LAN ASSOCIATES. Inc.

Consulting • Engineering • Planning

66 Cuna Street • St. Augustine, FL 32084

Ph: 904-824-6999 • Fax: 904-824-0726

PROJECT: (Description) CC Metals and Alloys, LLC
Cells #1 and #2

PROJECT NO: 2.3643.17

DATE: 7/2/2008

TO: New York State Dept. of Env. Conservation
270 Michigan Avenue
Buffalo, NY 14203-2999

If enclosures are not as noted, please inform us immediately.

If checked below, please:

ATTN: Mr. Mike Hinton

☐ Acknowledge receipt of enclosures

☐ Return enclosures to us

WE TRANSMIT:

X **Herewith** — **In accordance with your request**

Under separate cover

FOR YOUR:

Approval — **Signature** — **Files**

Review /Comment — X **Record** — **Other**

Use — **Information**

THE FOLLOWING:

Drawings — **Forms** — **Samples**

Specifications — **Literature** — **Other**

Letter — X **Report(s)**

COPIES	DATE	REV#	DESCRIPTION	ACTION
2	06/20/08		CC Metals &Alloys, LLC Witmer Road OM&M Manual	B

ACTION:
A. Action indicated on item transmitted
B. No action required
C. For signature and return to this office
D. For signature and forwarding as noted below under REMARKS
E. See REMARKS below

REMARKS:

Mr. Hinton:

Enclosed are the two addtional copies of the OM&M Manual for CC Metals and Alloys. If you need anything more, please do not hesitate to contact me.

COPIES TO:

BY: Guy D. VanDoren

LAN Associates
DAILY SHIPMENT DETAIL REPORT
02-Jul-2008 - 2:31:30 PM

2.3643.17

Pickup Date: 02-Jul-2008
Pickup Record No.: 9262 3891 13

UPS Account No.: X65951
Sorted By: Order of Shipment

All charges are in USD; fuel surcharge included where applicable.

Name / Address	Shipment Detail		Options	Published Rate Charges
Ship To: Mr. Mike Hinton New York State Dept of Env. Conserv 270 Michgan Ave Buffalo NY 14203-2999 United States (Residential)	Service Type: Total Packages: Billable Wt.: Transportation:	2ND DAY AIR 1 2.0 lb Shipper	Shipment Service Charge:	17.41
	Tracking No.: Package Type: Weight:	1ZX659510257252391 Package 2.0 lb	Package Service Charge: **UPS Total Charge:**	17.41 **17.41**

Summary Totals:

Shipment Option	Shpts	Pkgs	Pub Charges
Package Option		**Pkgs**	**Pub Charges**

Billing Option	Shpts	Pkgs	Pub Charges
Prepaid	1	1	17.41
TOTAL CHARGES			**17.41**

1 Shipment(s)
1 Package(s)

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LAN Associates, Inc.
DAILY SHIPMENT DETAIL REPORT
20-Jun-2008 - 2:50:47 PM

Pickup Date: 20-Jun-2008
Pickup Record No.: 4839 6524 25

UPS Account No.: X65951
Sorted By: Order of Shipment

All charges are in USD; fuel surcharge included where applicable.

Name / Address	Shipment Detail		Options	Published Rate Charges
Ship To: Mr. Michael J. Hinton, P.E. NY State Dept of Env. Conservation 270 Michigan Avenue BUFFALO NY 14203-2915 United States	Service Type: Total Packages: Billable Wt.: Transportation: Package Ref No.1:	GROUND 1 1.0 lb Shipper 2.3643.17	Shipment Service Charge:	5.05
	Tracking No.: Package Type: Weight: Package Ref No.1:	1ZX659510354254688 Package 1.0 lb 2.3643.17	Package Service Charge: **UPS Total Charge:**	5.05 **5.05**
Ship To: Mr. Matthew Farcucci New York State Dept of Health 584 Delaware Avenue BUFFALO NY 14202-1295 United States	Service Type: Total Packages: Billable Wt.: Transportation: Package Ref No.1:	GROUND 1 2.0 lb Shipper 2.3643.17	Shipment Service Charge:	5.51
	Tracking No.: Package Type: Weight: Package Ref No.1:	1ZX659510353522694 Package 2.0 lb 2.3643.17	Package Service Charge: **UPS Total Charge:**	5.51 **5.51**
Ship To: Ms. Alice Brown/Ed Bredniak CC Metals and Alloys, LLC 1542 N. Main Street CALVERT CITY KY 42029 United States	Service Type: Total Packages: Billable Wt.: Transportation: Package Ref No.1:	GROUND 1 1.0 lb Shipper 2.3643.17	Shipment Service Charge:	6.67
	Tracking No.: Package Type: Weight: Package Ref No.1: Delivery Area Surcharge:	1ZX659510355593908 Package 1.0 lb 2.3643.17 Rural Extended	Package Service Charge: **UPS Total Charge:**	6.67 **6.67**
Ship To: Ms. Sylvia Vartuoso Town of Niagara 7105 Lockport Road NIAGARA FALLS NY 14305 United States	Service Type: Total Packages: Billable Wt.: Transportation: Package Ref No.1:	GROUND 1 1.0 lb Shipper 2.3643.17	Shipment Service Charge:	5.05
	Tracking No.: Package Type: Weight: Package Ref No.1:	1ZX659510353292317 Package 1.0 lb 2.3643.17	Package Service Charge: **UPS Total Charge:**	5.05 **5.05**

Summary Totals:

Shipment Option	Shpts	Pkgs	Pub Charges
Package Option		**Pkgs**	**Pub Charges**

Billing Option	Shpts	Pkgs	Pub Charges
Prepaid	4	4	22.28
TOTAL CHARGES			**22.28**

4 Shipment(s)
4 Package(s)

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LAN ASSOCIATES. Inc.

Consulting • Engineering • Planning

66 Cuna Street • St. Augustine, FL 32084

Ph: 904-824-6999 • Fax: 904-824-0726

PROJECT: (Description) CC Metals and Alloys, LLC
Cells #1 and #2

PROJECT NO: 2.3643.17

DATE: 6/20/2008

TO: New York State Dept. of Env. Conservation
270 Michigan Avenue
Buffalo, NY 14203-2999

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ATTN: Mr. Mike Hinton

☐ Acknowledge receipt of enclosures

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X **Herewith** — **In accordance with your request**

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Letter — X **Report(s)**

COPIES	DATE	REV#	DESCRIPTION	ACTION
1	06/20/08		CC Metals &Alloys, LLC Witmer Road OM&M Manual (formerly known as the Vanadium site)	B

ACTION:
A. Action indicated on item transmitted
B. No action required
C. For signature and return to this office
D. For signature and forwarding as noted below under REMARKS
E. See REMARKS below

REMARKS:

COPIES TO:
Matthew Farcucci, NYSDOH (2)
Syliva Vartuoso, Town of Niagara (1)

BY: Guy D. VanDoren

Guy VanDoren/po.

LAN ASSOCIATES, INC.
CONSULTING • ENGINEERING • PLANNING

June 19, 2008

BY UPS DELIVERY

Mr. Michael J. Hinton, P.E.
Division of Environmental Remediation
New York State Department of Environmental Conservation
270 Michigan Avenue
Buffalo, NY 14203-2999

Subject: CC Metals and Alloys, LLC
Site #932001 Operable Unit #1
OM&M Manual
LAN Ref. #2.3643.17

Dear Mr. Hinton:

LAN Associates, Inc. (LAN) is in receipt of your letter dated May 21, 2008, for Operation Monitoring and Maintenance Manual (OM&M) approval with modifications. LAN will respond to each of your comments in the order they appear in your letter.

1) *The OM&M manual must be certified by a New York State Registered Professional Engineer.*

   The attached revised version of the OM&M manual has been certified by a NYS Registered Professional Engineer.

