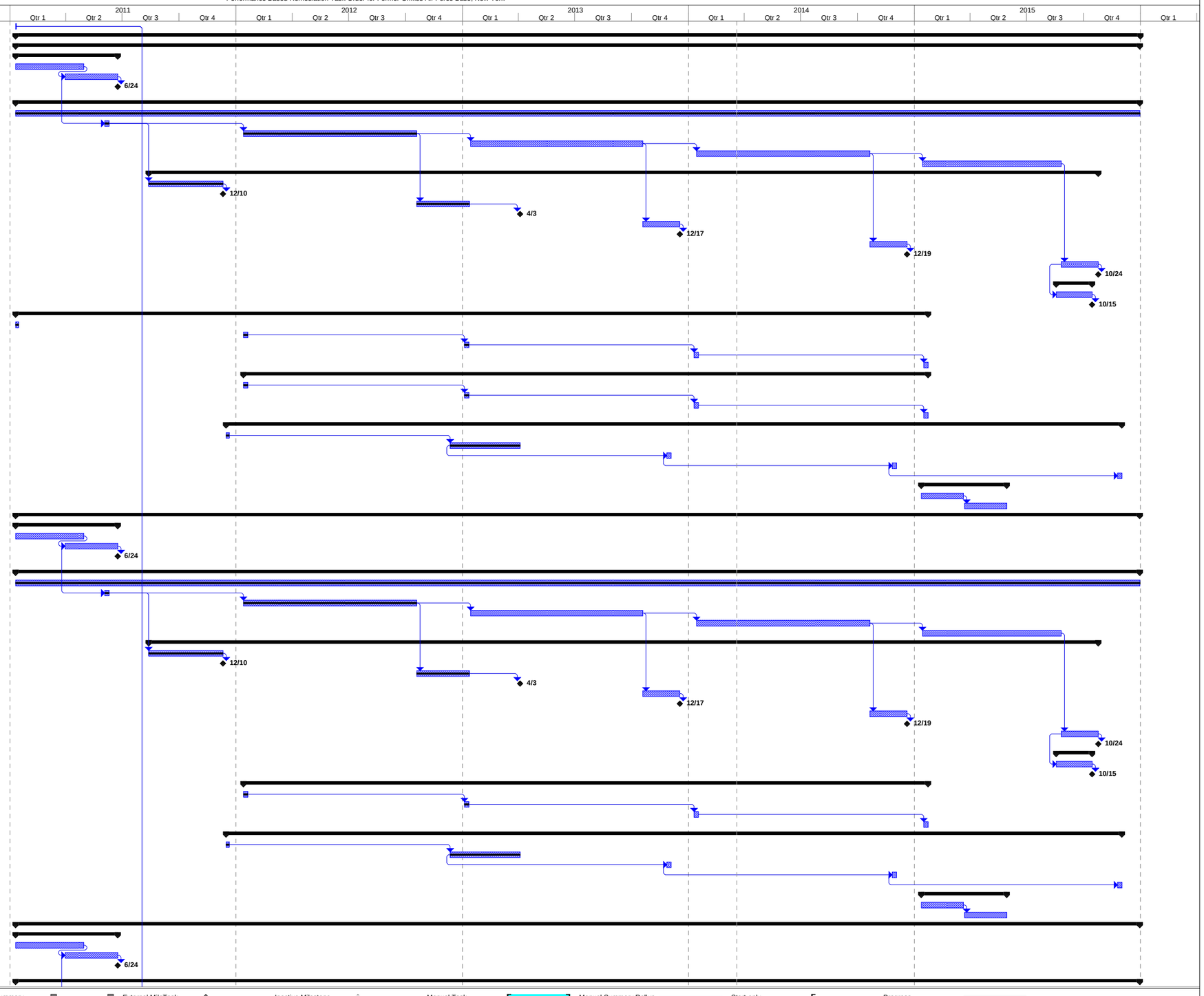


Project: Project1
Date: Thu 3/24/11

Task | Progress | Summary | External Tasks | Split
Split | Milestone | Project Summary | External MileTask

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York

ID	Task Name	Duration	Start	Finish
1	**Kick Off Meeting at Griffiss AFB**	**1 day**	**1/10/2011**	**1/10/2011**
2	**Griffiss PBR 2011**	**1299 days**	**1/10/2011**	**12/31/2015**
3	**LF001 - Landfill 1 (Optimized Exit Strategy)**	**1298 days**	**1/10/2011**	**12/31/2015**
4	**LF001 - Planning Documents**	**119 days**	**1/10/2011**	**6/24/2011**
5	LF001 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDE	80 days	1/10/2011	4/29/2011
6	LF001 - Design and Remedy Optimization Documents	85 edays	3/31/2011	6/24/2011
7	LF001 - Acceptance of Final Design and Remedy Optimization Documents	0 days	6/24/2011	6/24/2011
8	**LF001 - Field Work**	**1298 days**	**1/10/2011**	**12/31/2015**
9	LF001 - Landfill O&M	1816 edays	1/10/2011	12/31/2015
10	LF001 - Monitoring Year 1	5 days	6/3/2011	6/9/2011
11	LF001 - Monitoring Year 2	200 days	1/13/2012	10/18/2012
12	LF001 - Monitoring Year 3	200 days	1/14/2013	10/18/2013
13	LF001 - Monitoring Year 4	200 days	1/14/2014	10/20/2014
14	LF001 - Monitoring Year 5	160 days	1/14/2015	8/25/2015
15	**LF001 - Reporting**	**1095 days**	**8/12/2011**	**10/24/2015**
16	LF001 - Long-Term Monitoring Report - Year 1	120 edays	8/12/2011	12/10/2011
17	LF001 - Acceptance of Long-Term Monitoring Report - Year 1	0 days	12/10/2011	12/10/2011
18	LF001 - Long-Term Monitoring Report - Year 2	85 edays	10/18/2012	1/11/2013
19	LF001 - Acceptance of Long-Term Monitoring Report - Year 2	0 days	4/3/2013	4/3/2013
20	LF001 - Long-Term Monitoring Report - Year 3	60 edays	10/18/2013	12/17/2013
21	LF001 - Acceptance of Long-Term Monitoring Report - Year 3	0 days	12/17/2013	12/17/2013
22	LF001 - Long-Term Monitoring Report - Year 4	60 edays	10/20/2014	12/19/2014
23	LF001 - Acceptance of Long-Term Monitoring Report - Year 4	0 days	12/19/2014	12/19/2014
24	LF001 - Long-Term Monitoring Report - Year 5	60 edays	8/25/2015	10/24/2015
25	LF001 - Acceptance of Long-Term Monitoring Report - Year 5	0 days	10/24/2015	10/24/2015
26	**LF001 - Optimized Exit Strategy**	**42 days**	**8/18/2015**	**10/15/2015**
27	LF001 - Optimized Exit Strategy Document	58 edays	8/18/2015	10/15/2015
28	LF001 - Acceptance of Optimized Exit Strategy Document	0 days	10/15/2015	10/15/2015
29	**LF001 - PMP and PMP update**	**1054 days**	**1/10/2011**	**1/22/2015**
30	LF001 - AF acceptance of PMP Monitoring Year 1	5 edays	1/10/2011	1/15/2011
31	LF001 - AF acceptance of PMP Monitoring Year 2	5 days	1/13/2012	1/19/2012
32	LF001 - AF acceptance of PMP Monitoring Year 3	5 days	1/4/2013	1/10/2013
33	LF001 - AF acceptance of PMP Monitoring Year 4	5 days	1/10/2014	1/16/2014
34	LF001 - AF acceptance of PMP Monitoring Year 5	5 days	1/16/2015	1/22/2015
35	**LF001 - UFP QAPP and UFP QAPP update**	**790 days**	**1/13/2012**	**1/22/2015**
36	LF001 - AF acceptance of UFP QAPP Monitoring Year 2	5 days	1/13/2012	1/19/2012
37	LF001 - AF acceptance of UFP QAPP Monitoring Year 3	5 days	1/4/2013	1/10/2013
38	LF001 - AF acceptance of UFP QAPP Monitoring Year 4	5 days	1/10/2014	1/16/2014
39	LF001 - AF acceptance of UFP QAPP Monitoring Year 5	5 days	1/16/2015	1/22/2015
40	**LF001 - LUC/IC Site Inspections**	**1033 days**	**12/16/2011**	**12/1/2015**
41	LF001 - Acceptance of LUC/IC Site Inspection Report 1	5 edays	12/16/2011	12/21/2011
42	LF001 - Acceptance of LUC/IC Site Inspection Report 2	81 days	12/12/2012	4/3/2013
43	LF001 - Acceptance of LUC/IC Site Inspection Report 3	5 days	11/27/2013	12/3/2013
44	LF001 - Acceptance of LUC/IC Site Inspection Report 4	5 days	11/26/2014	12/2/2014
45	LF001 - Acceptance of LUC/IC Site Inspection Report 5	5 days	11/25/2015	12/1/2015
46	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
47	Five-Year Review Report	50 days	1/12/2015	3/20/2015
48	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
49	**LF002 - Landfill 2/3 (Optimized Exit Strategy)**	**1298 days**	**1/10/2011**	**12/31/2015**
50	**LF002 - Planning Documents**	**119 days**	**1/10/2011**	**6/24/2011**
51	LF002 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDE	80 days	1/10/2011	4/29/2011
52	LF002 - Design and Remedy Optimization Documents	85 edays	3/31/2011	6/24/2011
53	LF002 - Acceptance of Final Design and Remedy Optimization Documents	0 days	6/24/2011	6/24/2011
54	**LF002 - Field Work**	**1298 days**	**1/10/2011**	**12/31/2015**
55	LF002 - Landfill O&M	1816 edays	1/10/2011	12/31/2015
56	LF002 - Monitoring Year 1	5 days	6/3/2011	6/9/2011
57	LF002 - Monitoring Year 2	200 days	1/13/2012	10/18/2012
58	LF002 - Monitoring Year 3	200 days	1/14/2013	10/18/2013
59	LF002 - Monitoring Year 4	200 days	1/14/2014	10/20/2014
60	LF002 - Monitoring Year 5	160 days	1/14/2015	8/25/2015
61	**LF002 - Reporting**	**1095 days**	**8/12/2011**	**10/24/2015**
62	LF002 - Long-Term Monitoring Report - Year 1	120 edays	8/12/2011	12/10/2011
63	LF002 - Acceptance of Long-Term Monitoring Report - Year 1	0 days	12/10/2011	12/10/2011
64	LF002 - Long-Term Monitoring Report - Year 2	85 edays	10/18/2012	1/11/2013
65	LF002 - Acceptance of Long-Term Monitoring Report - Year 2	0 days	4/3/2013	4/3/2013
66	LF002 - Long-Term Monitoring Report - Year 3	60 edays	10/18/2013	12/17/2013
67	LF002 - Acceptance of Long-Term Monitoring Report - Year 3	0 days	12/17/2013	12/17/2013
68	LF002 - Long-Term Monitoring Report - Year 4	60 edays	10/20/2014	12/19/2014
69	LF002 - Acceptance of Long-Term Monitoring Report - Year 4	0 days	12/19/2014	12/19/2014
70	LF002 - Long-Term Monitoring Report - Year 5	60 edays	8/25/2015	10/24/2015
71	LF002 - Acceptance of Long-Term Monitoring Report - Year 5	0 days	10/24/2015	10/24/2015
72	**LF002 - Optimized Exit Strategy**	**42 days**	**8/18/2015**	**10/15/2015**
73	LF002 - Optimized Exit Strategy Document	58 edays	8/18/2015	10/15/2015
74	LF002 - Acceptance of Optimized Exit Strategy Document	0 days	10/15/2015	10/15/2015
75	**LF002 - UFP QAPP and UFP QAPP update**	**790 days**	**1/13/2012**	**1/22/2015**
76	LF002 - AF acceptance of UFP QAPP Monitoring Year 2	5 days	1/13/2012	1/19/2012
77	LF002 - AF acceptance of UFP QAPP Monitoring Year 3	5 days	1/4/2013	1/10/2013
78	LF002 - AF acceptance of UFP QAPP Monitoring Year 4	5 days	1/10/2014	1/16/2014
79	LF002 - AF acceptance of UFP QAPP Monitoring Year 5	5 days	1/16/2015	1/22/2015
80	**LF002 - LUC/IC Site Inspections**	**1033 days**	**12/16/2011**	**12/1/2015**
81	LF002 - Acceptance of LUC/IC Site Inspection Report 1	5 edays	12/16/2011	12/21/2011
82	LF002 - Acceptance of LUC/IC Site Inspection Report 2	81 days	12/12/2012	4/3/2013
83	LF002 - Acceptance of LUC/IC Site Inspection Report 3	5 days	11/27/2013	12/3/2013
84	LF002 - Acceptance of LUC/IC Site Inspection Report 4	5 days	11/26/2014	12/2/2014
85	LF002 - Acceptance of LUC/IC Site Inspection Report 5	5 days	11/25/2015	12/1/2015
86	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
87	Five-Year Review Report	50 days	1/12/2015	3/20/2015
88	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
89	**LF003 - Landfill 7 (Optimized Exit Strategy)**	**1298 days**	**1/10/2011**	**12/31/2015**
90	**LF003 - Planning Documents**	**119 days**	**1/10/2011**	**6/24/2011**
91	LF003 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDE	80 days	1/10/2011	4/29/2011
92	LF003 - Design and Remedy Optimization Documents	85 edays	3/31/2011	6/24/2011
93	LF003 - Acceptance of Final Design and Remedy Optimization Documents	0 days	6/24/2011	6/24/2011
94	**LF003 - Field Work**	**1298 days**	**1/10/2011**	**12/31/2015**



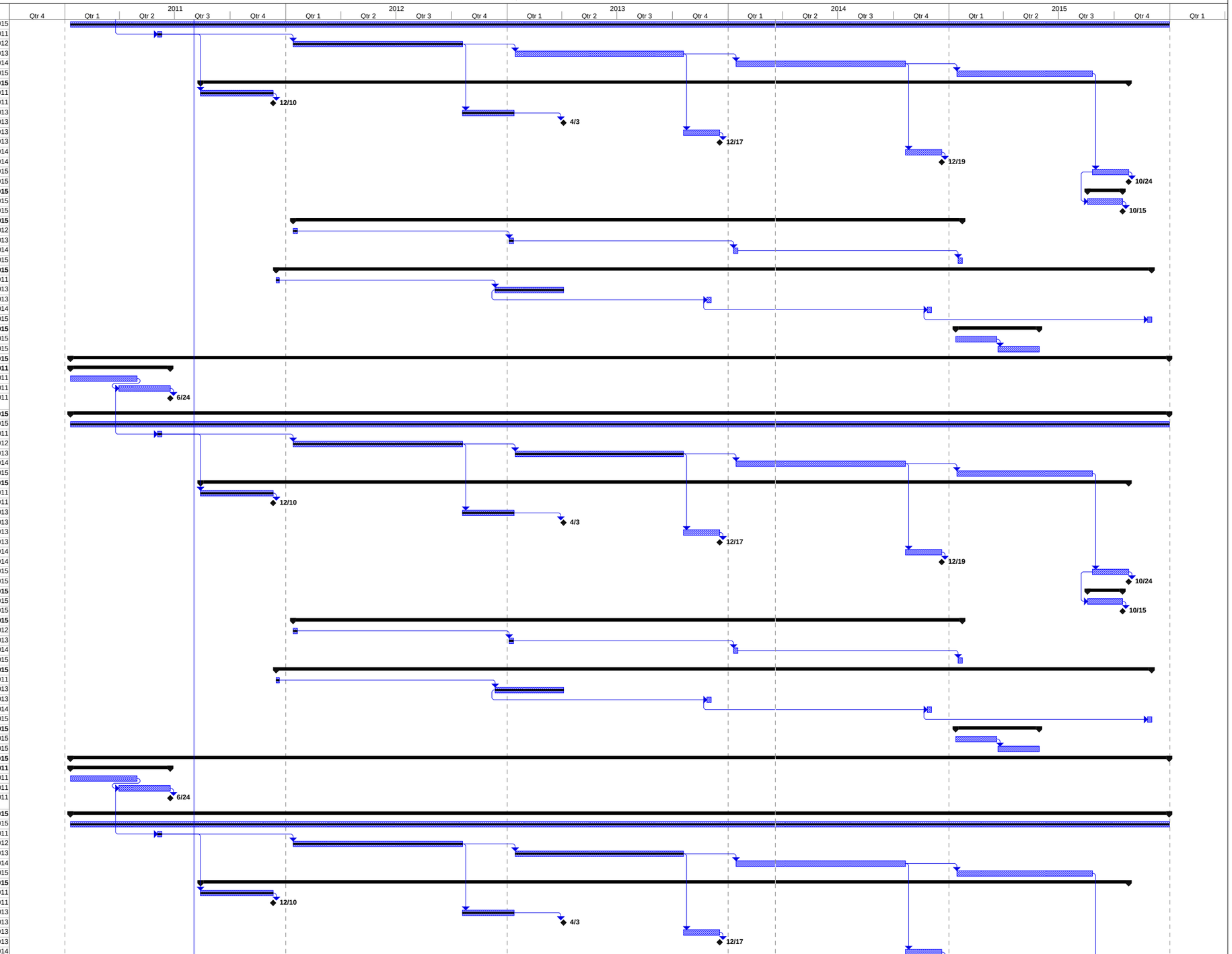
Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York