2) *Section 3.0, page 2 – Please add two additional goals of the Post Closure Maintenance Requirements. The fourth goal must be to the maintenance of the drainage pathways and controls implemented under the IRM order and completed in 1999. The fifth goal must be the annual certification that the Deed Restriction (Institutional Control) filed with the Niagara County Clerk and recorded May 3, 2001 in Book 3114 on page 291 is still in effect and has not been altered. In addition an inspection check list must be developed and included in this document for use during field inspections of the site. A copy of the completed checklist is to be included in the inspection report.*

   Section 3, page 2 has been edited to include both the fourth and fifth goals as indicated. A copy of the Deed Restriction has been included as Appendix A for in the Revised OM&M Manual. In addition, an inspection checklist has been included as Appendix B (see page 3 of Revised OM&M Manual).

3) *Section 3.0, page 3 – fourth paragraph, the vegetative cover on the landfill cells and the drainage areas must be mowed at least once per year preferably after September 1.*

The fourth paragraph has been edited to include a sentence stating that the vegetative cover on the landfill cells as well as the drainage areas will be mowed once per year.

4) *Section 3.0 – Please add a paragraph describing the existence of the Deed Restriction placed on the drainage area portion of the property.*

An additional paragraph has been added to Section 3.0 describing the existence of the Deed Restriction.

5) *Section 4.1.2, page 6 – Please provide more detail on the location of the sampling point SW-1. It is our understanding that the sampling point should be at the drainage structure flow control valve installed as part of the IRM project. The description found in this paragraph and the site plan in Appendix A do not appear to indicate the proper location for SW-1.*

Additional detail has been added to Section 4.1.2 identifying the location of sampling point SW-1. A new drawing, Well Location & Surface Water Drainage Map, has been added that clearly identifies the location of SW-1.

6) *Section 4.2, page 6 – Please revise the monitoring frequency and parameters to include all the compounds listed in this section with the exception of VOCs on a semi-annual basis. VOC analysis will be required annually. Also, please clearly indicate that the leachate sump will be sampled and provide a sample identifier similar to the monitoring wells and surface water sampling points.*

Section 4.2 has been edited to better explain the site monitoring frequency and parameters, specifically VOC analysis on an annual basis. Additionally the leachate sump sample has been given sample identifier (LS-1). This is also indicated on the Well Location and Surface Water Drainage Map.

7) *Section 4.7, page 12 – Due to the presence of an Institutional Control in the form of a Deed Restriction filed with the Niagara County Clerk, the Annual Summary Report must be certified by a Professional Engineer registered in the State of New York.*

Section 4.7 has been modified to include a sentence clarifying that the Annual Summary Report will be certified by a Professional Engineer registered in the State of New York.

8) *Appendix A – Please revise the site plan to indicate the property subdivision between the landfill area and the drainage area addressed by the IRM. Also provide drainage pathways and indicate the location of the outlet structure and sampling points including the landfill sump.*

A new site plan is included in the revised OM&M Manual as Appendix C. The new map, Well Location and Surface Water Drainage Map, clearly indicates the location of the property subdivision line as well as provides drainage pathways and the location for the leachate sump (LS-1).

9) *Appendix B – Please include the leachate sampling procedures in the SOP for Groundwater/Surface Water Sampling.*

Section 4.4 of the OM&M manual explains that the SOP for Groundwater/Surface Water Sampling is the same procedure used to sample the leachate sump (LS-1). The lab was questioned on this subject

and confirmed this point, adding that the only difference is the method used to obtain the grab sample. The sampling is performed using a bailer.

10) Please include an additional appendix with a copy of the deed restriction.

A copy of the deed restriction is included with the revised OM&M Manual as Appendix A.

The above referenced comments have been addressed in the attached revised version of the OM&M manual. As you requested, a new CD Rom has been included that provides a digital version of the manual and its attachments. If you have any further questions or comments please feel free to contact LAN Associates, Inc.

Very truly yours,

Guy D. VanDoren, P.E.
President

GDVD:jw
2.3643.17-L-NYSDEC OM&M-080619-gvd

Copies to: Mr. Ed Bredniak, CCMA

Enclosures: CD Rom (1)

OPERATION MONITORING AND MAINTENANCE MANUAL
CELLS 1 AND 2

for

CC METALS AND ALLOYS, LLC
WITMER ROAD
Town of Niagara, Niagara County, NY
Site #932001 Operable Unit #1

Submitted to:

New York State Department of Environmental Conservation
270 Michigan Avenue
Buffalo, NY 14203

LAN ASSOCIATES, INC.
CONSULTING • ENGINEERING • PLANNING

66 Cuna Street • St. Augustine, FL 32084 • Ph: (904) 824-6999 • Fax: (904) 824-0726 • www.lan-fl.com

LAN Ref. #2.3643.17
April 18, 2008
Revised: June 19, 2008

LAN ASSOCIATES, INC.
CONSULTING • ENGINEERING • PLANNING

OPERATION MONITORING AND MAINTENANCE MANUAL
CELLS 1 AND 2

FOR
CC METALS AND ALLOYS, LLC
WITMER ROAD

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LAN ASSOCIATES, INC.
CONSULTING • ENGINEERING • PLANNING

LIST OF APPENDICES

APPENDIX	TITLE
A	Deed Restriction
B	Inspection Checklist
C	Well Location & Surface Water Drainage Map
D	SOP Groundwater/Surface Water Sampling
E	TestAmerica Quality Assurance Manual

OPERATION MONITORING AND MAINTENANCE MANUAL
CELLS 1 AND 2

FOR
CC METALS AND ALLOYS, LLC
WITMER ROAD

1.0 INTRODUCTION

The following provides a post-closure maintenance and monitoring plan for the CC Metals and Alloys, LLC (CCMA) landfill Cells 1 and No. 2. These facilities are located at a 9.76 acre site adjacent to Witmer Road in the Town of Niagara. Waste disposed in Cell 1 includes ferrosilicon and ferrochromium metal baghouse dusts and waste disposed in Cell 2 contains ferroalloy dust.

Cell 1 was constructed in 1980 per a New York State Department of Environmental Conservation (NYSDEC) Part 360 Permit (#2133). It was closed in 1990 per a NYSDEC approved closure plan. Cell 2 was constructed in 1983 per a NYSDEC Part 360 Permit (#2585). Per NYSDEC Order on Consent 87-152A waste deposition into Cell 2 was stopped on September 30, 1991. Cell 2 was closed in 1992.

The principal objective of this manual is to provide the necessary instructions for the following:

1) Proper maintenance of all facility components,

2) Groundwater and surface water sampling and analysis, and

3) Interpretation of ground and surface water monitoring data. Adherence to this post-closure monitoring and maintenance program is required by 6 NYCRR Part 360 for a minimum period of thirty (30) years after final closure of Cells 1 and 2.

The information provided in this post-closure monitoring and maintenance operations manual is utilized by CCMA personnel and its consultants.

2.0 PROCEDURE FOR AMENDING POST-CLOSURE MONITORING AND MAINTENANCE OPERATIONS MANUAL

This post-closure monitoring and maintenance operations manual should be reviewed at regular intervals (initially once every three years) to ensure that it remains consistent with both the regulations and the technology concerning post-closure monitoring and maintenance at the Witmer Road site. All necessary modifications will be made under the

direction of a professional engineer licensed in the State of New York.

Since this plan (after approval) will be incorporated as a binding agreement between CCMA and the NYSDEC, any proposed modifications to this plan will be submitted to the NYSDEC for approval.

Upon receipt of NYSDEC approval, the changes will be made and the updated plan will be placed on file at the CC Metals and Alloys, Amherst, New York, office.

3.0 POST-CLOSURE MAINTENANCE REQUIREMENTS

The goals of the post-closure maintenance plan for the CCMA, Witmer Road Site, are as follows:

1) Ensure that structural integrity of closed Cells 1 and 2 is being properly maintained.

2) Correct any problems that might occur at the site before they have a chance to develop to such a degree that adverse environmental impacts might result.

3) Follow a program in which all involved parties (CCMA, regulatory agencies, and the public) have a sense of confidence that the site will not create problems which cannot be reasonably handled with minimum impacts.

4) Properly maintain the drainage pathways and controls implemented under the Interim Remedial Measure (IRM) order established in 1999.

5) Annual certification of the Deed Restriction (Institutional Control) filed with the Niagara County Clerk and recorded May 3, 2001, in Book 3114 on Page 291, ensuring it is still in effect and has not been altered. A copy of the Deed Restriction is included as Appendix A.

The post-closure maintenance plan can be summarized as follows:

1) LAN Associates, Inc. will be responsible for filing a Waste Management Facility Maintenance Inspection Report. Included in this inspection report will be a checklist which covers the following annual evaluation:

    a) Bank and cover erosion,
    b) Settlement,
    c) Cover soil integrity,
    d) Condition of vegetative cover, and
    e) Condition of monitoring wells.

2) If any problems are encountered during the inspections that may be of significant environmental concern, the necessary corrective actions will be undertaken as expeditiously as possible. Notice of these actions will be reported to the NYSDEC explaining the nature and location of the problem and the corrective action taken.

Post-closure maintenance requirements are expected to be minimal. However, areas where some maintenance may be necessary include landfill cover, berms, surface water drainage ditch, and groundwater monitoring wells.

Adequate information is not available to actually calculate how much subsidence will occur with Cells 1 and 2, however, only an insignificant amount of subsidence is expected. This is based on the results from compaction tests previously done on waste materials contained in Cells 1 and 2. In addition, the materials contained in these cells will not undergo any decomposition. Slopes utilized in the closures of Cells 1 and 2 will ensure that their final slope, after settling and subsidence, will be greater than three percent. A slope greater than three percent will allow for adequate surface water runoff rates.

Any deficiencies noted either during the sites scheduled or unscheduled inspections will be corrected as expeditiously as possible. While each situation must be evaluated on a case by case basis, a plan of action has been prepared to deal with those situations which are most likely to occur.

Landfill cover deterioration should be minimal. However, some will undoubtedly occur due to freeze-thaw effects, water erosion, etc. Such deterioration must be corrected as quickly as possible.

The vegetative growth covering the closed cells will be allowed to return to its natural state. The vegetative cover on the landfill cells as well as the drainage areas will be mowed once per year between September 1st and December 31st. If significant bare spots should develop, an attempt will be made to determine the cause. Factors which will be considered include the presence of excessive moisture, excessive dryness, wrong pH, or the absence of the proper soil nutrients. When the cause is determined, remedial action will be taken.

Both wind and water erosion of the landfill cover can occur. While this is not expected to be a significant problem, any erosion which does occur must be taken care of expeditiously. Repair will bring lines and grades to their original configuration. If the erosion can be attributed to inadequate original design, the necessary design modifications will be made and implemented (after receipt of NYSDEC approval). Future modifications could include changes in slope gradients or protection of slopes by riprap.

The facility's annual report will include notations concerning both scheduled and

unscheduled facility inspections. Inspections will be performed on an annual basis. An inspection checklist created specifically for the property will be used when performing the inspections. A copy of the inspection checklist is included as Appendix B. Annual inspections are appropriate because the landfill has been closed for 16 years with no disruption to the integrity of the system. Information will include the date and time of the inspection, inspector's name, and a summary of all problems observed and remedial actions taken.

Records of all inspections will be retained for a minimum period of seven (7) years (see Appendix B, Inspection Checklist). In addition, summary reports and records of all incidents requiring initiation of the site's contingency plan or resulting in human health or environmental damage will be prepared and maintained for a minimum period of seven (7) years.

It is important to note that the drainage system on the property is protected by a Deed Restriction. The Deed Restriction serves as a covenant for the land that binds all future property owners. Therefore, any person wishing to engage in any activity on the property that could interfere significantly with the completed closure and remedial program is required to obtain written approval from the NYSDEC and the New York State Department of Health, or any New York State agency created to protect the environment. A copy of the Deed Restriction is included in Appendix A.

4.0 POST-CLOSURE GROUNDWATER AND SURFACE WATER SAMPLING AND ANALYSIS PLAN

The following provides a post-closure site groundwater and surface water sampling and analysis plan for the Witmer Road landfill site. Its primary objective is to provide data relating to the site's groundwater and surface water quality during the solid waste management facility's post-closure period.

Factors which were given consideration in the design of this plan include the following:

1) Ground and surface water monitoring requirements at a non-hazardous waste management landfill facility as stipulated in 6 NYCRR Part 360 Solid Waste Management Facilities (effective December 31, 1988),

2) Physical and chemical characteristics of waste materials deposited in Cells No. 1 and 2,

3) Site's hydrological conditions,

4) Pollution potential of site as exemplified by the type of waste materials present, and

5) Groundwater use.

Items which are addressed in this post-closure groundwater and surface water sampling and analysis plan include the following:

1) Locations and construction of monitoring points,
2) Discussion of monitoring frequency and parameters,
3) Sampling personnel and equipment requirements,
4) Sampling procedures,
5) Sample handling,
6) Analytical procedures,
7) Laboratory quality assurance plan,
8) Data analysis,
9) Contingency monitoring requirements, and
10) Data reporting requirements.

By developing and implementing a comprehensive, site specific groundwater sampling and analysis program the potential for problems to arise when obtaining, handling, preserving, and analyzing samples will be minimized.

4.1 LOCATION AND CONSTRUCTION OF MONITORING POINTS

The post-closure monitoring program for Cells 1 and 2 includes groundwater and surface water monitoring. Implementation of this program during the facility's post-closure period will provide the required data to evaluate the effects of Cells 1 and 2 on both the site's ground and surface water. A series of five (5) wells will be utilized to monitor the quality of the groundwater contained in the permeable sediments overlying the bedrock. These wells were utilized to monitor the effects of Cells 1 and 2 on the site's groundwater during the operation of these facilities. Based upon previous data from these monitoring wells, groundwater flows in a southerly direction across the site. Surface water quality will be monitored using samples obtained from the site's drainage ditch.

4.1.1 Monitoring Well Location and Construction

Sample points (wells) 3R, 5R, 12, BR1, and 14N are indicated on the Well Location and Surface Water Drainage Map showing baseline locations, monitoring well elevations, and surface water drainage patterns (Appendix C). Based upon the site's previously noted groundwater flow direction (southerly), monitoring well 3R can be used to provide upgradient data while monitoring wells 5R, 12, BR1, and 14N provide data on

groundwater quality downgradient of the site's disposal areas (Cells 1 and 2).

"It has been reported that the wells are installed at the depth of refusal. Well #12 is constructed of 4-inch PVC with the lower two feet slotted with 1/16 inches wide horizontal slots spaced approximately 1 inch apart. The slots are covered with a stainless steel well screen. A sand pack was placed from the bottom of the well upward for approximately five feet. Bentonite pellets were utilized to provide a seal at the clayey-silt level. Loose bentonite was then placed around the monitoring well through most of the impervious lake sediment zone to the surface to prevent water seepage from the "perched" water table. Monitoring wells 3R, 5R, BR1, and 14N are constructed of 2-inch PVC risers attached to 5-foot lengths of PVC 10 slot screen. The PVC screens were installed immediately above the dense loamy glacial till which overlies the site's bedrock. The screen interval and associated sand column surrounding the screens extend partially above the screens. Bentonite pellet seals were utilized to separate the sand pack from the cement-bentonite grout seal. Each well casing is surrounded at the ground surface by a continuous pour concrete cap and well apron (minimum radius of 3 feet and minimum thickness of 4 inches)."[1]

4.1.2 Surface Water Monitoring Points

Cell 1 closure resulted in all waste materials being covered with a minimum of 18 inches of low permeability compacted soil (maximum permeability of 1.0 x 10 cm/sec) and 6 inches of soil capable of supporting vegetative growth. It is reported that Cell 2 was similarly closed. It is very unlikely that surface water runoff from the closed facilities has any contact with the waste materials previously deposited in Cells 1 and 2. However, samples will be taken from the location of the discharge flow control valve (SW-1) located in the southwest corner of site where surface water collects and flows into the stormwater drainage pipe and then offsite to the City of Niagara Falls sewer system.