ID	Task Name	Duration	Start	Finish
95	LF003 - Landfill O&M	1816 edays	1/10/2011	12/31/2015
96	LF003 - Monitoring Year 1	5 days	6/3/2011	6/9/2011
97	LF003 - Monitoring Year 2	200 days	1/13/2012	10/18/2012
98	LF003 - Monitoring Year 3	200 days	1/14/2013	10/18/2013
99	LF003 - Monitoring Year 4	200 days	1/14/2014	10/20/2014
100	LF003 - Monitoring Year 5	160 days	1/14/2015	8/25/2015
101	**LF003 - Reporting**	**1095 days**	**8/12/2011**	**10/24/2015**
102	LF003 - Long-Term Monitoring Report - Year 1	120 edays	8/12/2011	12/10/2011
103	LF003 - Acceptance of Long-Term Monitoring Report - Year 1	0 days	12/10/2011	12/10/2011
104	LF003 - Long-Term Monitoring Report - Year 2	85 edays	10/18/2012	1/11/2013
105	LF003 - Acceptance of Long-Term Monitoring Report - Year 2	0 days	4/3/2013	4/3/2013
106	LF003 - Long-Term Monitoring Report - Year 3	60 edays	10/18/2013	12/17/2013
107	LF003 - Acceptance of Long-Term Monitoring Report - Year 3	0 days	12/17/2013	12/17/2013
108	LF003 - Long-Term Monitoring Report - Year 4	60 edays	10/20/2014	12/19/2014
109	LF003 - Acceptance of Long-Term Monitoring Report - Year 4	0 days	12/19/2014	12/19/2014
110	LF003 - Long-Term Monitoring Report - Year 5	60 edays	8/25/2015	10/24/2015
111	LF003 - Acceptance of Long-Term Monitoring Report - Year 5	0 days	10/24/2015	10/24/2015
112	**LF003 - Optimized Exit Strategy**	**42 days**	**8/18/2015**	**10/15/2015**
113	LF003 - Optimized Exit Strategy Document	58 edays	8/18/2015	10/15/2015
114	LF003 - Acceptance of Optimized Exit Strategy Document	0 days	10/15/2015	10/15/2015
115	**LF003 - UFP QAPP and UFP QAPP update**	**790 days**	**1/13/2012**	**1/22/2015**
116	LF003 - AF acceptance of UFP QAPP Monitoring Year 2	5 days	1/13/2012	1/19/2012
117	LF003 - AF acceptance of UFP QAPP Monitoring Year 3	5 days	1/4/2013	1/10/2013
118	LF003 - AF acceptance of UFP QAPP Monitoring Year 4	5 days	1/10/2014	1/16/2014
119	LF003 - AF acceptance of UFP QAPP Monitoring Year 5	5 days	1/16/2015	1/22/2015
120	**LF003 - LUC/IC Site Inspections**	**1033 days**	**12/16/2011**	**12/1/2015**
121	LF003 - Acceptance of LUC/IC Site Inspection Report 1	5 edays	12/16/2011	12/21/2011
122	LF003 - Acceptance of LUC/IC Site Inspection Report 2	81 days	12/12/2012	4/3/2013
123	LF003 - Acceptance of LUC/IC Site Inspection Report 3	5 days	11/27/2013	12/3/2013
124	LF003 - Acceptance of LUC/IC Site Inspection Report 4	5 days	11/26/2014	12/2/2014
125	LF003 - Acceptance of LUC/IC Site Inspection Report 5	5 days	11/25/2015	12/1/2015
126	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
127	Five-Year Review Report	50 days	1/12/2015	3/20/2015
128	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
129	**LF007 - Landfill 5 (Optimized Exit Strategy)**	**1298 days**	**1/10/2011**	**12/31/2015**
130	**LF007 - Planning Documents**	**119 days**	**1/10/2011**	**6/24/2011**
131	LF007 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDE	80 days	1/10/2011	4/29/2011
132	LF007 - Design and Remedy Optimization Documents	85 edays	3/31/2011	6/24/2011
133	LF007 - Acceptance of Final Design and Remedy Optimization Documents	0 days	6/24/2011	6/24/2011
134	**LF007 - Field Work**	**1298 days**	**1/10/2011**	**12/31/2015**
135	LF007 - Landfilll O&M	1816 edays	1/10/2011	12/31/2015
136	LF007 - Monitoring Year 1	5 days	6/3/2011	6/9/2011
137	LF007 - Monitoring Year 2	200 days	1/13/2012	10/18/2012
138	LF007 - Monitoring Year 3	200 days	1/14/2013	10/18/2013
139	LF007 - Monitoring Year 4	200 days	1/14/2014	10/20/2014
140	LF007 - Monitoring Year 5	160 days	1/14/2015	8/25/2015
141	**LF007 - Reporting**	**1095 days**	**8/12/2011**	**10/24/2015**
142	LF007 - Long-Term Monitoring Report - Year 1	120 edays	8/12/2011	12/10/2011
143	LF007 - Acceptance of Long-Term Monitoring Report - Year 1	0 days	12/10/2011	12/10/2011
144	LF007 - Long-Term Monitoring Report - Year 2	85 edays	10/18/2012	1/11/2013
145	LF007 - Acceptance of Long-Term Monitoring Report - Year 2	0 days	4/3/2013	4/3/2013
146	LF007 - Long-Term Monitoring Report - Year 3	60 edays	10/18/2013	12/17/2013
147	LF007 - Acceptance of Long-Term Monitoring Report - Year 3	0 days	12/17/2013	12/17/2013
148	LF007 - Long-Term Monitoring Report - Year 4	60 edays	10/20/2014	12/19/2014
149	LF007 - Acceptance of Long-Term Monitoring Report - Year 4	0 days	12/19/2014	12/19/2014
150	LF007 - Long-Term Monitoring Report - Year 5	60 edays	8/25/2015	10/24/2015
151	LF007 - Acceptance of Long-Term Monitoring Report - Year 5	0 days	10/24/2015	10/24/2015
152	**LF007 - Optimized Exit Strategy**	**42 days**	**8/18/2015**	**10/15/2015**
153	LF007 - Optimized Exit Strategy Document	58 edays	8/18/2015	10/15/2015
154	LF007 - Acceptance of Optimized Exit Strategy Document	0 days	10/15/2015	10/15/2015
155	**LF007 - UFP QAPP and UFP QAPP update**	**790 days**	**1/13/2012**	**1/22/2015**
156	LF007 - AF acceptance of UFP QAPP Monitoring Year 2	5 days	1/13/2012	1/19/2012
157	LF007 - AF acceptance of UFP QAPP Monitoring Year 3	5 days	1/4/2013	1/10/2013
158	LF007 - AF acceptance of UFP QAPP Monitoring Year 4	5 days	1/10/2014	1/16/2014
159	LF007 - AF acceptance of UFP QAPP Monitoring Year 5	5 days	1/16/2015	1/22/2015
160	**LF007 - LUC/IC Site Inspections**	**1033 days**	**12/16/2011**	**12/1/2015**
161	LF007 - Acceptance of LUC/IC Site Inspection Report 1	5 edays	12/16/2011	12/21/2011
162	LF007 - Acceptance of LUC/IC Site Inspection Report 2	81 days	12/12/2012	4/3/2013
163	LF007 - Acceptance of LUC/IC Site Inspection Report 3	5 days	11/27/2013	12/3/2013
164	LF007 - Acceptance of LUC/IC Site Inspection Report 4	5 days	11/26/2014	12/2/2014
165	LF007 - Acceptance of LUC/IC Site Inspection Report 5	5 days	11/25/2015	12/1/2015
166	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
167	Five-Year Review Report	50 days	1/12/2015	3/20/2015
168	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
169	**LF009 - Landfill 6 (Optimized Exit Strategy)**	**1298 days**	**1/10/2011**	**12/31/2015**
170	**LF009 - Planning Documents**	**119 days**	**1/10/2011**	**6/24/2011**
171	LF009 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDE	80 days	1/10/2011	4/29/2011
172	LF009 - Design and Remedy Optimization Documents	85 edays	3/31/2011	6/24/2011
173	LF009 - Acceptance of Final Design and Remedy Optimization Documents	0 days	6/24/2011	6/24/2011
174	**LF009 - Field Work**	**1298 days**	**1/10/2011**	**12/31/2015**
175	LF009 - Landfilll O&M	1816 edays	1/10/2011	12/31/2015
176	LF009 - Monitoring Year 1	5 days	6/3/2011	6/9/2011
177	LF009 - Monitoring Year 2	200 days	1/13/2012	10/18/2012
178	LF009 - Monitoring Year 3	200 days	1/14/2013	10/18/2013
179	LF009 - Monitoring Year 4	200 days	1/14/2014	10/20/2014
180	LF009 - Monitoring Year 5	160 days	1/14/2015	8/25/2015
181	**LF009 - Reporting**	**1095 days**	**8/12/2011**	**10/24/2015**
182	LF009 - Long-Term Monitoring Report - Year 1	120 edays	8/12/2011	12/10/2011
183	LF009 - Acceptance of Long-Term Monitoring Report - Year 1	0 days	12/10/2011	12/10/2011
184	LF009 - Long-Term Monitoring Report - Year 2	85 edays	10/18/2012	1/11/2013
185	LF009 - Acceptance of Long-Term Monitoring Report - Year 2	0 days	4/3/2013	4/3/2013
186	LF009 - Long-Term Monitoring Report - Year 3	60 edays	10/18/2013	12/17/2013
187	LF009 - Acceptance of Long-Term Monitoring Report - Year 3	0 days	12/17/2013	12/17/2013
188	LF009 - Long-Term Monitoring Report - Year 4	60 edays	10/20/2014	12/19/2014



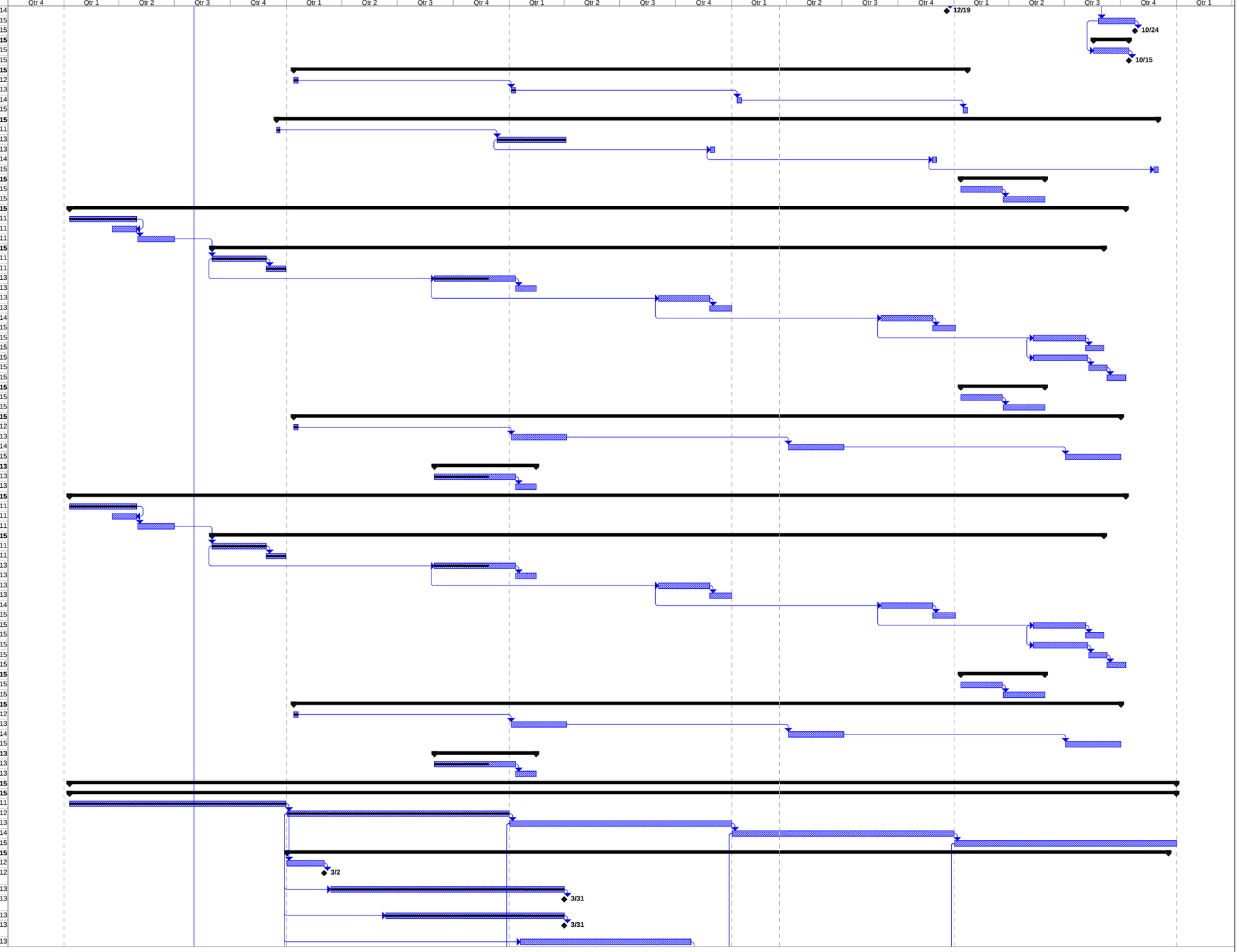
Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York

ID	Task Name	Duration	Start	Finish
189	LF009 - Acceptance of Long-Term Monitoring Report - Year 4	0 days	12/19/2014	12/19/2014
190	LF009 - Long-Term Monitoring Report - Year 5	60 edays	8/25/2015	10/24/2015
191	LF009 - Acceptance of Long-Term Monitoring Report - Year 5	0 days	10/24/2015	10/24/2015
192	**LF009 - Optimized Exit Strategy**	**42 days**	**8/18/2015**	**10/15/2015**
193	LF009 - Optimized Exit Strategy Document	58 edays	8/18/2015	10/15/2015
194	LF009 - Acceptance of Optimized Exit Strategy Document	0 days	10/15/2015	10/15/2015
195	**LF009 - UFP QAPP and UFP QAPP update**	**790 days**	**1/13/2012**	**1/22/2015**
196	LF009 - AF acceptance of UFP QAPP Monitoring Year 2	5 days	1/13/2012	1/19/2012
197	LF009 - AF acceptance of UFP QAPP Monitoring Year 3	5 days	1/4/2013	1/10/2013
198	LF009 - AF acceptance of UFP QAPP Monitoring Year 4	5 days	1/10/2014	1/16/2014
199	LF009 - AF acceptance of UFP QAPP Monitoring Year 5	5 days	1/16/2015	1/22/2015
200	**LF009 - LUC/IC Site Inspections**	**1033 days**	**12/16/2011**	**12/1/2015**
201	LF009 - Acceptance of LUC/IC Site Inspection Report 1	5 edays	12/16/2011	12/21/2011
202	LF009 - Acceptance of LUC/IC Site Inspection Report 2	81 days	12/12/2012	4/3/2013
203	LF009 - Acceptance of LUC/IC Site Inspection Report 3	5 days	11/27/2013	12/3/2013
204	LF009 - Acceptance of LUC/IC Site Inspection Report 4	5 days	11/26/2014	12/2/2014
205	LF009 - Acceptance of LUC/IC Site Inspection Report 5	5 days	11/25/2015	12/1/2015
206	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
207	Five-Year Review Report	50 days	1/12/2015	3/20/2015
208	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
209	**SD031 - Three Mile Creek (Closure Unrestricted Use)**	**1240 days**	**1/10/2011**	**10/9/2015**
210	SD031 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	1/10/2011	4/29/2011
211	SD031 - Prepare and Submit Design/plan remedy optimization	30 days	3/21/2011	4/29/2011
212	SD031 - Review and Acceptance of Design/Plan Remedy Optimization	44 days	5/2/2011	6/30/2011
213	**SD031 - Conduct Monitoring**	**1046 days**	**9/1/2011**	**9/3/2015**
214	SD031 - Conduct Monitoring, Prep and Submit CY11 LTM Report	63 days	9/1/2011	11/28/2011
215	SD031 - Review and Acceptance of CY11 LTM Report	24 days	11/29/2011	12/30/2011
216	SD031 - Conduct Monitoring, Prep and Submit CY12 LTM Report	95 days	8/31/2012	1/10/2013
217	SD031 - Review and Acceptance of CY12 LTM Report	24 days	1/11/2013	2/13/2013
218	SD031 - Conduct Monitoring, Prep and Submit CY13 LTM Report	60 days	9/3/2013	11/25/2013
219	SD031 - Review and Acceptance of CY13 LTM Report	26 days	11/26/2013	12/31/2013
220	SD031 - Conduct Monitoring, Prep and Submit CY14 LTM Report	61 days	9/3/2014	11/26/2014
221	SD031 - Review and Acceptance of CY14 LTM Report	27 days	11/27/2014	1/2/2015
222	SD031 - Conduct Monitoring, Prep and Submit CY15 LTM Report	62 days	5/11/2015	8/4/2015
223	SD031 - Review and Acceptance of CY15 LTM Report	22 days	8/5/2015	9/3/2015
224	SD031 - Achieve Site Closure	65 days	5/11/2015	8/7/2015
225	SD031 - Prepare and Submit Closure Report	22 days	8/10/2015	9/8/2015
226	SD031 - Review and Acceptance of Closure Report	23 days	9/9/2015	10/9/2015
227	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
228	Five-Year Review Report	50 days	1/12/2015	3/20/2015
229	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
230	**SD031 - UFP QAPP and UFP QAPP update**	**970 days**	**1/13/2012**	**10/1/2015**
231	SD031 - AF acceptance of UFP QAPP Monitoring Year 2	5 days	1/13/2012	1/19/2012
232	SD031 - AF acceptance of UFP QAPP Monitoring Year 3	65 days	1/4/2013	4/4/2013
233	SD031 - AF acceptance of UFP QAPP Monitoring Year 4	65 days	4/4/2014	7/3/2014
234	SD031 - AF acceptance of UFP QAPP Monitoring Year 5	65 days	7/3/2015	10/1/2015
235	**SD031 - RACR**	**119 days**	**8/31/2012**	**2/13/2013**
236	SD031 - Prep and Submit RACR	95 days	8/31/2012	1/10/2013
237	SD031 - Review and Acceptance of RACR	24 days	1/11/2013	2/13/2013
238	**SD032 - Six Mile Creek (Closure Unrestricted Use)**	**1240 days**	**1/10/2011**	**10/9/2015**
239	SD032 - Acceptance of Final UFP QAPP & H&S Plan by & NYSDEC	80 days	1/10/2011	4/29/2011
240	SD032 - Prepare and Submit Design/plan remedy optimization	30 days	3/21/2011	4/29/2011
241	SD032 - Review and Acceptance of Design/Plan Remedy Optimization	44 days	5/2/2011	6/30/2011
242	**SD032 - Conduct Monitoring**	**1046 days**	**9/1/2011**	**9/3/2015**
243	SD032 - Conduct Monitoring, Prep and Submit CY11 LTM Report	63 days	9/1/2011	11/28/2011
244	SD032 - Review and Acceptance of CY11 LTM Report	24 days	11/29/2011	12/30/2011
245	SD032 - Conduct Monitoring, Prep and Submit CY12 LTM Report	95 days	8/31/2012	1/10/2013
246	SD032 - Review and Acceptance of CY12 LTM Report	24 days	1/11/2013	2/13/2013
247	SD032 - Conduct Monitoring, Prep and Submit CY13 LTM Report	60 days	9/3/2013	11/25/2013
248	SD032 - Review and Acceptance of CY13 LTM Report	26 days	11/26/2013	12/31/2013
249	SD032 - Conduct Monitoring, Prep and Submit CY14 LTM Report	61 days	9/3/2014	11/26/2014
250	SD032 - Review and Acceptance of CY14 LTM Report	27 days	11/27/2014	1/2/2015
251	SD032 - Conduct Monitoring, Prep and Submit CY15 LTM Report	62 days	5/11/2015	8/4/2015
252	SD032 - Review and Acceptance of CY15 LTM Report	22 days	8/5/2015	9/3/2015
253	SD032 - Achieve Site Closure	65 days	5/11/2015	8/7/2015
254	SD032 - Prepare and Submit Closure Report	22 days	8/10/2015	9/8/2015
255	SD032 - Review and Acceptance of Closure Report	23 days	9/9/2015	10/9/2015
256	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
257	Five-Year Review Report	50 days	1/12/2015	3/20/2015
258	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
259	**SD032 - UFP QAPP and UFP QAPP update**	**970 days**	**1/13/2012**	**10/1/2015**
260	SD032 - AF acceptance of UFP QAPP Monitoring Year 2	5 days	1/13/2012	1/19/2012
261	SD032 - AF acceptance of UFP QAPP Monitoring Year 3	65 days	1/4/2013	4/4/2013
262	SD032 - AF acceptance of UFP QAPP Monitoring Year 4	65 days	4/4/2014	7/3/2014
263	SD032 - AF acceptance of UFP QAPP Monitoring Year 5	65 days	7/3/2015	10/1/2015
264	**SD032 - RACR**	**119 days**	**8/31/2012**	**2/13/2013**
265	SD032 - Prep and Submit RACR	95 days	8/31/2012	1/10/2013
266	SD032 - Review and Acceptance of RACR	24 days	1/11/2013	2/13/2013
267	**SD052 - SVI Systems (Optimized Exit Strategy)**	**1299 days**	**1/10/2011**	**12/31/2015**
268	**SD052 - Field Work**	**1299 days**	**1/10/2011**	**12/31/2015**
269	SD052 - O&M and Monitoring Year 1	355 edays	1/10/2011	12/31/2011
270	SD052 - O&M and Monitoring Year 2	364 edays	1/2/2012	12/31/2012
271	SD052 - O&M and Monitoring Year 3	364 edays	1/1/2013	12/31/2013
272	SD052 - O&M and Monitoring Year 4	364 edays	1/1/2014	12/31/2014
273	SD052 - O&M and Monitoring Year 5	364 edays	1/1/2015	12/31/2015
274	**SD052 - Reporting**	**1035 days**	**1/2/2012**	**12/18/2015**
275	SD052 - O&M and Quarterly Monitoring Report - Year 1 Q4	45 days	1/2/2012	3/2/2012
276	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 1 Q4	0 days	3/2/2012	3/2/2012
277	SD052 - O&M and Quarterly Monitoring Report - Year 2 Q1	383 edays	3/13/2012	3/31/2013
278	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 2 Q1	0 days	3/31/2013	3/31/2013
279	SD052 - O&M and Quarterly Monitoring Report - Year 2 Q2	293 edays	6/11/2012	3/31/2013
280	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 2 Q2	0 days	3/31/2013	3/31/2013
281	SD052 - O&M and Quarterly Monitoring Report - Year 2 Q3	280 edays	1/18/2013	10/25/2013