4.2 MONITORING FREQUENCY AND PARAMETERS

Groundwater sample points will include monitoring wells 3R, 5R, 12, BR1, and 14N. Based upon an isopotential map of the site's groundwater, monitoring well 3R will provide upgradient data while monitoring wells 5R, 12, BR1, and 14N will provide data on groundwater quality downgradient of Cells 1 and 2. Surface water sampling will be performed at point SW-1. In addition, samples will be

[1] Original Post Closure Monitoring and Maintenance Operations Manual by Snyder Engineering 1991

obtained from the landfill leachate sump (LS-1). Site monitoring frequency will be on a semi-annual basis. Samples will be analyzed on a semi-annual basis for routine parameters; specific conductivity, temperature, pH, Eh, turbidity, COD, TOC, TDS, SO4, Cl, Br, Pb, Mn, K, and Na. In addition, semi-annual samples will be analyzed for baseline parameters; As, Ba, Cr, Cr+6, Hg, Se, and B. Annual samples will be obtained for Volatile Organic Compounds (VOCs) that are specified in the New York State Regulation 6 Part NYCRR 360 baseline parameter list. The laboratory analytical method for the VOCs is SW-846 method 8260.

4.3 SAMPLING PERSONNEL AND EQUIPMENT REQUIREMENTS

The laboratory utilized to implement the site's post-closure groundwater and surface water monitoring program must be approved by the NYSDEC. The laboratory must be approved to perform the required analyses for all parameters of concern. All sampling personnel must be properly trained in the collection and handling of groundwater and surface water samples. They must be familiar with all equipment required to collect a representative sample of groundwater from wells such as those present at the Witmer Road site. Sampling personnel must have a minimum two years of technical training in chemistry, environmental science, or other technical discipline. This educational requirement may be waived for personnel with a minimum of five years experience in the collection of environmental samples.

4.4 SAMPLING PROCEDURES

Standard Operating Procedure (SOP) No. BR-FS-005, Groundwater/Surface Water Sampling is included in Appendix D. The procedure for the sampling of the sump (LS-1) is performed under the standard operating procedures outlined in Appendix D. The actual sample itself is obtained through the use of a bailer dropped down into the sump.

4.5 LABORATORY QUALITY ASSURANCE PLAN

The primary objective of the Quality Assurance Plan for CCMA groundwater and surface water monitoring program is to ensure that the analytical results obtained from the program are reliable, statistically valid, and properly documented. As previously noted, CCMA will only utilize a laboratory for program implementation which has been approved by the NYSDEC. The basis of this quality assurance program is the establishment of methods which will be followed in obtaining the analytical results for each sample. Procedures (including quality assurance samples, replicates, spikes, and standards calibration) will be established and used for validating the methods utilized by the analytical laboratory and as an indicator of potential sources of cross-contamination. This will help ensure that the laboratory generates precise, accurate, and reliable data.

Test America Laboratories, Inc. located in Buffalo, New York, is currently the laboratory chosen to perform the sampling. A complete quality assurance manual for Test America is included in Appendix E.

4.5.1 Personnel Responsibilities

LAN Associates, Inc. will be responsible for ensuring that the required groundwater and surface water monitoring program at the Witmer Road site is correctly carried out. Their responsibilities will include the following:

1) Overall responsibility for management of the analytical program and validity of all data,

2) Selection of an analytical laboratory to perform sample analyses,

3) Performance monitoring of analytical laboratory and review of all analytical protocols required for measuring and monitoring,

4) Submission of all analytical data to New York State Department of Environmental Conservation, Town of Niagara, and Niagara County Health Department.

A project coordinator is to be designated by the analytical laboratory. This individual is to have responsibility for the following:

1) Communication with CCMA Environmental Manager or designated representative regarding the groundwater and surface water analysis program,

2) Monitor sampling and/or analytical techniques and recommend modifications as required,

3) Verify that laboratory quality control and analytical procedures are being followed as specified in the Quality Control Plan when laboratory personnel are analyzing CCMA groundwater and surface water samples,

4) Review raw analytical data and check arithmetic calculations for a minimum of 20% of the samples analyzed (includes inspection of reduced data, calibration curves and bound laboratory notebooks),

5) Receive groundwater and surface water samples at the laboratory and verify that incoming samples correspond to the chain of

custody sheet,

6) Maintain records of all incoming samples and track samples while they are being processed,

7) Prepare quality control samples for analysis as required to satisfy quality assurance requirements,

8) Approve completed data and analytical report before transmittal to CC Metals and Alloys, LLC.

A sampling coordinator is to be designated by the analytical laboratory. This individual is to have responsibility for the following:

1) Determine appropriate sampling equipment and sample containers,

2) Train field personnel in the necessary sampling and field analytical procedures,

3) Insure that all samples are collected, labeled, preserved, and stored as specified in other sections of this report,

4) Check that all required sample documentation is correct and is transmitted with the samples,

5) Check on field sampling to insure that it is being done correctly.

4.5.2 Analytical Quality Assurance

Specific analytical methods often prescribe the necessary specific quality assurance procedures. In order to achieve a high degree of accuracy (degree of measurement or average of measurements agreement with an accepted reference or true value obtained from executing a method in a particular laboratory using an interference free matrix), the laboratory must do the following:

1) References used as reference standards must be the highest purity commercially available materials and must be certified by the supplier.

2) Each instrument utilized in performing the analyses must be checked on each day that the samples are run in order to demonstrate performance.

3) Recovery factors for individual contaminants are determined for the analytical method which is utilized.

4) Analytical results for spiked level of the contaminant under evaluation in a replicate sample must be within the required limits for the contaminant under evaluation.

Full documentation of all analyses must be kept in notebooks and be available for inspection at the designated laboratory by either a representative of CCMA or the NYSDEC.

4.5.3 Data Validation and Reporting

The principal steps that will be used to verify the data integrity during data collection and reporting are as follows:

1) Project coordinator will review raw data generated by the laboratory chemist. It will be reviewed against calibration and quality control records, to ensure both the adequacy of documentation and the reliability of the data.

2) When the previously noted review has been completed, the data will be considered validated and a report will be prepared for submission to CCMA.

3) All laboratory notes and records will be maintained and stored in an accessible place.

A variety of samples will be analyzed at regular intervals to assess possible contamination from either the field and/or the laboratory. These include blank, spiked, and replicate samples. Blank samples include:

1) Field blanks are exposed to field and sampling conditions and analyzed in order to assess possible contamination from the field. A bottle is filled with de-ionized water and is transported to the sampling location and is returned to the laboratory in a manner identical to the handling procedure used for the samples.

2) Method blanks are prepared in the laboratory and are analyzed in order to determine the background of each of the reagents or solvents used in an analysis.

Spiked samples will be spiked (as prescribed by the analytical method) with one or more selected compounds prior to extraction and analysis. Concentration data will be used to calculate the recovery of the compounds. Such samples will provide a measure of sample preparation and analysis procedures accuracy.

Replicate samples are analyzed in order to establish control and assess the precision of an analysis and/or of sampling. Field replicates are obtained in order to assess the adequacy of overall sampling and handling procedures. Laboratory replicates are prepared in the laboratory and analyzed in order to assess the reproducibility of the laboratory procedures used.

4.6 CONTINGENCY MONITORING REQUIREMENTS

All waste materials which have been deposited by CCMA Cells 1 and 2 at the Witmer Road site were approved by the NYSDEC. In the unlikely event that significant groundwater contamination is detected, a contingency plan will be enacted. Objectives of this groundwater contingency plan will be as follows:

1) Confirm whether significant quantities of contaminants have entered the groundwater at the CCMA Witmer Road site from the waste materials previously deposited by CCMA in Cells No. 1 and 2,

2) If significant quantities of contaminants have entered the groundwater, determine their consequences and the rate and extent of their migration.

Under normal circumstances, Objective #1 will be satisfied by the site's groundwater monitoring program as previously described. However, if a statistical analysis of monitoring data from upgradient and downgradient wells utilizing the Student's t-test at the 0.01 level of significance indicates a significant difference in groundwater quality, additional samples will be obtained and analyzed. If the difference cannot be attributed to sampling or analytical errors, a written notice that the facility may be affecting the groundwater must be sent within 14 days to Region 9 of the NYSDEC.

During the next semi-annual sampling event, each monitoring well involved in triggering the contingency monitoring plan will be sampled and analyzed for the baseline parameters as defined by Water Quality Analysis Table in 6 NYCRR Part 360-2.1 1(c)(6). Every attempt will be made to report the analytical results to the NYSDEC within 30 days after the sampling date. In any case, the results will be reported to the NYSDEC within 14 days after receipt of results from the certified analytical laboratory.

In the event that the NYSDEC determines that any potential contamination as reflected by the baseline monitoring results poses an immediate threat to public health or the environment, CCMA will provide the NYSDEC with a corrective action plan. Upon receipt of plan approval from the NYSDEC, CCMA will implement the corrective action plan.

When the corrective action plan is implemented, the sampling and analysis for baseline parameters will be performed at least semi-annually until the conditions for curtailing contingency water quality monitoring are satisfied as follows:

1) Elevated parameter(s) is demonstrated not to be landfill derived, or
2) Remediation of release by landfill is demonstrated to be complete.

In addition, the contingency water quality monitoring may be reduced or discontinued with the approval of the NYSDEC, if such monitoring is no longer necessary to protect public health or the environment.

If during analysis for baseline parameters, contamination by any toxic metal, cyanide, volatile organic compound, or other substance identified in Appendix 33 of 6 NYCRR Part 373-2 occurs, CCMA will sample the appropriate environmental monitoring points in the next scheduled sampling event after receiving the analytical results from the laboratory. Each sample will be analyzed for all the expanded parameters listed in the Water Quality Analysis Table. Unless the NYSDEC requires more frequent sampling to evaluate a potential or adverse environmental impact or perceived health risk or until the previously noted conditions for curtailing contingency water quality monitoring are satisfied, subsequent annual analyses of these monitoring points will include all routine parameters and those baseline and expanded parameters that were elevated or were implicated in the expected pattern.

4.7 REPORTING AND RECORDKEEPING REQUIREMENTS

Copies of all semi-annual monitoring reports will be sent to the following:

1) Ms. Mary McIntosh
   Senior Engineering Geologist
   New York State Department of Environmental Conservation
   Region 9
   270 Michigan Avenue
   Buffalo, New York 14203-2999

2) Town of Niagara
   7105 Lockport Road
   Niagara Falls, New York 14305

In addition, CCMA will prepare and submit an annual summary report concerning facility post-closure maintenance and monitoring. This report will be certified by a Professional Engineer registered in the State of New York. It will be submitted to the NYSDEC Region 9 Solid Waste Regional Engineer no later than 60 days after the first day of January each year. These records will be retained for a minimum period of seven years.

Analytical data records which will be retained during the post-closure period include the following:

1) All chemical analyses of waste materials,

2) All EP toxicity and TCLP test data performed on waste material samples,

3) All chemical analyses and associated monitoring well elevations obtained as part of the site's groundwater and surface water monitoring program.

Appendix A

CCMA
CC Metals and Alloys, Inc.

P.O. Box 217 Calvert City, KY 42029
Phone: (270) 395-7631

May 16, 2001

CERTIFIED MAIL RETURN RECEIPT REQUESTED
7000 1530 0001 4920 0042

Mr. John P. Cahill, Commissioner
New York State Department of
Environmental Conservation
50 Wolf Road
Albany, New York 12233-1010

Re: Declaration of Covenants and Restrictions For The Petition to Delist
CC Metals and Alloys, Inc.'s (formerly SKW Metals & Alloys, Inc.) Portion of
Vanadium Corporation of America Inactive Hazardous Waste Site No: 932001

Dear Mr. Cahill:

CC Metals and Alloys, Inc., formerly known as SKW Metals and Alloys, Inc. (CCMA or SKW), owns a portion (SKW Property) of the "Vanadium Corporation of America" (Vanadium Site) site No. 932001, which is listed in the New York State Department of Environmental Conservation's (Department) Registry of Inactive Hazardous Waste Disposal Sites (Registry). A Petition to Delist the SKW Property from the Vanadium Site Registry listing was submitted to you on February 26, 2001. As provided on Page 5 of the Petition, CCMA filed a Declaration of Covenants and Restrictions (Declaration) with Niagara County on April 30, 2001. The Declaration was recorded by Niagara County on May 3, 2001. A copy of the recorded Declaration is attached to this letter (Attachment 1) so that it can be included with the Petition to Delist. It is our understanding that submittal of the Declaration will fulfill our commitment as stated on page 5 of the Petition.

Sincerely,

Edward S. Bredniak
President

ESB:dl
2.3269.23-L-Delisting-010511-esb

Attachments: #1–Recorded Declaration of Covenants and Restrictions

Copies to: Mr. William Shaw - NYSDEC Albany Bureau of Hazardous Site Control (3 copies)
Mr. Michael Hinton - NYSDEC Region 9
Mr. Guy Van Doren - LAN Associates, Inc.
Mr. Paul D. Meosky - Hodgson and Russ, LLP

DO NOT DETACH - THIS IS PAGE 1 OF RECORDED DOCUMENT

NIAGARA COUNTY CLERK RECORDING PAGE

OFFICE OF THE CLERK COUNTY OF NIAGARA
WAYNE F. JAGOW, COUNTY CLERK
County Courthouse, 175 Hawley Street, P.O. Box 461, Lockport, NY 14095
Phone (716) 439-7027 Fax (716) 439-7066

INSTRUMENT DATE April 30, 2001

DOCUMENT TYPE Declaration of Covenants and Restrictions

(10)

Parties: (Print Names In Full)

1st Part CC Metals & Alloys

2nd Part _____

Town/City Niagara

Return To:

Paul Meosky
Hodgson Russ LLP
One M&T Plaza Suite 2000
Buffalo, NY 14203

ORIGINAL FILED

MAY 3 - 2001

WAYNE F. JAGOW
NIAGARA COUNTY CLERK
LOCKPORT, NEW YORK 14094

DOCUMENT # 1003276
BOOK 3114 PAGE 291
NUMBER OF PAGES 10
RECORDED 05/03/2001
04:27:33 P.M.
RECEIPT # 10039
PAID - COUNTY CLERK
WAYNE F. JAGOW

THIS SPACE RESERVED FOR COUNTY CLERK

MORTGAGE# _____

MORTGAGE AMOUNT
$ _____

(___)One\two family (___)Other

[ ] Check if to be apportioned

RECORDING TAX RECEIPT

BASIC $ _____
ADDITIONAL $ _____
SPECIAL $ _____
TOTAL $ _____

State of New York} ss
County of Niagara}
I do hereby certify that I have
Received on the within Mortgage, being
the amount of the Recording Tax
Imposed thereon & paid at recording.