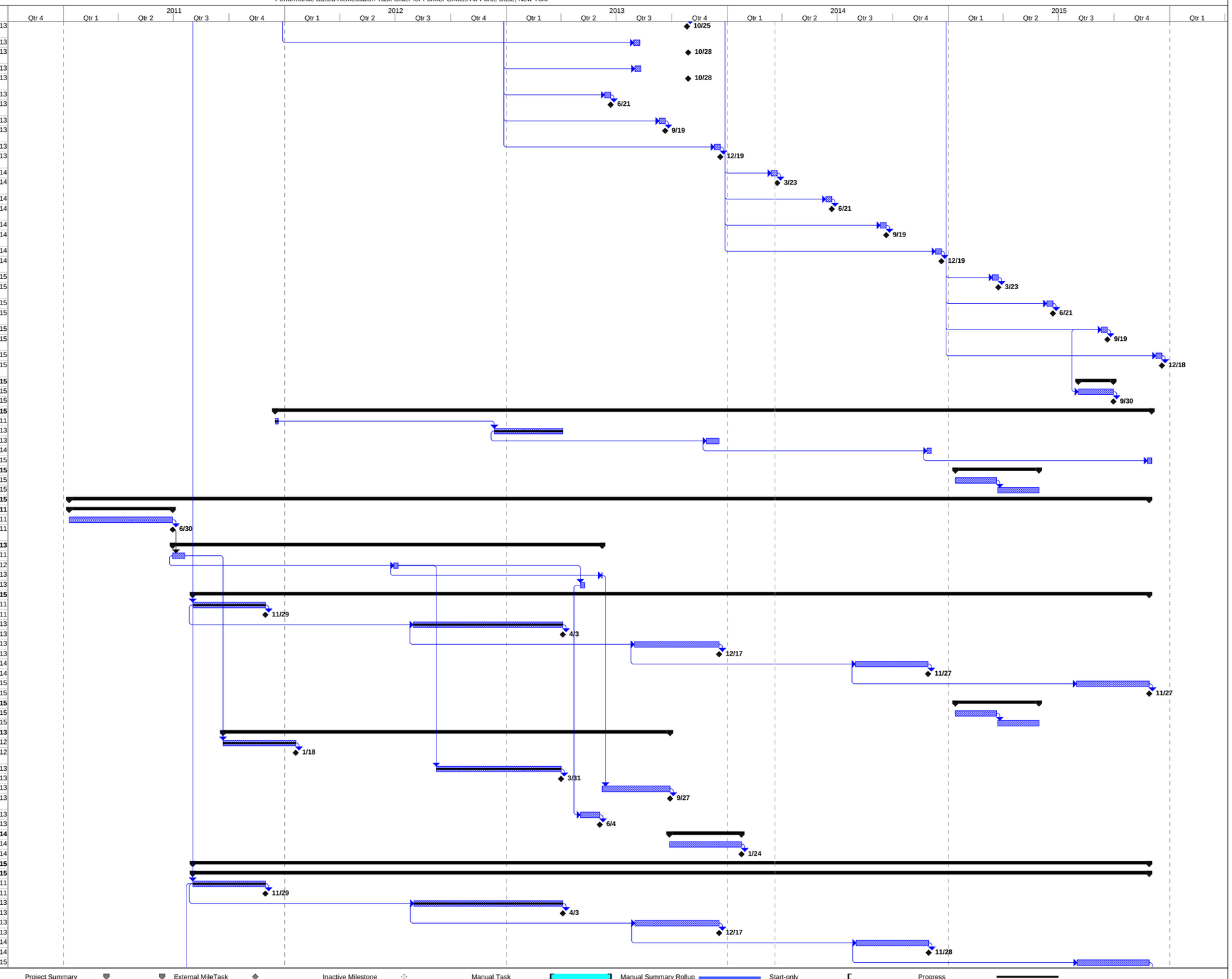
Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York

ID	Task Name	Duration	Start	Finish
282	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 2 Q3	0 days	10/25/2013	10/25/2013
283	SD052 - O&M and Quarterly Monitoring Report - Year 2 Q4	10 edays	7/29/2013	8/8/2013
284	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 2 Q4	0 days	10/28/2013	10/28/2013
285	SD052 - O&M and Quarterly Monitoring Report - Year 3 Q1	10 edays	7/31/2013	8/10/2013
286	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 3 Q1	0 days	10/28/2013	10/28/2013
287	SD052 - O&M and Quarterly Monitoring Report - Year 3 Q2	10 edays	6/11/2013	6/21/2013
288	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 3 Q2	0 days	6/21/2013	6/21/2013
289	SD052 - O&M and Quarterly Monitoring Report - Year 3 Q3	10 edays	9/9/2013	9/19/2013
290	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 3 Q3	0 days	9/19/2013	9/19/2013
291	SD052 - O&M and Quarterly Monitoring Report - Year 3 Q4	10 edays	12/9/2013	12/19/2013
292	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 3 Q4	0 days	12/19/2013	12/19/2013
293	SD052 - O&M and Quarterly Monitoring Report - Year 4 Q1	10 edays	3/13/2014	3/23/2014
294	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 4 Q1	0 days	3/23/2014	3/23/2014
295	SD052 - O&M and Quarterly Monitoring Report - Year 4 Q2	10 edays	6/11/2014	6/21/2014
296	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 4 Q2	0 days	6/21/2014	6/21/2014
297	SD052 - O&M and Quarterly Monitoring Report - Year 4 Q3	10 edays	9/9/2014	9/19/2014
298	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 4 Q3	0 days	9/19/2014	9/19/2014
299	SD052 - O&M and Quarterly Monitoring Report - Year 4 Q4	10 edays	12/9/2014	12/19/2014
300	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 4 Q4	0 days	12/19/2014	12/19/2014
301	SD052 - O&M and Quarterly Monitoring Report - Year 5 Q1	10 edays	3/13/2015	3/23/2015
302	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 5 Q1	0 days	3/23/2015	3/23/2015
303	SD052 - O&M and Quarterly Monitoring Report - Year 5 Q2	10 edays	6/11/2015	6/21/2015
304	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 5 Q2	0 days	6/21/2015	6/21/2015
305	SD052 - O&M and Quarterly Monitoring Report - Year 5 Q3	10 edays	9/9/2015	9/19/2015
306	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 5 Q3	0 days	9/19/2015	9/19/2015
307	SD052 - O&M and Quarterly Monitoring Report - Year 5 Q4	10 edays	12/8/2015	12/18/2015
308	SD052 - Acceptance of O&M and Quarterly Monitoring Report - Year 5 Q4	0 days	12/18/2015	12/18/2015
309	**SD052 - Optimized Exit Strategy**	**42 days**	**8/3/2015**	**9/30/2015**
310	SD052 - Optimized Exit Strategy Document	58 edays	8/3/2015	9/30/2015
311	SD052 - Acceptance of Optimized Exit Strategy Document	0 days	9/30/2015	9/30/2015
312	**SD052 - LUC/IC Site Inspections**	**1033 days**	**12/16/2011**	**12/1/2015**
313	SD052 - Acceptance of LUC/IC Site Inspection Report 1	5 edays	12/16/2011	12/21/2011
314	SD052 - Acceptance of LUC/IC Site Inspection Report 2	81 days	12/12/2012	4/3/2013
315	SD052 - Acceptance of LUC/IC Site Inspection Report 3	15 days	11/27/2013	12/17/2013
316	SD052 - Acceptance of LUC/IC Site Inspection Report 4	5 days	11/26/2014	12/2/2014
317	SD052 - Acceptance of LUC/IC Site Inspection Report 5	5 days	11/25/2015	12/1/2015
318	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
319	Five-Year Review Report	50 days	1/12/2015	3/20/2015
320	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
321	**SS060 - STW-1300 (Bldg. 35/36) (Closure with Restrictions)**	**1275 days**	**1/10/2011**	**11/27/2015**
322	**SS060 - Planning Documents**	**123 days**	**1/10/2011**	**6/30/2011**
323	SS060 - Design and Remedy Optimization Documents	171 edays	1/10/2011	6/30/2011
324	SS060 - Acceptance of Final Design and Remedy Optimization Documents	0 days	6/30/2011	6/30/2011
325	**SS060 - Field Work**	**507 days**	**6/30/2011**	**6/7/2013**
326	SS060 - Remedy Implementation and Monitoring Year 1	20 edays	6/30/2011	7/20/2011
327	SS060 - Monitoring Year 2	5 days	6/29/2012	7/5/2012
328	SS060 - Monitoring Year 3	1 day	6/7/2013	6/7/2013
329	SS060 - Well Abandonment	5 days	5/3/2013	5/9/2013
330	**SS060 - LUC/IC Inspection**	**1129 days**	**8/2/2011**	**11/27/2015**
331	SS060 - LUC/IC Inspection Report - Year 1	86 days	8/2/2011	11/29/2011
332	SS060 - Acceptance of LUC/IC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
333	SS060 - LUC/IC Inspection Report - Year 2	177 days	7/31/2012	4/3/2013
334	SS060 - Acceptance of LUC/IC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
335	SS060 - LUC/IC Inspection Report - Year 3	100 days	7/31/2013	12/17/2013
336	SS060 - Acceptance of LUC/IC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
337	SS060 - LUC/IC Inspection Report - Year 4	86 days	7/31/2014	11/27/2014
338	SS060 - Acceptance of LUC/IC Inspection Report - Year 4	0 days	11/27/2014	11/27/2014
339	SS060 - LUC/IC Inspection Report - Year 5	86 days	7/31/2015	11/27/2015
340	SS060 - Acceptance of LUC/IC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
341	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
342	Five-Year Review Report	50 days	1/12/2015	3/20/2015
343	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
344	**SS060 - Reporting**	**528 days**	**9/20/2011**	**9/27/2013**
345	SS060 - Remedy Implementation and Annual Monitoring Report - Year 1	120 edays	9/20/2011	1/18/2012
346	SS060 - Acceptance of Remedy Implementation and Annual Monitoring Report - Year 1	0 days	1/18/2012	1/18/2012
347	SS060 - Annual Monitoring Report - Year 2	206 edays	9/6/2012	3/31/2013
348	SS060 - Acceptance of Annual Monitoring Report - Year 2	0 days	3/31/2013	3/31/2013
349	SS060 - Annual Monitoring Report - Year 3	112 edays	6/7/2013	9/27/2013
350	SS060 - Acceptance of Site Closure and Annual Monitoring Report - Year 3	0 days	9/27/2013	9/27/2013
351	SS060 - Well Abandonment Report	32 edays	5/3/2013	6/4/2013
352	SS060 - Acceptance of Well Abandonment Report	0 days	6/4/2013	6/4/2013
353	**SS060 - Site Closure**	**85 days**	**9/27/2013**	**1/24/2014**
354	SS060 - Site Closure Document	119 edays	9/27/2013	1/24/2014
355	SS060 - Acceptance of Site Closure Document	0 days	1/24/2014	1/24/2014
356	**ST006 - Building 101 (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
357	**ST006 - LUC/IC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
358	ST006 - LUC/IC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
359	ST006 - Acceptance of LUC/IC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
360	ST006 - LUC/IC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
361	ST006 - Acceptance of LUC/IC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
362	ST006 - LUC/IC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
363	ST006 - Acceptance of LUC/IC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
364	ST006 - LUC/IC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
365	ST006 - Acceptance of LUC/IC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
366	ST006 - LUC/IC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015



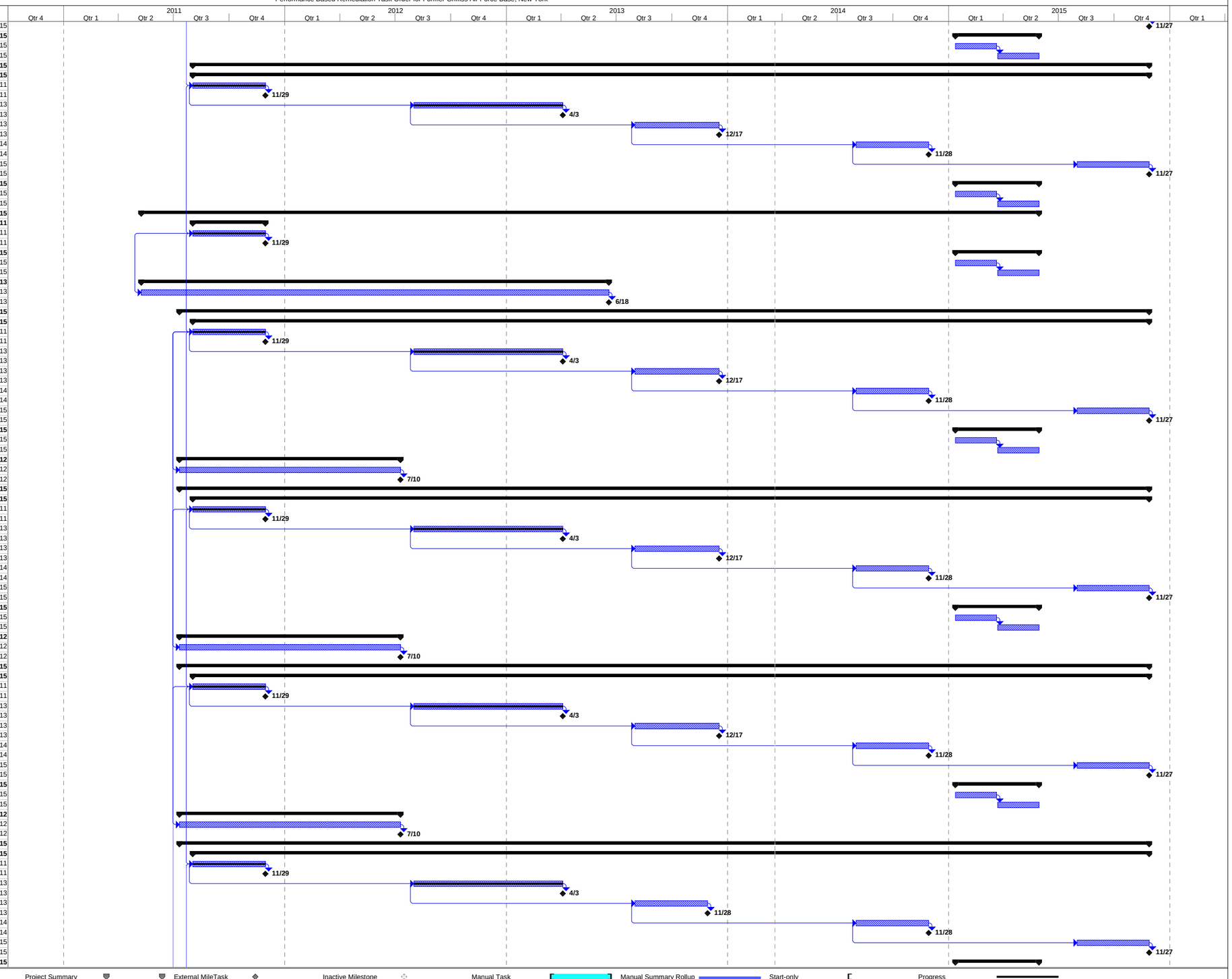
Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York