#_____
REAL ESTATE TRANSFER TAX

$ _____

__/__/__

NIAGARA COUNTY

Dated _____, 20___

Mortgage Tax Clerk of Niagara County

This sheet constitutes the Clerk's endorsement required by section 319 of the Real Property Law of the State of New York

DO NOT DETACH

DECLARATION OF COVENANTS AND RESTRICTIONS

This Declaration of Covenants and Restrictions, made this 30th day of April 2001, by CC Metals and Alloys, Inc. ("CCMA"), (formerly known as SKW Metals and Alloys, Inc.), a corporation organized and existing under the laws of the State of Delaware and having an office for the transaction of business at 300 Corporate Parkway, Amherst, New York.

RECITALS

WHEREAS, CCMA owns a portion of the Vanadium Corporation of America site ("Vanadium Site"), listed as Site No. 932001 in the New York State Department of Environmental Conservation's ("NYSDEC") Registry of Inactive Hazardous Waste Disposal Sites; and

WHEREAS, CCMA's portion of the Vanadium Site is located on Witmer Road in the Town of Niagara, County of Niagara, State of New York, which is more particularly described in Appendix A attached to this Declaration of Covenants and Restrictions and made a part hereof, and hereinafter referred to as the "Property"; and

WHEREAS, a landfill occupies a part of the northern portion of the Property, such landfill was properly closed in 1992-1993 in accordance with NYSDEC regulations and the NYSDEC approved the landfill closure in 1994; and

WHEREAS, in response to the NYSDEC's inclusion of the Vanadium Site on the Registry, CCMA entered into an Order on Consent in 1998 with the NYSDEC, Index No. B9-

0470-94-12, a copy of which was attached to and made a part of a Declaration of Covenants and Restrictions which was recorded in the Niagara County Clerk's Office on July 30, 1998 in Liber 2848 at page 25; and

WHEREAS, pursuant to the Order on Consent, CCMA undertook remedial measures to address conditions in an area in the southeast portion of the Property, which measures included regrading to (i) eliminate off-site surface water runoff from entering the Property, (ii) isolate on-site stormwater to prevent contact with underlying soil and groundwater, (iii) produce a site drainage system for the Property to control stormwater discharge from the Property, and (iv) eliminate on-site low lying areas where surface water could accumulate; and

WHEREAS, the NYSDEC approved the remedial measures completion report in a letter dated January 13, 2000, and the Declaration of Covenants and Restrictions recorded at the Niagara County Clerk Office on July 30, 1998 automatically terminated upon satisfaction of the obligations imposed under the Order on Consent; and

WHEREAS, CCMA in February 2001 submitted to the NYSDEC a Petition to Delist the Property from the Registry, and the NYSDEC has requested that this Declaration of Covenants and Restrictions be filed with the Niagara County Clerk.

WHEREAS, documentation of the landfill closure, Order on Consent, remedial measures and Petition to Delist the Property from the Registry are part of the public record kept in the NYSDEC's Region 9 office in Buffalo.

NOW, THEREFORE, CCMA, for itself and its successors and/or assigns, covenants that:

First, the Declaration of Covenants and Restrictions recorded in the Niagara County Clerk's Office on July 30, 1998 was automatically terminated of record with the approval by the NYSDEC of the remedial measures completion report (see Appendix B for NYSDEC's approval letter dated January 13, 2000).

Second, the Property, more particularly described in Appendix A, is subject to this Declaration of Covenants and Restrictions.

Third, unless prior written approval by the NYSDEC and the New York State Department of Health, or any New York State agency or agencies subsequently created to protect the environment of the State and the health of the State's citizens, is first obtained, no person may engage in any activity, including regrading, that will, or that reasonably is anticipated to, interfere significantly with any completed closure or remedial program affecting the Property, including any activity that will result in the disturbance or excavation of the on-site landfill or contour of the Property including the drainage system.

Fourth, this Declaration is and shall be deemed a covenant that shall run with the land and shall be binding upon all future owners of the Property.

Fifth, any deed of conveyance regarding the Property shall recite that the said conveyance is subject to this Declaration of Covenants and Restrictions.

IN WITNESS WHEREOF, CCMA has executed this instrument on this 30TH day of April, 2001.

CC METALS AND ALLOYS, INC.

By: Edward S. Bredniak
Edward S. Bredniak
President

COMMONWEALTH OF KENTUCKY)

: SS.

COUNTY OF MARSHALL)

On the 30 day of April, in the year 2001, before me, the undersigned, personally appeared EDWARD S. BREDNIAK, personally known to me, or proved to me on the basis of satisfactory evidence to be the individual whose name is subscribed to the within instrument and acknowledged to me that he executed the same in his capacity, and that by his signature on the instrument, the individual, or the person upon behalf of which the individual acted, executed the instrument, and that such individual made such appearance before the undersigned in the City of CALVERT City; Commonwealth of Kentucky.

Marlene Latta
Notary Public

My commission expires January 27, 2004

APPENDIX A

That Tract or Parcel of Land situate in the Town of Niagara, County of Niagara and State of New York, being part of Lot 24, Township 13, Range 9 of the Holland Land Company's Survey, bounded and described as follows:

BEGINNING at a point in the center line of the Witmer Road at the southwest corner of lands conveyed by the Niagara Falls Power Company to the Vanadium Corporation of America by deed recorded in the Niagara County Clerk's Office in Liber 660 at Page 319.

Running thence easterly along the southerly line of lands so conveyed, a distance of 2264.89' to a point.

Running thence northerly at right angles to the last previous course, a distance of 295.2' to a point.

Running thence northwesterly on a line deflecting to the left 67° 47' 54" from the last previous course, a distance of 105.52' to the point of curve.

Running thence northwesterly and westerly on a curve to the left, said curve having a radius of 326.5', an arc distance of 135.47' to the point of tangency.

Running thence westerly along said line of tangency, a distance of 51.0' to a point.

Running thence northwesterly, on a line deflecting to the right 53° 00' 55" from the last course, a distance of 284.0' to a point.

Running thence northerly, on a line deflecting to the right 38° 10' 30" from the last previous course, a distance of 483.75' to a point on the southerly line of lands appropriated by the Power Authority of the State of New York as shown on Power Authority of the State of New York Map No. 1295, Parcel 1295.

Running thence westerly at right angles to the last previous course and along the southerly line of lands appropriated by the Power Authority of the State of New York as aforesaid, a distance of 906.56' to the center line of the Witmer Road.

Running thence southwesterly, along the center line of the Witmer Road, said center line being also the Mile Line, a distance of 1386.83' to the point of beginning.

EXCEPTING and reserving a triangular parcel of land in the southwest corner of the above described parcel conveyed to the Niagara Mohawk Power Corporation by deed recorded in Liber 1352 at Page 358, August 31, 1960 described as follows:

ALL THAT TRACT OR PARCEL OF LAND situate in the Town of Niagara, County of Niagara and State of New York, being part of Lot 24, Township 13, Range 9 of the Holland Land Company's Survey, bounded and described as follows:

BEGINNING at the point of intersection of the easterly line of Witmer Road and the southerly line of lands conveyed to Niagara Falls Power Company by deed dated March 22, 1940 recorded in Niagara County Clerk's Office in Liber 660 of Deeds page 319; thence northerly along the easterly line of Witmer Road 80.40 feet to a point; thence southerly along a line which is at right angles to the southerly line of lands conveyed as aforesaid 61.32 feet to a point; thence westerly along said southerly line 52 feet to the point of beginning;

ALSO EXCEPTING therefrom the premises described as follows:

ALL THAT TRACT OR PARCEL OF LAND, situate in the Town of Niagara, County of Niagara and State of New York, being part of Lot No. 24, Township 13, Range 9 of the Holland Land Company's Survey, bounded and described as follows:

BEGINNING at a point in the center line of Witmer Road at the southwest corner of land conveyed to Vanadium Corporation of America by deed recorded in liber 660 of Deeds at page 319; thence easterly along the south line of land so conveyed a distance of 836.5 feet; thence northerly at a right angle a distance of 598 feet; thence westerly at a right angle a distance of 329.24 feet to the center line of Witmer Road; thence southwesterly along the center line of Witmer Road, said center line being also the Mile Line, a distance of 784.17 feet to the point of beginning.