ID	Task Name	Duration	Start	Finish
367	ST006 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
368	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
369	Five-Year Review Report	50 days	1/12/2015	3/20/2015
370	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
371	**SS008 - Building 112 (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
372	**SS008 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
373	SS008 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
374	SS008 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
375	SS008 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
376	SS008 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
377	SS008 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
378	SS008 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
379	SS008 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
380	SS008 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
381	SS008 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
382	SS008 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
383	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
384	Five-Year Review Report	50 days	1/12/2015	3/20/2015
385	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
386	**DP011 - Building 3 (Closure with no Restrictions)**	**1060 days**	**5/9/2011**	**5/29/2015**
387	**DP011 - LUCIC Inspection**	**86 days**	**8/1/2011**	**11/29/2011**
388	DP011 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
389	DP011 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
390	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
391	Five-Year Review Report	50 days	1/12/2015	3/20/2015
392	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
393	**DP011 - Removal of LUCIC**	**552 days**	**5/9/2011**	**6/18/2013**
394	DP011 - Soil and Groundwater Sampling/Reporting	552 days	5/9/2011	6/18/2013
395	DP011 - Acceptance of LUCIC Removal	0 days	6/18/2013	6/18/2013
396	**DP012 - Building 301 (Optimized Exit Strategy)**	**1145 days**	**7/11/2011**	**11/27/2015**
397	**DP012 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
398	DP012 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
399	DP012 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
400	DP012 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
401	DP012 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
402	DP012 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
403	DP012 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
404	DP012 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
405	DP012 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
406	DP012 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
407	DP012 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
408	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
409	Five-Year Review Report	50 days	1/12/2015	3/20/2015
410	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
411	**DP012 - Removal of Groundwater LUCIC**	**261 days**	**7/11/2011**	**7/10/2012**
412	DP012 - Groundwater LUCIC Removal Documentation Report	365 edays	7/11/2011	7/10/2012
413	DP012 - Acceptance of LUCIC Removal	0 days	7/10/2012	7/10/2012
414	**DP013 - Building 255 (Optimized Exit Strategy)**	**1145 days**	**7/11/2011**	**11/27/2015**
415	**DP013 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
416	DP013 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
417	DP013 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
418	DP013 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
419	DP013 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
420	DP013 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
421	DP013 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
422	DP013 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
423	DP013 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
424	DP013 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
425	DP013 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
426	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
427	Five-Year Review Report	50 days	1/12/2015	3/20/2015
428	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
429	**DP013 - Removal of Groundwater LUCIC**	**261 days**	**7/11/2011**	**7/10/2012**
430	DP013 - Groundwater LUCIC Removal Documentation Report	365 edays	7/11/2011	7/10/2012
431	DP013 - Acceptance of LUCIC Removal	0 days	7/10/2012	7/10/2012
432	**DP015 - Building 219 (Optimized Exit Strategy)**	**1145 days**	**7/11/2011**	**11/27/2015**
433	**DP015 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
434	DP015 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
435	DP015 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
436	DP015 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
437	DP015 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
438	DP015 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
439	DP015 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
440	DP015 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
441	DP015 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
442	DP015 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
443	DP015 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
444	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
445	Five-Year Review Report	50 days	1/12/2015	3/20/2015
446	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
447	**DP015 - Removal of Groundwater LUCIC**	**261 days**	**7/11/2011**	**7/10/2012**
448	DP015 - Groundwater LUCIC Removal Documentation Report	365 edays	7/11/2011	7/10/2012
449	DP015 - Acceptance of LUCIC Removal	0 days	7/10/2012	7/10/2012
450	**SS017 - Lot 69 (Optimized Exit Strategy)**	**1145 days**	**7/11/2011**	**11/27/2015**
451	**SS017 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
452	SS017 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
453	SS017 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
454	SS017 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
455	SS017 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
456	SS017 - LUCIC Inspection Report - Year 3	120 edays	7/31/2013	11/28/2013
457	SS017 - Acceptance of LUCIC Inspection Report	0 days	11/28/2013	11/28/2013
458	SS017 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
459	SS017 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
460	SS017 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
461	SS017 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
462	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**



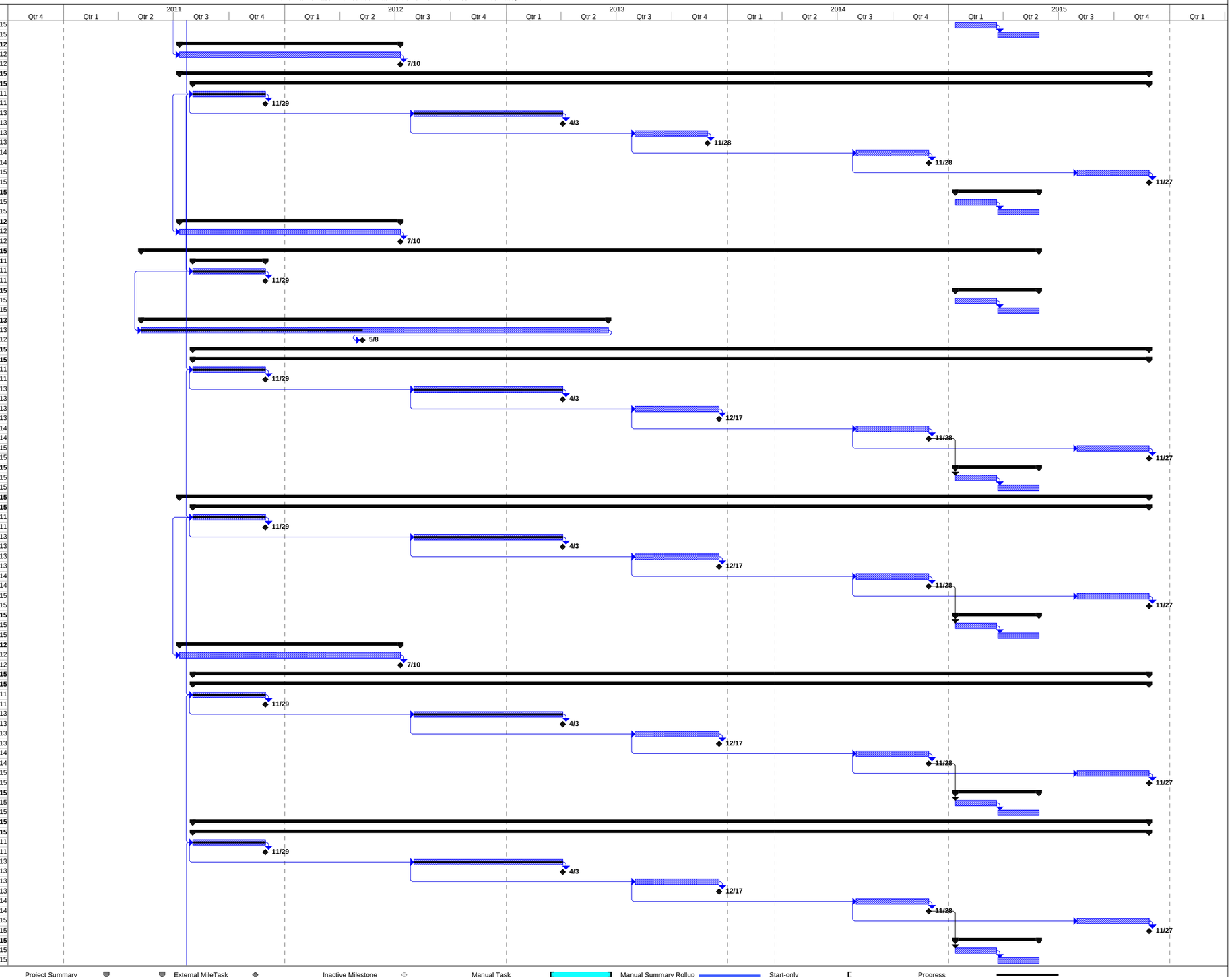
Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York

ID	Task Name	Duration	Start	Finish
463	Five-Year Review Report	50 days	1/12/2015	3/20/2015
464	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
465	**SS017 - Removal of Groundwater LUCIC**	**261 days**	**7/11/2011**	**7/10/2012**
466	SS017 - Groundwater LUCIC Removal Documentation Report	365 edays	7/11/2011	7/10/2012
467	SS017 - Acceptance of LUCIC Removal	0 days	7/10/2012	7/10/2012
468	**DP022 - Building 222 (Optimized Exit Strategy)**	**1145 days**	**7/11/2011**	**11/27/2015**
469	**DP022 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
470	DP022 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
471	DP022 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
472	DP022 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
473	DP022 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
474	DP022 - LUCIC Inspection Report - Year 3	120 edays	7/31/2013	11/28/2013
475	DP022 - Acceptance of LUCIC Inspection Report - Year 3	0 days	11/28/2013	11/28/2013
476	DP022 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
477	DP022 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
478	DP022 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
479	DP022 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
480	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
481	Five-Year Review Report	50 days	1/12/2015	3/20/2015
482	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
483	**DP022 - Removal of Groundwater LUCIC**	**261 days**	**7/11/2011**	**7/10/2012**
484	DP022 - Groundwater LUCIC Removal Documentation Report	365 edays	7/11/2011	7/10/2012
485	DP022 - Acceptance of LUCIC Removal	0 days	7/10/2012	7/10/2012
486	**SS023 - Building 20 (Closure with no Restrictions)**	**1060 days**	**5/9/2011**	**5/29/2015**
487	**SS023 - LUCIC Inspection**	**86 days**	**8/1/2011**	**11/29/2011**
488	SS023 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
489	SS023 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
490	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
491	Five-Year Review Report	50 days	1/12/2015	3/20/2015
492	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
493	**SS023 - Removal of LUCIC**	**551 days**	**5/9/2011**	**6/18/2013**
494	SS023 - Soil and Groundwater Sampling/Reporting	771 edays	5/9/2011	6/18/2013
495	SS023 - Acceptance of LUCIC Removal	0 days	5/8/2012	5/8/2012
496	**SS024 - Fire Demonstration Area (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
497	**SS024 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
498	SS024 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
499	SS024 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
500	SS024 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
501	SS024 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
502	SS024 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
503	SS024 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
504	SS024 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
505	SS024 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
506	SS024 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
507	SS024 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
508	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
509	Five-Year Review Report	50 days	1/12/2015	3/20/2015
510	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
511	**SS025 - Site T-9 (Optimized Exit Strategy)**	**1145 days**	**7/11/2011**	**11/27/2015**
512	**SS025 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
513	SS025 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
514	SS025 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
515	SS025 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
516	SS025 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
517	SS025 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
518	SS025 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
519	SS025 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
520	SS025 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
521	SS025 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
522	SS025 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
523	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
524	Five-Year Review Report	50 days	1/12/2015	3/20/2015
525	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
526	**SS025 - Removal of Groundwater LUCIC**	**261 days**	**7/11/2011**	**7/10/2012**
527	SS025 - Groundwater LUCIC Removal Documentation Report	365 edays	7/11/2011	7/10/2012
528	SS025 - Acceptance of LUCIC Removal	0 days	7/10/2012	7/10/2012
529	**FT030 - Fire Protection Training Area (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
530	**FT030 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
531	FT030 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
532	FT030 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
533	FT030 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
534	FT030 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
535	FT030 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
536	FT030 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
537	FT030 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
538	FT030 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
539	FT030 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
540	FT030 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
541	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
542	Five-Year Review Report	50 days	1/12/2015	3/20/2015
543	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
544	**SS033 - Coal Storage Yard Area (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
545	**SS033 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
546	SS033 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
547	SS033 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
548	SS033 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
549	SS033 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
550	SS033 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
551	SS033 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
552	SS033 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
553	SS033 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
554	SS033 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
555	SS033 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
556	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
557	Five-Year Review Report	50 days	1/12/2015	3/20/2015
558	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015



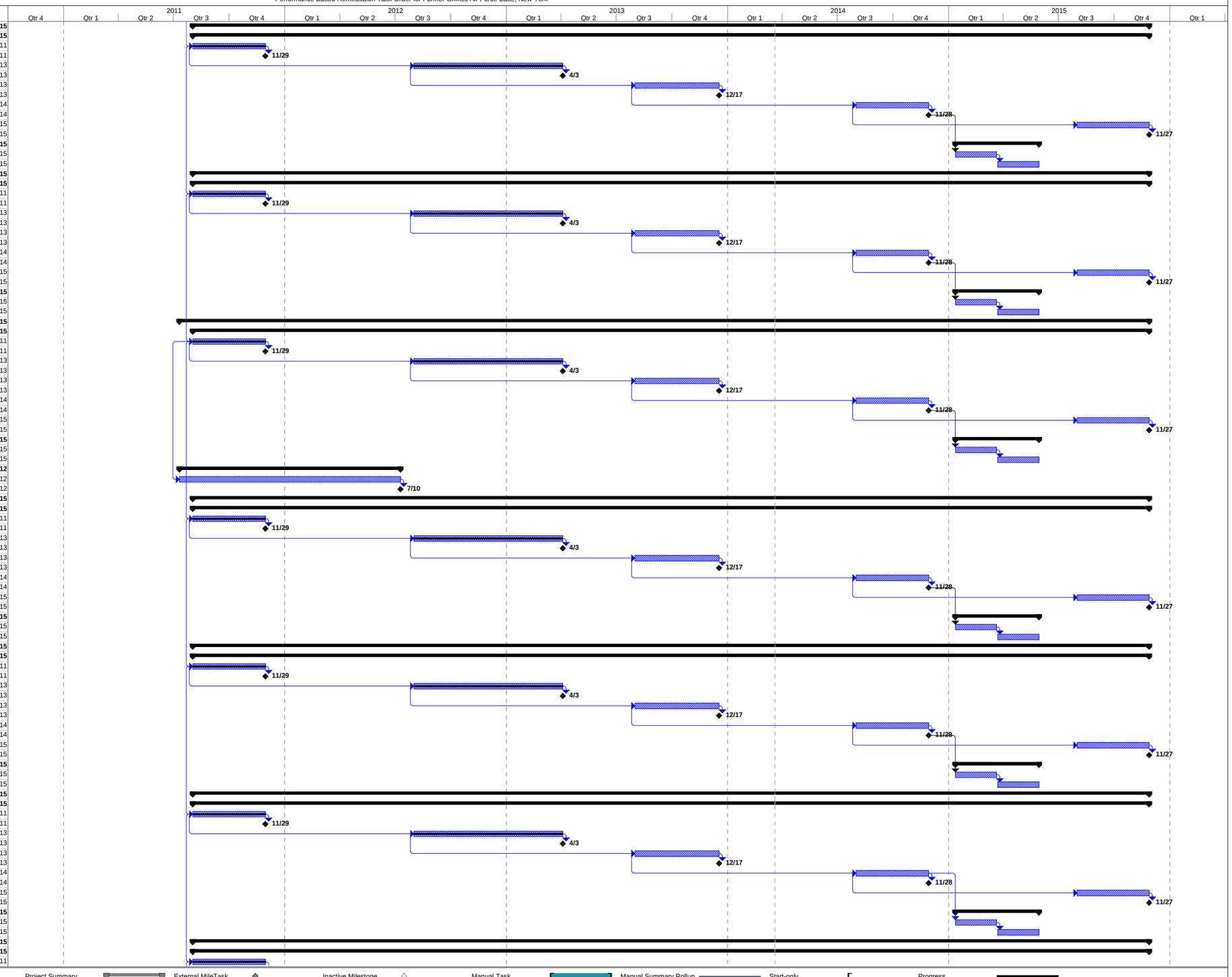
Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York

ID	Task Name	Duration	Start	Finish
559	**ST036 - Building 110 (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
560	**ST036 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
561	ST036 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
562	ST036 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
563	ST036 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
564	ST036 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
565	ST036 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
566	ST036 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
567	ST036 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
568	ST036 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
569	ST036 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
570	ST036 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
571	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
572	Five-Year Review Report	50 days	1/12/2015	3/20/2015
573	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
574	**SS044 - Electrical Power Substation (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
575	**SS044 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
576	SS044 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
577	SS044 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
578	SS044 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
579	SS044 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
580	SS044 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
581	SS044 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
582	SS044 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
583	SS044 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
584	SS044 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
585	SS044 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
586	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
587	Five-Year Review Report	50 days	1/12/2015	3/20/2015
588	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
589	**SD050 - Building 214 (Optimized Exit Strategy)**	**1145 days**	**7/11/2011**	**11/27/2015**
590	**SD050 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
591	SD050 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
592	SD050 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
593	SD050 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
594	SD050 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
595	SD050 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
596	SD050 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
597	SD050 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
598	SD050 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
599	SD050 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
600	SD050 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
601	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
602	Five-Year Review Report	50 days	1/12/2015	3/20/2015
603	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
604	**SD050 - Removal of Groundwater LUCIC**	**261 days**	**7/11/2011**	**7/10/2012**
605	SD050 - Groundwater LUCIC Removal Documentation Report	365 edays	7/11/2011	7/10/2012
606	SD050 - Acceptance of LUCIC Removal	0 days	7/10/2012	7/10/2012
607	**SD52-01 - Apron 2 Chlorinated Plume (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
608	**SD52-01 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
609	SD52-01 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
610	SD52-01 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
611	SD52-01 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
612	SD52-01 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
613	SD52-01 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
614	SD52-01 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
615	SD52-01 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
616	SD52-01 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
617	SD52-01 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
618	SD52-01 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
619	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
620	Five-Year Review Report	50 days	1/12/2015	3/20/2015
621	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
622	**SD52-02 - Building 775 Chlorinated Plume (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
623	**SD52-02 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
624	SD52-02 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
625	SD52-02 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
626	SD52-02 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
627	SD52-02 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
628	SD52-02 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
629	SD52-02 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
630	SD52-02 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
631	SD52-02 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
632	SD52-02 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
633	SD52-02 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
634	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
635	Five-Year Review Report	50 days	1/12/2015	3/20/2015
636	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
637	**SD52-04 - Landfilll 6 Chlorinated Plume (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
638	**SD52-04 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
639	SD52-04 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
640	SD52-04 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
641	SD52-04 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
642	SD52-04 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
643	SD52-04 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
644	SD52-04 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
645	SD52-04 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
646	SD52-04 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
647	SD52-04 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
648	SD52-04 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
649	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
650	Five-Year Review Report	50 days	1/12/2015	3/20/2015
651	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
652	**SD52-05 - Building 817 Chlorinated Plume (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
653	**SD52-05 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
654	SD52-05 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011

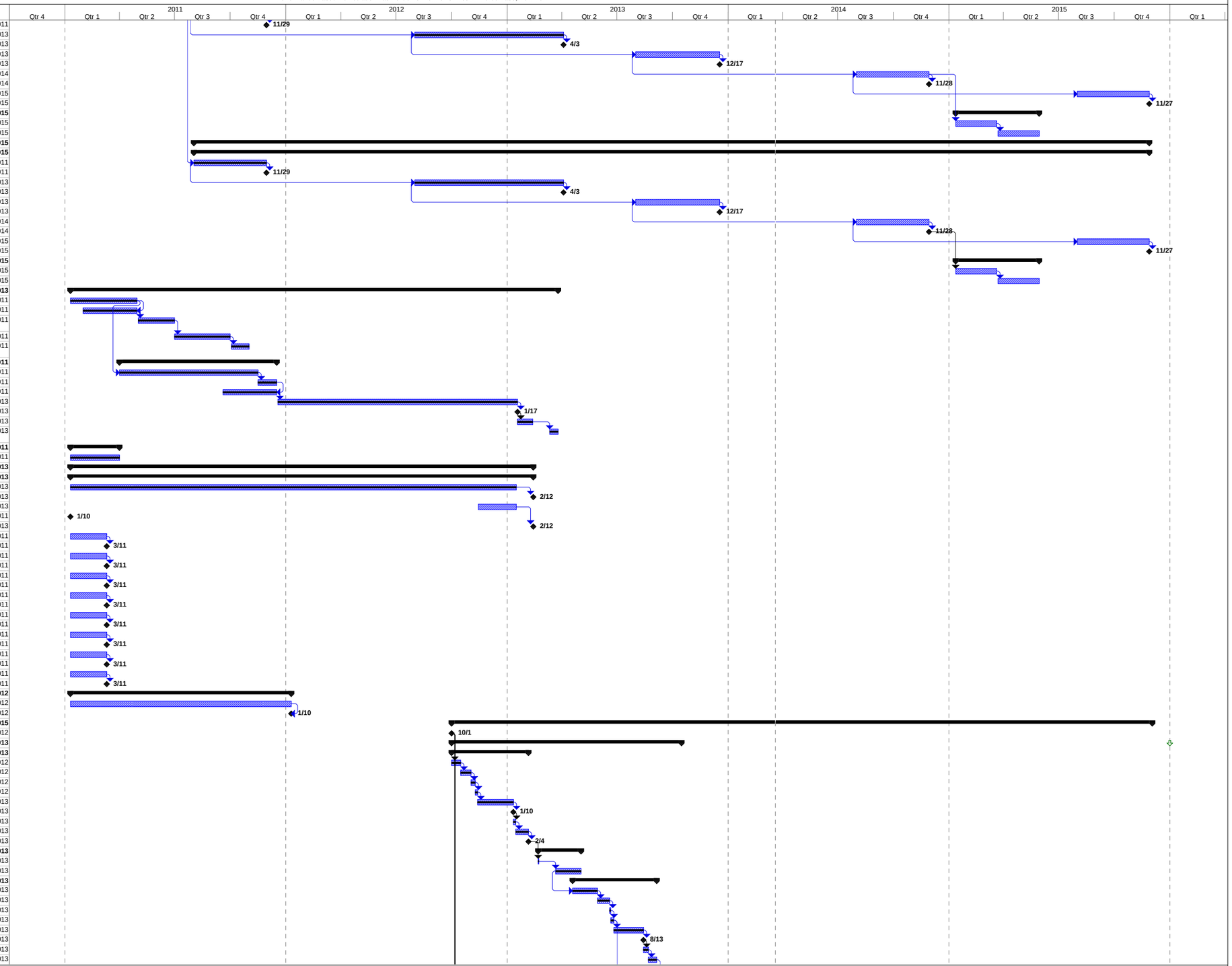


Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

ID	Task Name	Duration	Start	Finish
655	SD52-05 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
656	SD52-05 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
657	SD52-05 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
658	SD52-05 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
659	SD52-05 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
660	SD52-05 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
661	SD52-05 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
662	SD52-05 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
663	SD52-05 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
664	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
665	Five-Year Review Report	50 days	1/12/2015	3/20/2015
666	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
667	**SS062 - AOC-9 (Status Quo)**	**1129 days**	**8/1/2011**	**11/27/2015**
668	**SS062 - LUCIC Inspection**	**1129 days**	**8/1/2011**	**11/27/2015**
669	SS062 - LUCIC Inspection Report - Year 1	120 edays	8/1/2011	11/29/2011
670	SS062 - Acceptance of LUCIC Inspection Report - Year 1	0 days	11/29/2011	11/29/2011
671	SS062 - LUCIC Inspection Report - Year 2	176 days	8/1/2012	4/3/2013
672	SS062 - Acceptance of LUCIC Inspection Report - Year 2	0 days	4/3/2013	4/3/2013
673	SS062 - LUCIC Inspection Report - Year 3	139 edays	7/31/2013	12/17/2013
674	SS062 - Acceptance of LUCIC Inspection Report - Year 3	0 days	12/17/2013	12/17/2013
675	SS062 - LUCIC Inspection Report - Year 4	120 edays	7/31/2014	11/28/2014
676	SS062 - Acceptance of LUCIC Inspection Report - Year 4	0 days	11/28/2014	11/28/2014
677	SS062 - LUCIC Inspection Report - Year 5	119 edays	7/31/2015	11/27/2015
678	SS062 - Acceptance of LUCIC Inspection Report - Year 5	0 days	11/27/2015	11/27/2015
679	**Five-Year Review -**	**100 days**	**1/12/2015**	**5/29/2015**
680	Five-Year Review Report	50 days	1/12/2015	3/20/2015
681	Acceptance of Five-Year Review Report	50 days	3/23/2015	5/29/2015
682	**SS070 - Building 150 (Closure Unrestricted Use)**	**576 days**	**1/10/2011**	**3/25/2013**
683	SS070 - Acceptance of Final UFP QAPP & H&S Plan by AFCEE & NYSDEC	80 days	1/10/2011	4/29/2011
684	SS070 - Prepare and Submit Design/plan remedy optimization	65 days	1/31/2011	4/29/2011
685	SS070 - AFCEE Review and Acceptance of Design/Plan Remedy Optimization	44 days	5/2/2011	6/30/2011
686	SS070 - Implement Remedy Optimization Systems and Submit Report	66 days	7/1/2011	9/30/2011
687	SS070 - AFCEE Review and Acceptance of Optimization System and Report	21 days	10/3/2011	10/31/2011
688	**SS070 - Conduct Monitoring**	**186 days**	**4/1/2011**	**12/16/2011**
689	SS070 - Conduct Monitoring, Prep and Submit CY11 LTM Report	163 days	4/1/2011	11/15/2011
690	SS070 - AFCEE Review and Acceptance of CY11 LTM Report	23 days	11/16/2011	12/16/2011
691	SS070 - Achieve Site Closure	65 days	9/19/2011	12/16/2011
692	SS070 - Prepare and Submit Closure Report	284 days	12/19/2011	1/17/2013
693	SS070 - AFCEE Review and Acceptance of Closure Report	0 days	1/17/2013	1/17/2013
694	SS070 - NYSDEC Closure of Spill Number	17 days	1/18/2013	2/11/2013
695	SS070 - Wells Decommissioned (Acceptance of Well Abandonment Report)	10 days	3/12/2013	3/25/2013
696	**ST037 - Pumphouse 5 (Closure Unrestricted Use)**	**59 days**	**1/10/2011**	**3/31/2011**
697	ST037 - Acceptance of Final Site Closure Report by AFCEE & NYSDEC	59 days	1/10/2011	3/31/2011
698	**Unknown Sites**	**547 days**	**1/10/2011**	**2/12/2013**
699	**Investigate 10 Unknown Sites**	**547 days**	**1/10/2011**	**2/12/2013**
700	Investigation Report - Site 1	527 days	1/10/2011	1/15/2013
701	Acceptance of Investigation Report - Site 1	0 days	2/12/2013	2/12/2013
702	Investigation Report - Site 2	45 days	11/14/2012	1/15/2013
703	Acceptance of Investigation Report - Site 2	0 days	1/10/2011	1/10/2011
704	Acceptance of Investigation Report - Site 2	0 days	2/12/2013	2/12/2013
705	Investigation Report - Site 3	60 edays	1/10/2011	3/11/2011
706	Acceptance of Investigation Report - Site 3	0 days	3/11/2011	3/11/2011
707	Investigation Report - Site 4	60 edays	1/10/2011	3/11/2011
708	Acceptance of Investigation Report - Site 4	0 days	3/11/2011	3/11/2011
709	Investigation Report - Site 5	60 edays	1/10/2011	3/11/2011
710	Acceptance of Investigation Report - Site 5	0 days	3/11/2011	3/11/2011
711	Investigation Report - Site 6	60 edays	1/10/2011	3/11/2011
712	Acceptance of Investigation Report - Site 6	0 days	3/11/2011	3/11/2011
713	Investigation Report - Site 7	60 edays	1/10/2011	3/11/2011
714	Acceptance of Investigation Report - Site 7	0 days	3/11/2011	3/11/2011
715	Investigation Report - Site 8	60 edays	1/10/2011	3/11/2011
716	Acceptance of Investigation Report - Site 8	0 days	3/11/2011	3/11/2011
717	Investigation Report - Site 9	60 edays	1/10/2011	3/11/2011
718	Acceptance of Investigation Report - Site 9	0 days	3/11/2011	3/11/2011
719	Investigation Report - Site 10	60 edays	1/10/2011	3/11/2011
720	Acceptance of Investigation Report - Site 10	0 days	3/11/2011	3/11/2011
721	**Five-Year Review - Site Specific with Costs**	**261 days**	**1/10/2011**	**1/10/2012**
722	Five-Year Review Report	365 edays	1/10/2011	1/10/2012
723	Acceptance of Five-Year Review Report	0 days	1/10/2012	1/10/2012
724	**Griffiss MOD 2 IMS**	**828 days**	**10/1/2012**	**12/2/2015**
725	NTP	0 days	10/1/2012	10/1/2012
726	**ST006 (Building 101) - Conduct SVI Evaluation and Achieve SC**	**272 days**	**10/1/2012**	**10/15/2013**
727	**SVI Evaluation Work Plan**	**91 days**	**10/1/2012**	**2/4/2013**
728	Prepare and Submit Internal Draft	11 days	10/1/2012	10/15/2012
729	AF Review of Internal Draft/Provide Comments	13 days	10/16/2012	11/1/2012
730	Respond to AF Comments	5 days	11/2/2012	11/8/2012
731	Prepare and Submit Draft	2 days	11/9/2012	11/12/2012
732	Regulator Review of Draft /Provide Comments	43 days	11/13/2012	1/10/2013
733	Regulatory Response to Draft	0 days	1/10/2013	1/10/2013
734	Respond to Regulator Comments on Draft and Prepare Final	2 days	1/11/2013	1/14/2013
735	Regulatory and Air Force Review of Final	15 days	1/15/2013	2/4/2013
736	Regulatory Approval of Final	0 days	2/4/2013	2/4/2013
737	**SVI Evaluation**	**51 days**	**2/21/2013**	**5/2/2013**
738	SVI Sampling	1 day	2/21/2013	2/21/2013
739	Human Health Risk Assessment	30 days	3/22/2013	5/2/2013
740	**SVI Evaluation Report**	**99 days**	**4/19/2013**	**9/4/2013**
741	Prepare and Submit Internal Draft	29 days	4/19/2013	5/29/2013
742	AF Review of Internal Draft/Provide Comments	14 days	5/30/2013	6/18/2013
743	Respond to AF Comments	2 days	6/19/2013	6/20/2013
744	Prepare and Submit Draft	3 days	6/21/2013	6/25/2013
745	Regulator Review of Draft /Provide Comments	35 days	6/26/2013	8/13/2013
746	Regulatory Response to Draft	0 days	8/13/2013	8/13/2013
747	Respond to Regulator Comments on Draft and Prepare Final	6 days	8/14/2013	8/21/2013
748	Regulatory and Air Force Review of Final	10 days	8/22/2013	9/4/2013