New York State Department of Environmental Conservation
Division of Environmental Remediation, Region 9
270 Michigan Avenue, Buffalo, New York, 14203-2999
Phone: (716) 851-7220 • **FAX:** (716) 851-7226
Website: www.dec.state.ny.us

John P. Cahill
Commissioner

JAN 17 2000

January 13, 2000

Mr. Edward Bredniak
CC Metals and Alloys Inc.
P.O. Box 217
Calvert City, Kentucky 42029

Dear Mr. Bredniak:

Vanadium Corporation of America
Site #932001
SKW Operable Unit 1
Revised IRM Completion Report

The New York State Departments of Environmental Conservation (NYSDEC) and Health (NYS DOH) have reviewed the IRM Completion Report prepared by LAN Associates and dated November 11, 1999. This report is acceptable to the NYSDEC and represents the completion of the agreed upon IRM program.

With the completion of the IRM program, all costs incurred by the state during the implementation of this program must be reimbursed as indicated in the IRM consent order. The NYSDEC will send an itemized bill to you in the near future.

The Department requests the addition of the surface water discharge point and monitoring well BR-1 to the existing Part 360 quarterly sampling program for the facility, and that these points be sampled on the same schedule and for the same parameters as the other monitoring points. If you have any questions regarding the Part 360 sampling requirements please call Ms. Mary McIntosh at (716) 851-7220.

Finally, with the completion of the IRM program a long-term maintenance plan must be prepared to ensure the continued effectiveness of the IRM. An Operation and Maintenance Plan must be prepared that includes a deed restriction to restrict the future use of the property and provides a schedule for the mowing of the site, routine inspections to identify and correct areas that may settle, erode or are damaged by burrowing animals or site activities. The deed restriction must be filed in the Niagara County Clerks office to advise future owners of the concerns associated with this site.

The NYSDEC is pleased with the IRM program implemented by CC Metals and Alloys, Inc. which accomplished the goals established for the IRM. Please keep in mind the fact that the SKW property is just one of three operable units established for the Vanadium Corporation of America site. The NYSDEC is negotiating with the other property owners to address the issues associated with the remaining parcels. Following completion of work at the individual parcels, the NYSDEC intends to prepare a Proposed Remedial Action Plan (PRAP) and Record of Decision (ROD) that will summarize the remedial activities at the Vanadium site for all the operable units and recommend any further work if needed.

If you have any questions, please call me at (716) 851-7220.

Sincerely,

Michael J. Hinton, P.E.
Environmental Engineer II
Division of Environmental Remediation
Region 9

MJH:tml

cc: Mr. Daniel King, NYSDEC, Division of Environmental Remediation, Region 9
Maura Desmond, Esq., NYSDEC, Division of Environmental Enforcement, Buffalo
Ms. Karen Mauirano, NYSDEC, Division of Environmental Remediation, Albany
Ms. Dawn Hettrick, NYS DOH, Albany
Ms. Mary McIntosh, NYSDEC, Division of Solid and Hazardous Materials, Region 9
Mr. Guy Van Doren, P.E. LAN Associates

Appendix B

LAN ASSOCIATES, INC.
CONSULTING • ENGINEERING • PLANNING

Date _____

Weather Conditions _____

Inspector _____

CC Metals and Alloys, LLC
Witmer Road Landfill Inspection Checklist

General Instructions

The inspector should note the various observations he/she makes under the various sections and questions. If any corrective actions need to be taken, they will be noted on the Checklist Of Recommended Corrective Actions, Page 4 of 4. If any unusual conditions are encountered during the inspections, they should be reported to the engineer (LAN Associates, Inc., 66 Cuna Street, St. Augustine, FL 32084, 904-824-6999).

Landfill Cover

1) Observe any areas on the cover that indicate signs of subsidence (e.g., obvious visible low spots on the cover surface where significant amounts of standing water can accumulate in puddles during significant precipitation events, check for the presence of large cracks on the surface of the cover, etc).

2) Check for erosional swales, washouts, etc. in the landfill cover caused by stormwater runoff. During windy conditions, observe any evidence of dust blowing off the cover.

3) Inspect landfill vegetative cover for overall health and consistency. (e.g. check for bare spots in the vegetative cover.)

4) Inspect vegetative cover for existence of unwanted woody species or the abnormal growth of weeds that may out-compete the natural vegetation.

Monitoring Wells and Sampling Locations

1) Check the general condition of the individual monitoring wells; make sure the bollards are intact (have not been knocked over by a vehicle), check for cracks on the concrete pad (monitor any minor cracks to ensure they do not widen and compromise the pad's integrity otherwise repairs may be necessary), make sure that the padlocks are in working condition (not stiff when unlocking the padlock), make sure that the plug on the PVC riser is present and that the threads are in good condition.

2) Inspect the drainage flow control valve and piping system for functionality and condition (SW-1).

3) Inspect the sump collection tank for cracks or any visible problems that may effect the integrity of the system (LS-1).

Surface Water Drainage

1) Inspect the overall function of the surface water drainage system. Look for signs of erosion or subsidence that could lead to offsite surface water drainage or pooling water onsite.

2) Check all stormwater drainage systems (e.g. piping, manholes, drains) for overall function. Make sure there are no blockages or diversions.

Property

1) Check the condition of fences and gates throughout the property.

2) Conduct a thorough investigation of the entire site for any areas of concern.