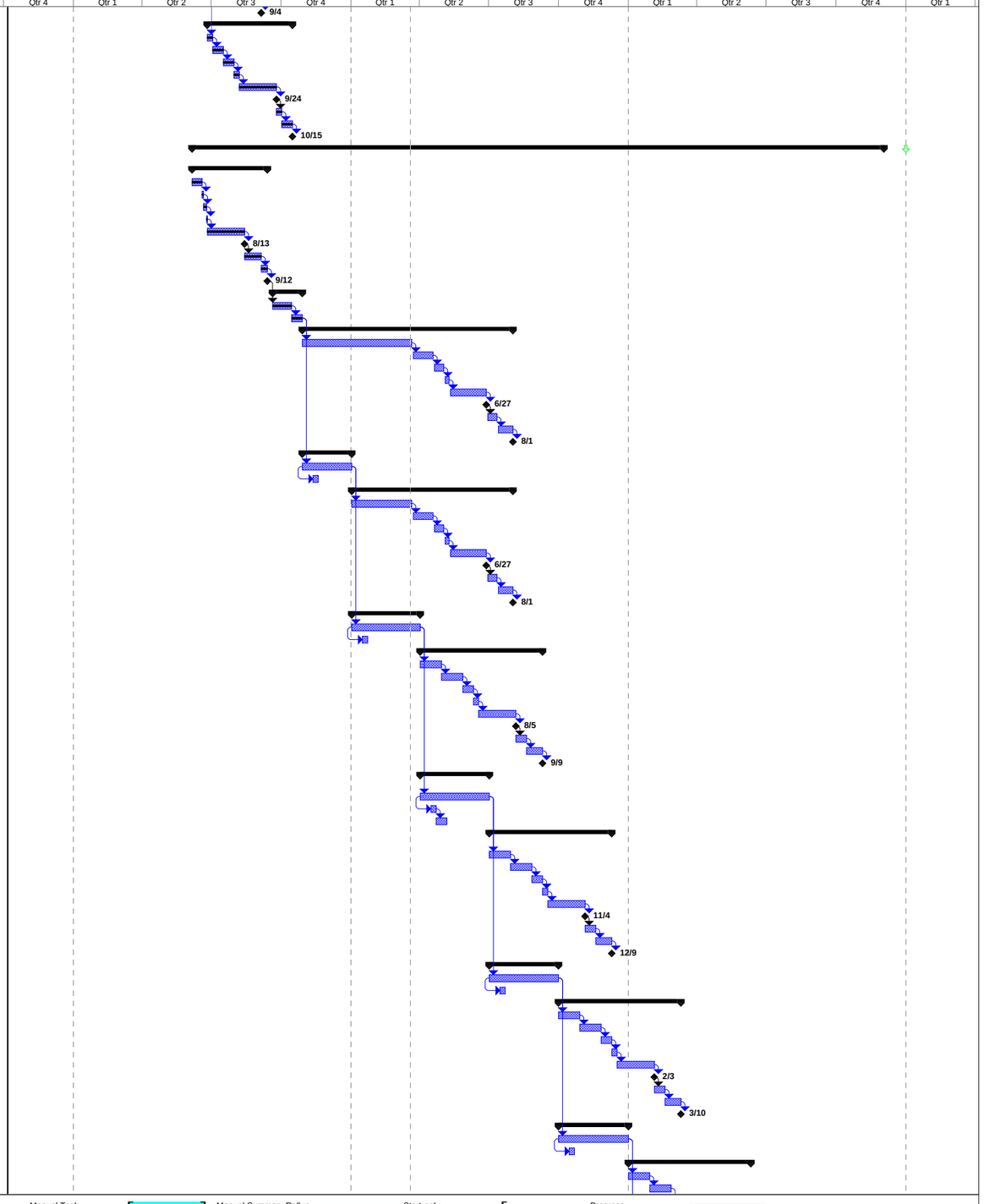


Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

ID	Task Name	Duration	Start	Finish
749	Regulatory Approval of Final	0 days	9/4/2013	9/4/2013
750	**Explanation of Significant Differences**	**80 days**	**6/26/2013**	**10/15/2013**
751	Prepare and Submit Internal Draft	5 days	6/26/2013	7/2/2013
752	AF Review of Internal Draft/Provide Comments	10 days	7/3/2013	7/16/2013
753	Respond to AF Comments	10 days	7/17/2013	7/30/2013
754	Prepare and Submit Draft	5 days	7/31/2013	8/6/2013
755	Regulator Review of Draft /Provide Comments	35 days	8/7/2013	9/24/2013
756	Regulatory Response to Draft	0 days	9/24/2013	9/24/2013
757	Respond to Regulator Comments on Draft and Prepare Final	5 days	9/25/2013	10/1/2013
758	Regulatory and Air Force Review of Final	10 days	10/2/2013	10/15/2013
759	Regulatory Approval of Final	0 days	10/15/2013	10/15/2013
760	**ST006 (Building 101) - Option 2, Conduct SVE System Installation and Operation and Achieve SC**	**650 days**	**6/6/2013**	**12/2/2015**
761	**Pilot Study (SVE System Installation and Operation) Work Plan**	**71 days**	**6/6/2013**	**9/12/2013**
762	Prepare and Submit Internal Draft	9 days	6/6/2013	6/18/2013
763	AF Review of Internal Draft/Provide Comments	2 days	6/19/2013	6/20/2013
764	Respond to AF Comments	2 days	6/21/2013	6/24/2013
765	Prepare and Submit Draft	1 day	6/25/2013	6/25/2013
766	Regulator Review of Draft /Provide Comments	35 days	6/26/2013	8/13/2013
767	Regulatory Response to Draft	0 days	8/13/2013	8/13/2013
768	Respond to Regulator Comments on Draft and Prepare Final	16 days	8/14/2013	9/4/2013
769	Regulatory and Air Force Review of Final	6 days	9/5/2013	9/12/2013
770	Regulatory Approval of Final	0 days	9/12/2013	9/12/2013
771	**Pilot Study (SVE System Installation)**	**27 days**	**9/20/2013**	**10/28/2013**
772	Installation of System	17 days	9/20/2013	10/14/2013
773	System Start-up and Monitoring	10 days	10/15/2013	10/28/2013
774	**Pilot Study (SVE System Start-up and O&M) Report**	**199 days**	**10/29/2013**	**8/1/2014**
775	Prepare and Submit Internal Draft	104 days	10/29/2013	3/21/2014
776	AF Review of Internal Draft/Provide Comments	20 days	3/24/2014	4/18/2014
777	Respond to AF Comments	10 days	4/21/2014	5/2/2014
778	Prepare and Submit Draft	5 days	5/5/2014	5/9/2014
779	Regulator Review of Draft /Provide Comments	35 days	5/12/2014	6/27/2014
780	Regulatory Response to Draft	0 days	6/27/2014	6/27/2014
781	Respond to Regulator Comments on Draft and Prepare Final	10 days	6/30/2014	7/11/2014
782	Regulatory and Air Force Review of Final	15 days	7/14/2014	8/1/2014
783	Regulatory Approval of Final	0 days	8/1/2014	8/1/2014
784	**SVE System O&M - 4Q/CY13**	**47 days**	**10/29/2013**	**1/1/2014**
785	Weekly System O&M	47 days	10/29/2013	1/1/2014
786	Monitoring	5 days	11/12/2013	11/18/2013
787	**SVE System O&M Report - 4Q/CY13**	**152 days**	**1/2/2014**	**8/1/2014**
788	Prepare and Submit Internal Draft	57 days	1/2/2014	3/21/2014
789	AF Review of Internal Draft/Provide Comments	20 days	3/24/2014	4/18/2014
790	Respond to AF Comments	10 days	4/21/2014	5/2/2014
791	Prepare and Submit Draft	5 days	5/5/2014	5/9/2014
792	Regulator Review of Draft /Provide Comments	35 days	5/12/2014	6/27/2014
793	Regulatory Response to Draft	0 days	6/27/2014	6/27/2014
794	Respond to Regulator Comments on Draft and Prepare Final	10 days	6/30/2014	7/11/2014
795	Regulatory and Air Force Review of Final	15 days	7/14/2014	8/1/2014
796	Regulatory Approval of Final	0 days	8/1/2014	8/1/2014
797	**SVE System O&M - 1Q/CY14**	**64 days**	**1/2/2014**	**4/1/2014**
798	Weekly System O&M	64 days	1/2/2014	4/1/2014
799	Monitoring	5 days	1/16/2014	1/22/2014
800	**SVE System O&M Report - 1Q/CY14**	**115 days**	**4/2/2014**	**9/9/2014**
801	Prepare and Submit Internal Draft	20 days	4/2/2014	4/29/2014
802	AF Review of Internal Draft/Provide Comments	20 days	4/30/2014	5/27/2014
803	Respond to AF Comments	10 days	5/28/2014	6/10/2014
804	Prepare and Submit Draft	5 days	6/11/2014	6/17/2014
805	Regulator Review of Draft /Provide Comments	35 days	6/18/2014	8/5/2014
806	Regulatory Response to Draft	0 days	8/5/2014	8/5/2014
807	Respond to Regulator Comments on Draft and Prepare Final	10 days	8/6/2014	8/19/2014
808	Regulatory and Air Force Review of Final	15 days	8/20/2014	9/9/2014
809	Regulatory Approval of Final	0 days	9/9/2014	9/9/2014
810	**SVE System O&M - 2Q/CY14 and Installation of Full Scale SVE System**	**65 days**	**4/2/2014**	**7/1/2014**
811	Weekly System O&M	65 days	4/2/2014	7/1/2014
812	Monitoring	5 days	4/16/2014	4/22/2014
813	Installation of Additional SVE wells	10 days	4/23/2014	5/6/2014
814	**SVE System O&M Report - 2Q/CY14 and Full Scale SVE System O&M Installation and O&M Report**	**115 days**	**7/2/2014**	**12/9/2014**
815	Prepare and Submit Internal Draft	20 days	7/2/2014	7/29/2014
816	AF Review of Internal Draft/Provide Comments	20 days	7/30/2014	8/26/2014
817	Respond to AF Comments	10 days	8/27/2014	9/9/2014
818	Prepare and Submit Draft	5 days	9/10/2014	9/16/2014
819	Regulator Review of Draft /Provide Comments	35 days	9/17/2014	11/4/2014
820	Regulatory Response to Draft	0 days	11/4/2014	11/4/2014
821	Respond to Regulator Comments on Draft and Prepare Final	10 days	11/5/2014	11/18/2014
822	Regulatory and Air Force Review of Final	15 days	11/19/2014	12/9/2014
823	Regulatory Approval of Final	0 days	12/9/2014	12/9/2014
824	**SVE System O&M - 3Q/CY14**	**65 days**	**7/2/2014**	**9/30/2014**
825	Weekly System O&M	65 days	7/2/2014	9/30/2014
826	Monitoring	5 days	7/16/2014	7/22/2014
827	**SVE System O&M Report - 3Q/CY14**	**115 days**	**10/1/2014**	**3/10/2015**
828	Prepare and Submit Internal Draft	20 days	10/1/2014	10/28/2014
829	AF Review of Internal Draft/Provide Comments	20 days	10/29/2014	11/25/2014
830	Respond to AF Comments	10 days	11/26/2014	12/9/2014
831	Prepare and Submit Draft	5 days	12/10/2014	12/16/2014
832	Regulator Review of Draft /Provide Comments	35 days	12/17/2014	2/3/2015
833	Regulatory Response to Draft	0 days	2/3/2015	2/3/2015
834	Respond to Regulator Comments on Draft and Prepare Final	10 days	2/4/2015	2/17/2015
835	Regulatory and Air Force Review of Final	15 days	2/18/2015	3/10/2015
836	Regulatory Approval of Final	0 days	3/10/2015	3/10/2015
837	**SVE System O&M - 4Q/CY14**	**66 days**	**10/1/2014**	**12/31/2014**
838	Weekly System O&M	66 days	10/1/2014	12/31/2014
839	Monitoring	5 days	10/15/2014	10/21/2014
840	**SVE System O&M Report - 4Q/CY14**	**115 days**	**1/1/2015**	**6/10/2015**
841	Prepare and Submit Internal Draft	20 days	1/1/2015	1/28/2015
842	AF Review of Internal Draft/Provide Comments	20 days	1/29/2015	2/25/2015



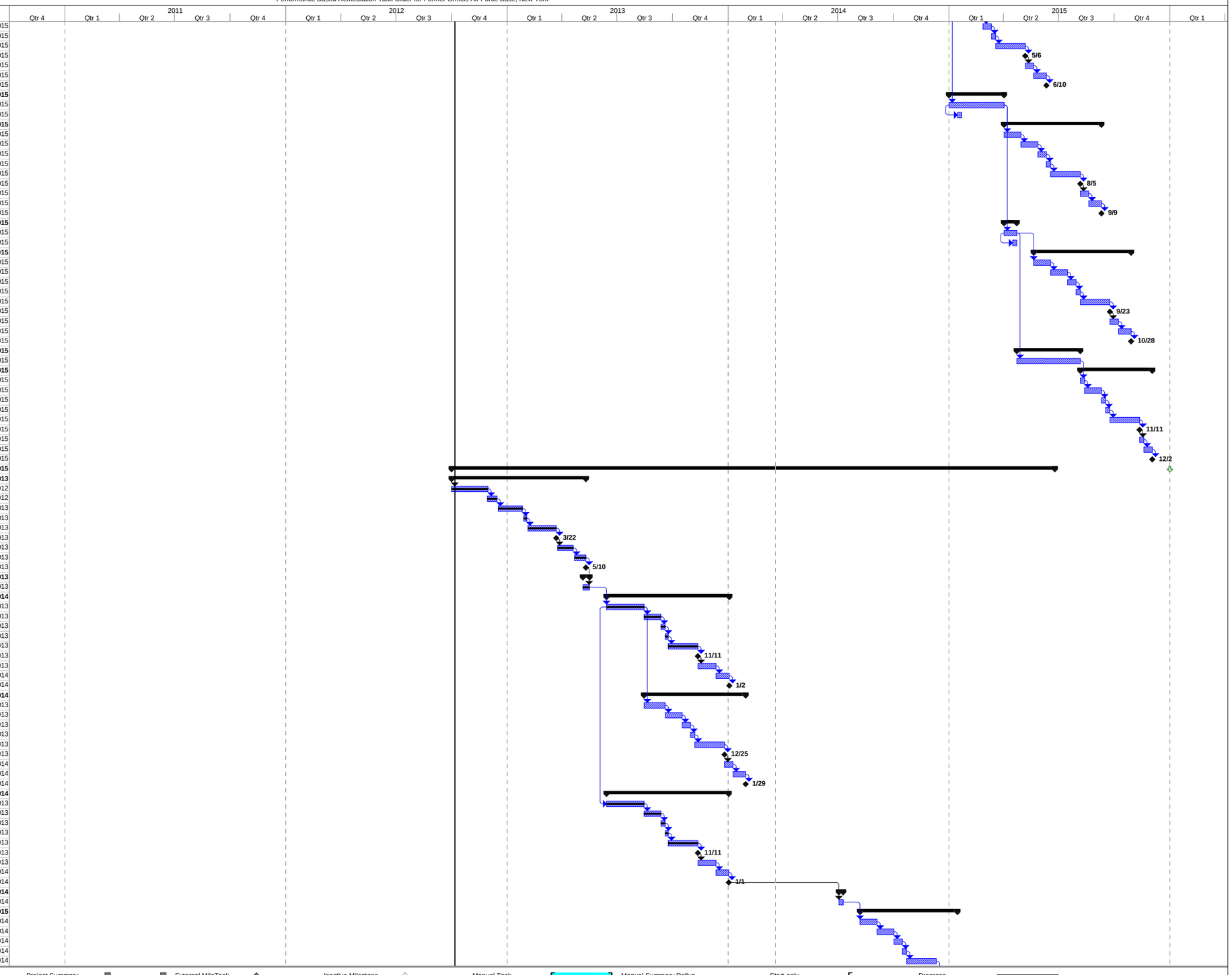
Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York

ID	Task Name	Duration	Start	Finish
843	Respond to AF Comments	10 days	2/26/2015	3/11/2015
844	Prepare and Submit Draft	5 days	3/12/2015	3/18/2015
845	Regulator Review of Draft /Provide Comments	35 days	3/19/2015	5/6/2015
846	Regulatory Response to Draft	0 days	5/6/2015	5/6/2015
847	Respond to Regulator Comments on Draft and Prepare Final	10 days	5/7/2015	5/20/2015
848	Regulatory and Air Force Review of Final	15 days	5/21/2015	6/10/2015
849	Regulatory Approval of Final	0 days	6/10/2015	6/10/2015
850	**SVE System O&M - 1Q/CY15**	**65 days**	**1/1/2015**	**4/1/2015**
851	Weekly System O&M	65 days	1/1/2015	4/1/2015
852	Monitoring	5 days	1/15/2015	1/21/2015
853	**SVE System O&M Report - 1Q/CY15**	**115 days**	**4/2/2015**	**9/9/2015**
854	Prepare and Submit Internal Draft	20 days	4/2/2015	4/29/2015
855	AF Review of Internal Draft/Provide Comments	20 days	4/30/2015	5/27/2015
856	Respond to AF Comments	10 days	5/28/2015	6/10/2015
857	Prepare and Submit Draft	5 days	6/11/2015	6/17/2015
858	Regulator Review of Draft /Provide Comments	35 days	6/18/2015	8/5/2015
859	Regulatory Response to Draft	0 days	8/5/2015	8/5/2015
860	Respond to Regulator Comments on Draft and Prepare Final	10 days	8/6/2015	8/19/2015
861	Regulatory and Air Force Review of Final	15 days	8/20/2015	9/9/2015
862	Regulatory Approval of Final	0 days	9/9/2015	9/9/2015
863	**SVE System O&M - 2Q/CY15**	**15 days**	**4/2/2015**	**4/22/2015**
864	Weekly System O&M	15 days	4/2/2015	4/22/2015
865	Monitoring	5 days	4/16/2015	4/22/2015
866	**SVE System O&M Report - 2Q/CY15**	**115 days**	**5/21/2015**	**10/28/2015**
867	Prepare and Submit Internal Draft	20 days	5/21/2015	6/17/2015
868	AF Review of Internal Draft/Provide Comments	20 days	6/18/2015	7/15/2015
869	Respond to AF Comments	10 days	7/16/2015	7/29/2015
870	Prepare and Submit Draft	5 days	7/30/2015	8/5/2015
871	Regulator Review of Draft /Provide Comments	35 days	8/6/2015	9/23/2015
872	Regulatory Response to Draft	0 days	9/23/2015	9/23/2015
873	Respond to Regulator Comments on Draft and Prepare Final	10 days	9/24/2015	10/7/2015
874	Regulatory and Air Force Review of Final	15 days	10/8/2015	10/28/2015
875	Regulatory Approval of Final	0 days	10/28/2015	10/28/2015
876	**SVE System Rebound**	**75 days**	**4/23/2015**	**8/5/2015**
877	Weekly System Rebound Monitoring	75 days	4/23/2015	8/5/2015
878	**SVE System Rebound Report/Site Closure Report**	**85 days**	**8/6/2015**	**12/2/2015**
879	Prepare and Submit Internal Draft	5 days	8/6/2015	8/12/2015
880	AF Review of Internal Draft/Provide Comments	20 days	8/13/2015	9/9/2015
881	Respond to AF Comments	5 days	9/10/2015	9/16/2015
882	Prepare and Submit Draft	5 days	9/17/2015	9/23/2015
883	Regulator Review of Draft /Provide Comments	35 days	9/24/2015	11/11/2015
884	Regulatory Response to Draft	0 days	11/11/2015	11/11/2015
885	Respond to Regulator Comments on Draft and Prepare Final	5 days	11/12/2015	11/18/2015
886	Regulatory and Air Force Review of Final	10 days	11/19/2015	12/2/2015
887	Regulatory Approval of Final	0 days	12/2/2015	12/2/2015
888	**DP012 (Building 301)- Achieve SC**	**713 days**	**10/1/2012**	**6/24/2015**
889	**Site Investigation Work Plan**	**160 days**	**10/1/2012**	**5/10/2013**
890	Prepare and Submit Internal Draft	44 days	10/1/2012	11/29/2012
891	AF Review of Internal Draft/Provide Comments	12 days	11/29/2012	12/14/2012
892	Respond to AF Comments	30 days	12/17/2012	1/25/2013
893	Prepare and Submit Draft	5 days	1/28/2013	2/1/2013
894	Regulator Review of Draft /Provide Comments	35 days	2/4/2013	3/22/2013
895	Regulatory Response to Draft	0 days	3/22/2013	3/22/2013
896	Respond to Regulator Comments on Draft and Prepare Final	20 days	3/25/2013	4/19/2013
897	Regulatory and Air Force Review of Final	15 days	4/22/2013	5/10/2013
898	Regulatory Approval of Final	0 days	5/10/2013	5/10/2013
899	**Site Investigation**	**9 days**	**5/6/2013**	**5/16/2013**
900	Geophysical and Soil Investigation Field Activities	9 days	5/6/2013	5/16/2013
901	**Site Investigation Report**	**145 days**	**6/14/2013**	**1/2/2014**
902	Prepare and Submit Internal Draft	44 days	6/14/2013	8/14/2013
903	AF Review of Internal Draft/Provide Comments	20 days	8/15/2013	9/11/2013
904	Respond to AF Comments	5 days	9/12/2013	9/18/2013
905	Prepare and Submit Draft	3 days	9/19/2013	9/23/2013
906	Regulator Review of Draft /Provide Comments	35 days	9/24/2013	11/11/2013
907	Regulatory Response to Draft	0 days	11/11/2013	11/11/2013
908	Respond to Regulator Comments on Draft and Prepare Final	22 days	11/12/2013	12/11/2013
909	Regulatory and Air Force Review of Final	16 days	12/12/2013	1/2/2014
910	Regulatory Approval of Final	0 days	1/2/2014	1/2/2014
911	**ESD**	**120 days**	**8/15/2013**	**1/29/2014**
912	Prepare and Submit Internal Draft	25 days	8/15/2013	9/18/2013
913	AF Review of Internal Draft/Provide Comments	20 days	9/19/2013	10/16/2013
914	Respond to AF Comments	10 days	10/17/2013	10/30/2013
915	Prepare and Submit Draft	5 days	10/31/2013	11/6/2013
916	Regulator Review of Draft /Provide Comments	35 days	11/7/2013	12/25/2013
917	Regulatory Response to Draft	0 days	12/25/2013	12/25/2013
918	Respond to Regulator Comments on Draft and Prepare Final	10 days	12/26/2013	1/8/2014
919	Regulatory and Air Force Review of Final	15 days	1/9/2014	1/29/2014
920	Regulatory Approval of Final	0 days	1/29/2014	1/29/2014
921	**Remedial Action Work Plan**	**144 days**	**6/14/2013**	**1/1/2014**
922	Prepare and Submit Internal Draft	44 days	6/14/2013	8/14/2013
923	AF Review of Internal Draft/Provide Comments	20 days	8/15/2013	9/11/2013
924	Respond to AF Comments	5 days	9/12/2013	9/18/2013
925	Prepare and Submit Draft	3 days	9/19/2013	9/23/2013
926	Regulator Review of Draft /Provide Comments	35 days	9/24/2013	11/11/2013
927	Regulatory Response to Draft	0 days	11/11/2013	11/11/2013
928	Respond to Regulator Comments on Draft and Prepare Final	22 days	11/12/2013	12/11/2013
929	Regulatory and Air Force Review of Final	15 days	12/12/2013	1/1/2014
930	Regulatory Approval of Final	0 days	1/1/2014	1/1/2014
931	**Remedial Action**	**5 days**	**7/3/2014**	**7/9/2014**
932	Removal Action, Confirmation Sampling and Site Restoration	5 days	7/3/2014	7/9/2014
933	**Remedial Action Completion Report**	**115 days**	**8/7/2014**	**1/14/2015**
934	Prepare and Submit Internal Draft	20 days	8/7/2014	9/3/2014
935	AF Review of Internal Draft/Provide Comments	20 days	9/4/2014	10/1/2014
936	Respond to AF Comments	10 days	10/2/2014	10/15/2014
937	Prepare and Submit Draft	5 days	10/16/2014	10/22/2014
938	Regulator Review of Draft /Provide Comments	35 days	10/23/2014	12/10/2014

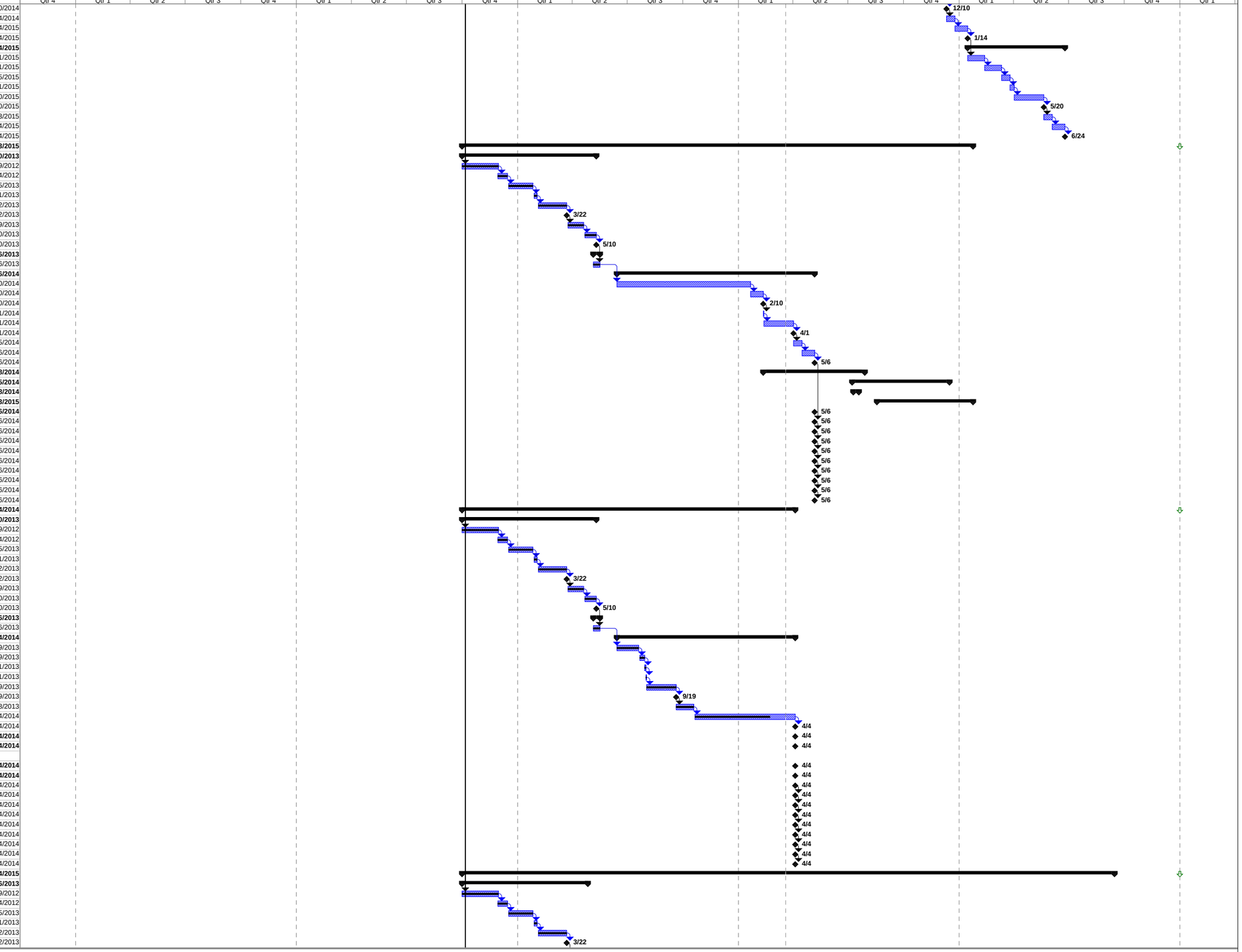


Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

ID	Task Name	Duration	Start	Finish
939	Regulatory Response to Draft	0 days	12/10/2014	12/10/2014
940	Respond to Regulator Comments on Draft and Prepare Final	10 days	12/11/2014	12/24/2014
941	Regulatory and Air Force Review of Final	15 days	12/25/2014	1/14/2015
942	Regulatory Approval of Final	0 days	1/14/2015	1/14/2015
943	**Site Close out Report**	**115 days**	**1/15/2015**	**6/24/2015**
944	Prepare and Submit Internal Draft	20 days	1/15/2015	2/11/2015
945	AF Review of Internal Draft/Provide Comments	20 days	2/12/2015	3/11/2015
946	Respond to AF Comments	10 days	3/12/2015	3/25/2015
947	Prepare and Submit Draft	5 days	3/26/2015	4/1/2015
948	Regulator Review of Draft /Provide Comments	35 days	4/2/2015	5/20/2015
949	Regulatory Response to Draft	0 days	5/20/2015	5/20/2015
950	Respond to Regulator Comments on Draft and Prepare Final	10 days	5/21/2015	6/3/2015
951	Regulatory and Air Force Review of Final	15 days	6/4/2015	6/24/2015
952	Regulatory Approval of Final	0 days	6/24/2015	6/24/2015
953	**DP013 (Building 255) - Achieve SC**	**605 days**	**10/1/2012**	**1/23/2015**
954	**Site Investigation Work Plan**	**160 days**	**10/1/2012**	**5/10/2013**
955	Prepare and Submit Internal Draft	44 days	10/1/2012	11/29/2012
956	AF Review of Internal Draft/Provide Comments	12 days	11/29/2012	12/14/2012
957	Respond to AF Comments	30 days	12/17/2012	1/25/2013
958	Prepare and Submit Draft	5 days	1/28/2013	2/1/2013
959	Regulator Review of Draft /Provide Comments	35 days	2/4/2013	3/22/2013
960	Regulatory Response to Draft	0 days	3/22/2013	3/22/2013
961	Respond to Regulator Comments on Draft and Prepare Final	20 days	3/25/2013	4/19/2013
962	Regulatory and Air Force Review of Final	15 days	4/22/2013	5/10/2013
963	Regulatory Approval of Final	0 days	5/10/2013	5/10/2013
964	**Site Investigation**	**9 days**	**5/6/2013**	**5/16/2013**
965	Geophysical and Soil Investigation Field Activities	9 days	5/6/2013	5/16/2013
966	**Site Investigation Report**	**233 days**	**6/14/2013**	**5/6/2014**
967	Prepare and Submit Internal Draft	157 days	6/14/2013	1/20/2014
968	AF Review of Internal Draft/Provide Comments	15 days	1/21/2014	2/10/2014
969	Respond to AF Comments	0 days	2/10/2014	2/10/2014
970	Prepare and Submit Draft	1 day	2/11/2014	2/11/2014
971	Regulator Review of Draft /Provide Comments	35 days	2/12/2014	4/1/2014
972	Regulatory Response to Draft	0 days	4/1/2014	4/1/2014
973	Respond to Regulator Comments on Draft and Prepare Final	10 days	4/2/2014	4/15/2014
974	Regulatory and Air Force Review of Final	15 days	4/16/2014	5/6/2014
975	Regulatory Approval of Final	0 days	5/6/2014	5/6/2014
976	**ESD/Request Closure of site East of former Building**	**120 days**	**2/11/2014**	**7/28/2014**
986	**Remedial Action Work Plan**	**115 days**	**7/8/2014**	**12/15/2014**
996	**Remedial Action**	**7 days**	**7/10/2014**	**7/18/2014**
998	**Remedial Action Completion Report**	**115 days**	**8/18/2014**	**1/23/2015**
1008	**Site Close out Report**	**0 days**	**5/6/2014**	**5/6/2014**
1009	Prepare and Submit Internal Draft	0 days	5/6/2014	5/6/2014
1010	AF Review of Internal Draft/Provide Comments	0 days	5/6/2014	5/6/2014
1011	Respond to AF Comments	0 days	5/6/2014	5/6/2014
1012	Prepare and Submit Draft	0 days	5/6/2014	5/6/2014
1013	Regulator Review of Draft /Provide Comments	0 days	5/6/2014	5/6/2014
1014	Regulatory Response to Draft	0 days	5/6/2014	5/6/2014
1015	Respond to Regulator Comments on Draft and Prepare Final	0 days	5/6/2014	5/6/2014
1016	Regulatory and Air Force Review of Final	0 days	5/6/2014	5/6/2014
1017	Regulatory Approval of Final	0 days	5/6/2014	5/6/2014
1018	**DP015 (Building 219) - Achieve SC**	**395 days**	**10/1/2012**	**4/4/2014**
1019	**Site Investigation Work Plan**	**160 days**	**10/1/2012**	**5/10/2013**
1020	Prepare and Submit Internal Draft	44 days	10/1/2012	11/29/2012
1021	AF Review of Internal Draft/Provide Comments	12 days	11/29/2012	12/14/2012
1022	Respond to AF Comments	30 days	12/17/2012	1/25/2013
1023	Prepare and Submit Draft	5 days	1/28/2013	2/1/2013
1024	Regulator Review of Draft /Provide Comments	35 days	2/4/2013	3/22/2013
1025	Regulatory Response to Draft	0 days	3/22/2013	3/22/2013
1026	Respond to Regulator Comments on Draft and Prepare Final	20 days	3/25/2013	4/19/2013
1027	Regulatory and Air Force Review of Final	15 days	4/22/2013	5/10/2013
1028	Regulatory Approval of Final	0 days	5/10/2013	5/10/2013
1029	**Site Investigation**	**9 days**	**5/6/2013**	**5/16/2013**
1030	Geophysical and Soil Investigation Field Activities	9 days	5/6/2013	5/16/2013
1031	**Site Investigation Report (SC Report)**	**211 days**	**6/14/2013**	**4/4/2014**
1032	Prepare and Submit Internal Draft	26 days	6/14/2013	7/19/2013
1033	AF Review of Internal Draft/Provide Comments	6 days	7/22/2013	7/29/2013
1034	Respond to AF Comments	2 days	7/30/2013	7/31/2013
1035	Prepare and Submit Draft	1 day	8/1/2013	8/1/2013
1036	Regulator Review of Draft /Provide Comments	35 days	8/2/2013	9/19/2013
1037	Regulatory Response to Draft	0 days	9/19/2013	9/19/2013
1038	Respond to Regulator Comments on Draft and Prepare Final	21 days	9/20/2013	10/18/2013
1039	Regulatory and Air Force Review of Final	120 days	10/21/2013	4/4/2014
1040	Regulatory Approval of Final	0 days	4/4/2014	4/4/2014
1041	**ESD**	**0 days**	**4/4/2014**	**4/4/2014**
1051	**Remedial Action Work Plan**	**0 days**	**4/4/2014**	**4/4/2014**
1061	**Remedial Action**			
1063	**Remedial Action Completion Report**	**0 days**	**4/4/2014**	**4/4/2014**
1073	**Site Close out Report**	**0 days**	**4/4/2014**	**4/4/2014**
1074	Prepare and Submit Internal Draft	0 days	4/4/2014	4/4/2014
1075	AF Review of Internal Draft/Provide Comments	0 days	4/4/2014	4/4/2014
1076	Respond to AF Comments	0 days	4/4/2014	4/4/2014
1077	Prepare and Submit Draft	0 days	4/4/2014	4/4/2014
1078	Regulator Review of Draft /Provide Comments	0 days	4/4/2014	4/4/2014
1079	Regulatory Response to Draft	0 days	4/4/2014	4/4/2014
1080	Respond to Regulator Comments on Draft and Prepare Final	0 days	4/4/2014	4/4/2014
1081	Regulatory and Air Force Review of Final	0 days	4/4/2014	4/4/2014
1082	Regulatory Approval of Final	0 days	4/4/2014	4/4/2014
1083	**SS024 (Fire Demonstration Area) - Achieve SC**	**771 days**	**10/1/2012**	**9/14/2015**
1084	**Soil and Background Investigation Work Plan**	**150 days**	**10/1/2012**	**4/26/2013**
1085	Prepare and Submit Internal Draft	44 days	10/1/2012	11/29/2012
1086	AF Review of Internal Draft/Provide Comments	12 days	11/29/2012	12/14/2012
1087	Respond to AF Comments	30 days	12/17/2012	1/25/2013
1088	Prepare and Submit Draft	5 days	1/28/2013	2/1/2013
1089	Regulator Review of Draft /Provide Comments	35 days	2/4/2013	3/22/2013
1090	Regulatory Response to Draft	0 days	3/22/2013	3/22/2013

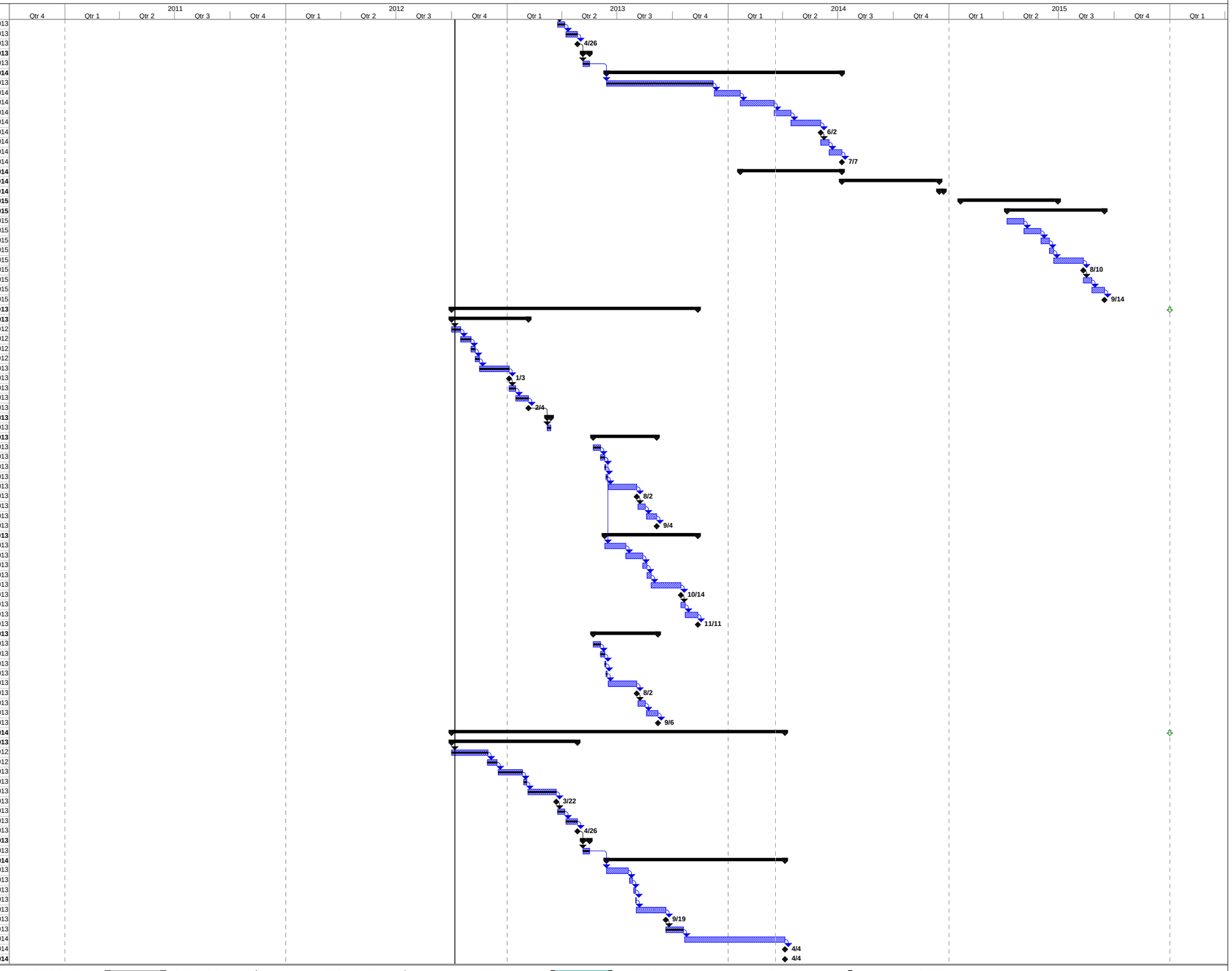


Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

ID	Task Name	Duration	Start	Finish
1091	Respond to Regulator Comments on Draft and Prepare Final	10 days	3/25/2013	4/5/2013
1092	Regulatory and Air Force Review of Final	15 days	4/8/2013	4/26/2013
1093	Regulatory Approval of Final	0 days	4/26/2013	4/26/2013
1094	**Soil and Background Investigation**	**9 days**	**5/6/2013**	**5/16/2013**
1095	Soil and Background Investigation Field Activities	9 days	5/6/2013	5/16/2013
1096	**Soil and Background Investigation Report**	**277 days**	**6/14/2013**	**7/7/2014**
1097	Prepare and Submit Internal Draft	126 days	6/14/2013	12/6/2013
1098	AF Review of Internal Draft/Provide Comments	31 days	12/9/2013	1/20/2014
1099	Respond to AF Comments	40 days	1/21/2014	3/17/2014
1100	Prepare and Submit Draft	20 days	3/18/2014	4/14/2014
1101	Regulator Review of Draft /Provide Comments	35 days	4/15/2014	6/2/2014
1102	Regulatory Response to Draft	0 days	6/2/2014	6/2/2014
1103	Respond to Regulator Comments on Draft and Prepare Final	10 days	6/3/2014	6/16/2014
1104	Regulatory and Air Force Review of Final	15 days	6/17/2014	7/7/2014
1105	Regulatory Approval of Final	0 days	7/7/2014	7/7/2014
1106	**ESD**	**120 days**	**1/21/2014**	**7/7/2014**
1116	**Remedial Action Work Plan**	**115 days**	**7/8/2014**	**12/15/2014**
1126	**Remedial Action**	**5 days**	**12/16/2014**	**12/22/2014**
1128	**Remedial Action Completion Report**	**115 days**	**1/20/2015**	**6/29/2015**
1138	**Site Close out Report**	**115 days**	**4/7/2015**	**9/14/2015**
1139	Prepare and Submit Internal Draft	20 days	4/7/2015	5/4/2015
1140	AF Review of Internal Draft/Provide Comments	20 days	5/5/2015	6/1/2015
1141	Respond to AF Comments	10 days	6/2/2015	6/15/2015
1142	Prepare and Submit Draft	5 days	6/16/2015	6/22/2015
1143	Regulator Review of Draft /Provide Comments	35 days	6/23/2015	8/10/2015
1144	Regulatory Response to Draft	0 days	8/10/2015	8/10/2015
1145	Respond to Regulator Comments on Draft and Prepare Final	10 days	8/11/2015	8/24/2015
1146	Regulatory and Air Force Review of Final	15 days	8/25/2015	9/14/2015
1147	Regulatory Approval of Final	0 days	9/14/2015	9/14/2015
1148	**FT030 - Conduct SVI Evaluation and Achieve SC**	**291 days**	**10/1/2012**	**11/11/2013**
1149	**SVI Evaluation Work Plan**	**91 days**	**10/1/2012**	**2/4/2013**
1150	Prepare and Submit Internal Draft	11 days	10/1/2012	10/15/2012
1151	AF Review of Internal Draft/Provide Comments	13 days	10/16/2012	11/1/2012
1152	Respond to AF Comments	5 days	11/2/2012	11/8/2012
1153	Prepare and Submit Draft	5 days	11/9/2012	11/15/2012
1154	Regulator Review of Draft /Provide Comments	35 days	11/16/2012	1/3/2013
1155	Regulatory Response to Draft	0 days	1/3/2013	1/3/2013
1156	Respond to Regulator Comments on Draft and Prepare Final	7 days	1/4/2013	1/14/2013
1157	Regulatory and Air Force Review of Final	15 days	1/15/2013	2/4/2013
1158	Regulatory Approval of Final	0 days	2/4/2013	2/4/2013
1159	**SVI Evaluation**	**4 days**	**3/8/2013**	**3/13/2013**
1160	SVI Sampling	4 days	3/8/2013	3/13/2013
1161	**SVI Evaluation Report**	**75 days**	**5/23/2013**	**9/4/2013**
1162	Prepare and Submit Internal Draft	8 days	5/23/2013	6/3/2013
1163	AF Review of Internal Draft/Provide Comments	5 days	6/4/2013	6/10/2013
1164	Respond to AF Comments	2 days	6/11/2013	6/12/2013
1165	Prepare and Submit Draft	2 days	6/13/2013	6/14/2013
1166	Regulator Review of Draft /Provide Comments	35 days	6/17/2013	8/2/2013
1167	Regulatory Response to Draft	0 days	8/2/2013	8/2/2013
1168	Respond to Regulator Comments on Draft and Prepare Final	10 days	8/5/2013	8/16/2013
1169	Regulatory and Air Force Review of Final	13 days	8/19/2013	9/4/2013
1170	Regulatory Approval of Final	0 days	9/4/2013	9/4/2013
1171	**ESD**	**110 days**	**6/11/2013**	**11/11/2013**
1172	Prepare and Submit Internal Draft	25 days	6/11/2013	7/15/2013
1173	AF Review of Internal Draft/Provide Comments	20 days	7/16/2013	8/12/2013
1174	Respond to AF Comments	5 days	8/13/2013	8/19/2013
1175	Prepare and Submit Draft	5 days	8/20/2013	8/26/2013
1176	Regulator Review of Draft /Provide Comments	35 days	8/27/2013	10/14/2013
1177	Regulatory Response to Draft	0 days	10/14/2013	10/14/2013
1178	Respond to Regulator Comments on Draft and Prepare Final	5 days	10/15/2013	10/21/2013
1179	Regulatory and Air Force Review of Final	15 days	10/22/2013	11/11/2013
1180	Regulatory Approval of Final	0 days	11/11/2013	11/11/2013
1181	**Risk Assessment Report**	**77 days**	**5/23/2013**	**9/6/2013**
1182	Prepare and Submit Internal Draft	8 days	5/23/2013	6/3/2013
1183	AF Review of Internal Draft/Provide Comments	5 days	6/4/2013	6/10/2013
1184	Respond to AF Comments	2 days	6/11/2013	6/12/2013
1185	Prepare and Submit Draft	2 days	6/13/2013	6/14/2013
1186	Regulator Review of Draft /Provide Comments	35 days	6/17/2013	8/2/2013
1187	Regulatory Response to Draft	0 days	8/2/2013	8/2/2013
1188	Respond to Regulator Comments on Draft and Prepare Final	10 days	8/5/2013	8/16/2013
1189	Regulatory and Air Force Review of Final	15 days	8/19/2013	9/6/2013
1190	Regulatory Approval of Final	0 days	9/6/2013	9/6/2013
1191	**SD050 (Building 214) - Achieve SC**	**395 days**	**10/1/2012**	**4/4/2014**
1192	**Site Investigation Work Plan**	**150 days**	**10/1/2012**	**4/26/2013**
1193	Prepare and Submit Internal Draft	44 days	10/1/2012	11/29/2012
1194	AF Review of Internal Draft/Provide Comments	12 days	11/29/2012	12/14/2012
1195	Respond to AF Comments	30 days	12/17/2012	1/25/2013
1196	Prepare and Submit Draft	5 days	1/28/2013	2/1/2013
1197	Regulator Review of Draft /Provide Comments	35 days	2/4/2013	3/22/2013
1198	Regulatory Response to Draft	0 days	3/22/2013	3/22/2013
1199	Respond to Regulator Comments on Draft and Prepare Final	10 days	3/25/2013	4/5/2013
1200	Regulatory and Air Force Review of Final	15 days	4/8/2013	4/26/2013
1201	Regulatory Approval of Final	0 days	4/26/2013	4/26/2013
1202	**Site Investigation**	**9 days**	**5/6/2013**	**5/16/2013**
1203	Soil Investigation Field Activities	9 days	5/6/2013	5/16/2013
1204	**Site Investigation Report (SC Report)**	**211 days**	**6/14/2013**	**4/4/2014**
1205	Prepare and Submit Internal Draft	26 days	6/14/2013	7/19/2013
1206	AF Review of Internal Draft/Provide Comments	5 days	7/22/2013	7/26/2013
1207	Respond to AF Comments	3 days	7/29/2013	7/31/2013
1208	Prepare and Submit Draft	1 day	8/1/2013	8/1/2013
1209	Regulator Review of Draft /Provide Comments	35 days	8/2/2013	9/19/2013
1210	Regulatory Response to Draft	0 days	9/19/2013	9/19/2013
1211	Respond to Regulator Comments on Draft and Prepare Final	21 days	9/20/2013	10/18/2013
1212	Regulatory and Air Force Review of Final	120 days	10/21/2013	4/4/2014
1213	Regulatory Approval of Final	0 days	4/4/2014	4/4/2014
1214	**ESD**	**0 days**	**4/4/2014**	**4/4/2014**



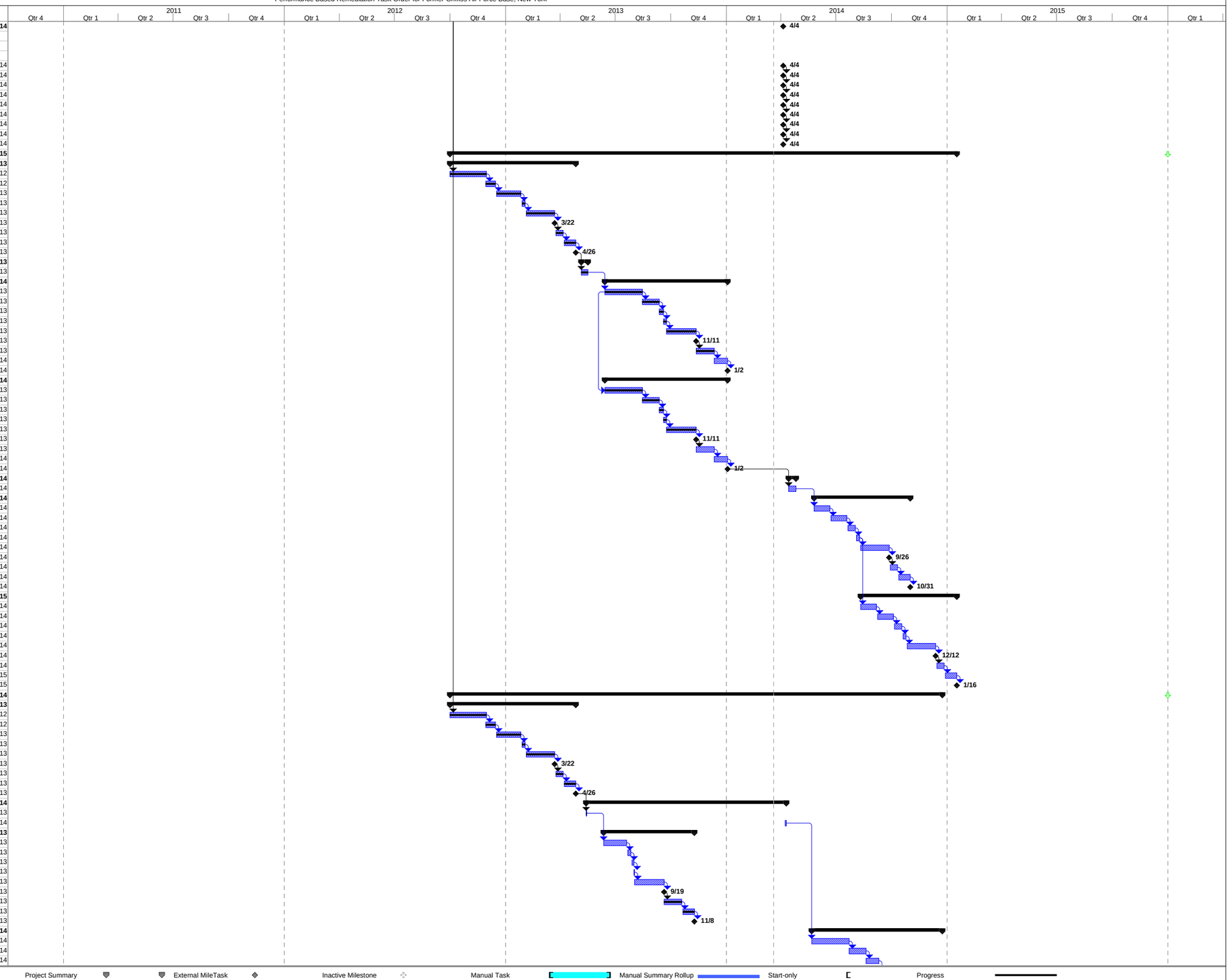
Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York

ID	Task Name	Duration	Start	Finish
1224	**Remedial Action Work Plan**	**0 days**	**4/4/2014**	**4/4/2014**
1234	**Remedial Action**	**1 day**		
1236	**Remedial Action Completion Report**			
1246	**Site Close out Report**	**1 day**		
1247	Prepare and Submit Internal Draft	0 days	4/4/2014	4/4/2014
1248	AF Review of Internal Draft/Provide Comments	0 days	4/4/2014	4/4/2014
1249	Respond to AF Comments	0 days	4/4/2014	4/4/2014
1250	Prepare and Submit Draft	0 days	4/4/2014	4/4/2014
1251	Regulator Review of Draft /Provide Comments	0 days	4/4/2014	4/4/2014
1252	Regulatory Response to Draft	0 days	4/4/2014	4/4/2014
1253	Respond to Regulator Comments on Draft and Prepare Final	0 days	4/4/2014	4/4/2014
1254	Regulatory and Air Force Review of Final	0 days	4/4/2014	4/4/2014
1255	Regulatory Approval of Final	0 days	4/4/2014	4/4/2014
1256	**AOI 72- Achieve SC**	**600 days**	**10/1/2012**	**1/16/2015**
1257	**Site Investigation Work Plan**	**150 days**	**10/1/2012**	**4/26/2013**
1258	Prepare and Submit Internal Draft	44 days	10/1/2012	11/29/2012
1259	AF Review of Internal Draft/Provide Comments	12 days	11/29/2012	12/14/2012
1260	Respond to AF Comments	30 days	12/17/2012	1/25/2013
1261	Prepare and Submit Draft	5 days	1/28/2013	2/1/2013
1262	Regulator Review of Draft /Provide Comments	35 days	2/4/2013	3/22/2013
1263	Regulatory Response to Draft	0 days	3/22/2013	3/22/2013
1264	Respond to Regulator Comments on Draft and Prepare Final	10 days	3/25/2013	4/5/2013
1265	Regulatory and Air Force Review of Final	15 days	4/8/2013	4/26/2013
1266	Regulatory Approval of Final	0 days	4/26/2013	4/26/2013
1267	**Soil Investigation**	**9 days**	**5/6/2013**	**5/16/2013**
1268	Surface and SubSurface Soil Sampling	9 days	5/6/2013	5/16/2013
1269	**Site Investigation Report**	**145 days**	**6/14/2013**	**1/2/2014**
1270	Prepare and Submit Internal Draft	44 days	6/14/2013	8/14/2013
1271	AF Review of Internal Draft/Provide Comments	20 days	8/15/2013	9/11/2013
1272	Respond to AF Comments	5 days	9/12/2013	9/18/2013
1273	Prepare and Submit Draft	3 days	9/19/2013	9/23/2013
1274	Regulator Review of Draft /Provide Comments	35 days	9/24/2013	11/11/2013
1275	Regulatory Response to Draft	0 days	11/11/2013	11/11/2013
1276	Respond to Regulator Comments on Draft and Prepare Final	22 days	11/12/2013	12/11/2013
1277	Regulatory and Air Force Review of Final	16 days	12/12/2013	1/2/2014
1278	Regulatory Approval of Final	0 days	1/2/2014	1/2/2014
1279	**Remedial Action Work Plan**	**145 days**	**6/14/2013**	**1/2/2014**
1280	Prepare and Submit Internal Draft	44 days	6/14/2013	8/14/2013
1281	AF Review of Internal Draft/Provide Comments	20 days	8/15/2013	9/11/2013
1282	Respond to AF Comments	5 days	9/12/2013	9/18/2013
1283	Prepare and Submit Draft	3 days	9/19/2013	9/23/2013
1284	Regulator Review of Draft /Provide Comments	35 days	9/24/2013	11/11/2013
1285	Regulatory Response to Draft	0 days	11/11/2013	11/11/2013
1286	Respond to Regulator Comments on Draft and Prepare Final	22 days	11/12/2013	12/11/2013
1287	Regulatory and Air Force Review of Final	16 days	12/12/2013	1/2/2014
1288	Regulatory Approval of Final	0 days	1/2/2014	1/2/2014
1289	**Remedial Action**	**10 days**	**4/14/2014**	**4/25/2014**
1290	Removal Action, Confirmation Sampling and Site Restoration	10 days	4/14/2014	4/25/2014
1291	**Remedial Action Completion Report**	**115 days**	**5/26/2014**	**10/31/2014**
1292	Prepare and Submit Internal Draft	20 days	5/26/2014	6/20/2014
1293	AF Review of Internal Draft/Provide Comments	20 days	6/23/2014	7/18/2014
1294	Respond to AF Comments	10 days	7/21/2014	8/1/2014
1295	Prepare and Submit Draft	5 days	8/4/2014	8/8/2014
1296	Regulator Review of Draft /Provide Comments	35 days	8/11/2014	9/26/2014
1297	Regulatory Response to Draft	0 days	9/26/2014	9/26/2014
1298	Respond to Regulator Comments on Draft and Prepare Final	10 days	9/29/2014	10/10/2014
1299	Regulatory and Air Force Review of Final	15 days	10/13/2014	10/31/2014
1300	Regulatory Approval of Final	0 days	10/31/2014	10/31/2014
1301	**Site Close out Report**	**115 days**	**8/11/2014**	**1/16/2015**
1302	Prepare and Submit Internal Draft	20 days	8/11/2014	9/5/2014
1303	AF Review of Internal Draft/Provide Comments	20 days	9/8/2014	10/3/2014
1304	Respond to AF Comments	10 days	10/6/2014	10/17/2014
1305	Prepare and Submit Draft	5 days	10/20/2014	10/24/2014
1306	Regulator Review of Draft /Provide Comments	35 days	10/27/2014	12/12/2014
1307	Regulatory Response to Draft	0 days	12/12/2014	12/12/2014
1308	Respond to Regulator Comments on Draft and Prepare Final	10 days	12/15/2014	12/26/2014
1309	Regulatory and Air Force Review of Final	15 days	12/29/2014	1/16/2015
1310	Regulatory Approval of Final	0 days	1/16/2015	1/16/2015
1311	**DW211 (Building 211 Drywell) - Achieve SC**	**582 days**	**10/1/2012**	**12/23/2014**
1312	**Site Investigation Work Plan**	**150 days**	**10/1/2012**	**4/26/2013**
1313	Prepare and Submit Internal Draft	44 days	10/1/2012	11/29/2012
1314	AF Review of Internal Draft/Provide Comments	12 days	11/29/2012	12/14/2012
1315	Respond to AF Comments	30 days	12/17/2012	1/25/2013
1316	Prepare and Submit Draft	5 days	1/28/2013	2/1/2013
1317	Regulator Review of Draft /Provide Comments	35 days	2/4/2013	3/22/2013
1318	Regulatory Response to Draft	0 days	3/22/2013	3/22/2013
1319	Respond to Regulator Comments on Draft and Prepare Final	10 days	3/25/2013	4/5/2013
1320	Regulatory and Air Force Review of Final	15 days	4/8/2013	4/26/2013
1321	Regulatory Approval of Final	0 days	4/26/2013	4/26/2013
1322	**Sampling**	**237 days**	**5/14/2013**	**4/9/2014**
1323	Concrete Sampling	1 day	5/14/2013	5/14/2013
1324	Additional Concrete Sampling	2 days	4/8/2014	4/9/2014
1325	**Site Investigation Report**	**108 days**	**6/12/2013**	**11/8/2013**
1326	Prepare and Submit Internal Draft	28 days	6/12/2013	7/19/2013
1327	AF Review of Internal Draft/Provide Comments	5 days	7/22/2013	7/26/2013
1328	Respond to AF Comments	3 days	7/29/2013	7/31/2013
1329	Prepare and Submit Draft	1 day	8/1/2013	8/1/2013
1330	Regulator Review of Draft /Provide Comments	35 days	8/2/2013	9/19/2013
1331	Regulatory Response to Draft	0 days	9/19/2013	9/19/2013
1332	Respond to Regulator Comments on Draft and Prepare Final	21 days	9/20/2013	10/18/2013
1333	Regulatory and Air Force Review of Final	15 days	10/21/2013	11/8/2013
1334	Regulatory Approval of Final	0 days	11/8/2013	11/8/2013
1335	**Site Close out Report**	**154 days**	**5/22/2014**	**12/23/2014**
1336	Prepare and Submit Internal Draft	44 days	5/22/2014	7/22/2014
1337	AF Review of Internal Draft/Provide Comments	20 days	7/23/2014	8/19/2014
1338	Respond to AF Comments	15 days	8/20/2014	9/9/2014



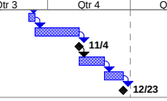
Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split

Performance Based Remediation Task Order for Former Griffiss Air Force Base, New York

ID	Task Name	Duration	Start	Finish
1339	Prepare and Submit Draft	5 days	9/10/2014	9/16/2014
1340	Regulator Review of Draft /Provide Comments	35 days	9/17/2014	11/4/2014
1341	Regulatory Response to Draft	0 days	11/4/2014	11/4/2014
1342	Respond to Regulator Comments on Draft and Prepare Final	20 days	11/5/2014	12/2/2014
1343	Regulatory and Air Force Review of Final	15 days	12/3/2014	12/23/2014
1344	Regulatory Approval of Final	0 days	12/23/2014	12/23/2014



Project: Project1

Task | Milestone | Project Summary | External MileTask | Inactive Milestone | Manual Task | Manual Summary Rollup | Start-only | Progress

Split | Summary | External Tasks | Inactive Task | Inactive Summary | Duration-only | Manual Summary | Finish-only | Split