LAN ASSOCIATES, INC.
CONSULTING • ENGINEERING • PLANNING

Date _____

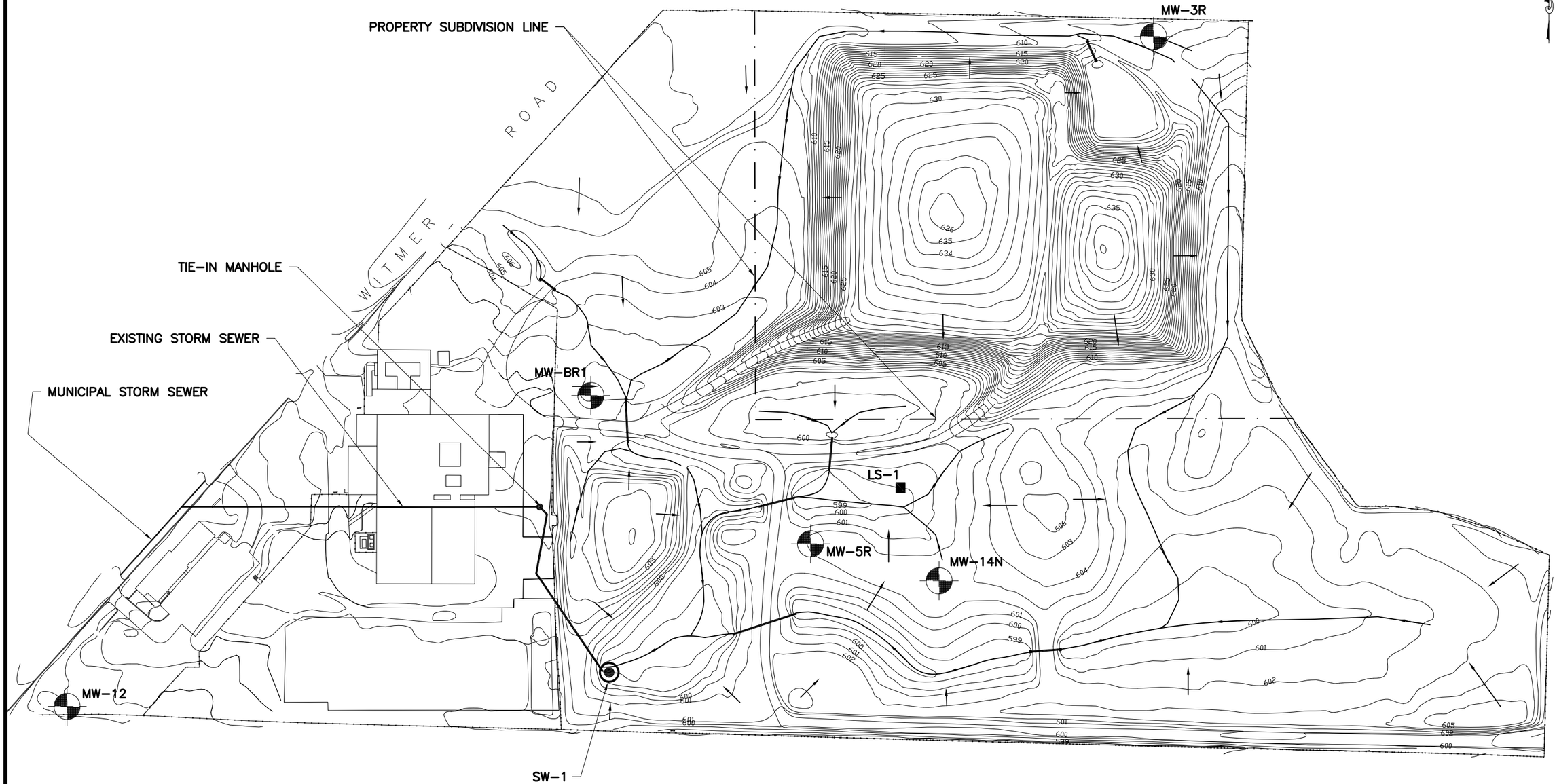
Weather Conditions _____

Inspector _____

CC Metals and Alloys, LLC
Checklist of Recommended Corrective Actions

Item Number	Item	Action Taken	Date of Correction	Signature

Appendix C



NOTE:
DRAWING BASED ON LOCKWOOD MAPPING, INC.
TOPOGRAPHIC SERIES 6781S1 THRU 6781S11

GRAPHIC SCALE

150 0 75 150 300 600

(IN FEET)
1 inch = 150 ft.

LEGEND:

- MW-00 — Monitoring Well
- Surface Water Sample
- Sump Sample
- Surface Water Flow Direction

DATE: 08/16/08
REV: NA
CHECKED : KEH
DRAWN : DCH
SCALE : 1"=150'

WELL LOCATION & SURFACE WATER DRAINAGE MAP

Calvert City Metals and Alloys, LLC
PO Box 217, Highway 95
Calvert City, Kentucky 42029

LAN ASSOCIATES, INC.
CONSULTING • ENGINEERING • PLANNING
66 CUNA STREET, ST. AUGUSTINE, FL 32084-3684 (904)824-6999

APPENDIX: C

JOB NO. 2.3643.17

Appendix D

Title: Groundwater/Surface Water Sampling

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

11-12-2007	11-12-2007
Roger Senf Date	Kenneth Kasperek Date
Department Manager	Technical Director
11-12-2007	11-12-2007
Verl D. Preston Date	Chris A. Spencer Date
Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. AFS-GW-12

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

Facility Distribution No. _____ **Distributed To:_____**

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AFS-GW-12	3	May 16, 2007	1 of 7

TITLE: GROUNDWATER/SURFACE WATER SAMPLING

Supercedes: Revision 2

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Verl D. Preston, Quality Manager	Verl D. Preston	5/16/07
Christopher A. Spencer, Laboratory Director		5/16/07
Roger J. Senf, Field Services Supervisor		5/16/07

1.0 SCOPE

This S.O.P. will discuss the procedures for the sampling of ground water and surface water.

2.0 SUMMARY

This S.O.P. describes the methods of collecting groundwater surface water

3.0 DEFINITIONS

H.A.S.P.- Health and Safety Plan

4.0 RESPONSIBILITIES

Field personnel are to obtain a representative sample.

5.0 SAFETY

A site/area safety analysis must be conducted prior to commencing sampling activities. In the event that the site/area cannot be accessed or sampled in a safe manner, do not proceed with site/area sampling activities. The site manager and STL Buffalo's Field Services Supervisor must be advised of the unsafe condition(s), and the condition(s) must be addressed to the satisfaction of Buffalo's Field Services Supervisor prior to access/sampling.

Upon the satisfactory determination of a site/area safety analysis, hand and eye protection should be worn. Air monitoring should be performed if specified in the sites Health and Safety Plan.

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

TITLE: GROUNDWATER/SURFACE WATER SAMPLING

Supercedes: Revision 2

5.1 PRIMARY MATERIALS USED

The following is a list of the materials that could be encountered during this procedure, which have a serious or significant hazard rating. **This list does not include all materials that could be encountered. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.**

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.
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.
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 the 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 can cause burns that may result in permanent impairment of vision-even blindness.
Sulfuric	Corrosive	1 Mg/M3-	Inhalation produces damaging effects on the mucous

Acid	Oxidizer Dehydrator Poison Carcinogen	TWA	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.			

The safety procedures for handling hazardous wastes must be consistent with those outlined in the Corporate Safety Manual and Chemical Hygiene Plan.

6.0 PROCEDURE

The following sections discuss the general methodology to be employed in the collection of groundwater and surface water samples. Field data will be recorded on water resistant preprinted field observation forms. Field activities will be sequentially recorded with a waterproof permanent marker.

6.1 Groundwater

6.1.1 Monitoring Well Inspection

As part of groundwater sampling, the sampling team shall inspect the exterior of the protective casing and/or monitoring well riser pipe to determine if the well integrity is intact. The condition of the concrete surface seal will be inspected and described on the field data collection form. Information to be recorded is as follows:

- Condition of surface seal: good; cracked (to what degree); none, etc.
- Height of protective casing above ground surface
- Height of riser pipe below top protective casing
- Condition of protective casing: unlocked; damaged; loose

6.1.2 Site Preparation

A container lined with new plastic sheeting will be placed next to the well to avoid contact of the bailer or other sampling equipment with the ground surface. This method is more advantageous than placing plastic sheeting on the ground surface, which is easily stepped on and does not contain any spilled liquids. The plastic sheeting will be collected subsequent to sampling and properly disposed.

During all aspects of the sample collection procedure, sampling personnel shall wear protective gloves to minimize the potential for cross contamination. Further, sampling personnel will exercise care in all aspects of the work to insure sample integrity.

Prior to sampling, the protective casing will be unlocked and carefully removed to avoid the introduction of any foreign material into the well. If necessary, the exterior and interior of the exposed riser pipe will be carefully wiped with filter paper (or equivalent) and distilled water.

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TITLE: GROUNDWATER/SURFACE WATER SAMPLING

Supercedes: Revision 2

6.1.3 Health and Safety Monitoring

Air monitoring measurements will be made in the area of the monitoring well as required. This information will be noted on the appropriate field form and will be provided as part of the field sampling report.

6.1.4 Water Level Monitoring

Water level measurements will be made in all monitoring wells before and after purging. Water levels will be measured from marks on the protective casing or riser pipe to insure consistent water level data. When possible, water levels will be measured from the top of the well riser pipe.

Water level depths shall be measured using the "wetted tape" method or electronic water level indicator. The "wetted tape" method is considered the precise and accurate method of measuring water levels. Sampling personnel will exercise care to insure the collection of precise measurements.

Chalk will be applied to the lower few feet of a weight steel tape and the tape will be carefully lowered into the well. Once the weight encounters the groundwater, the tape will be lowered a short distance and the depth measured at the protective casing or riser pipe. The tape will be removed from the well and the depth of the wetted portion of the tape will be subtracted from the depth noted at the surface measuring point. All measurements will be made to the nearest 0.01 feet. If using electronic water level indicator, lower probe until signal is audible. Place measuring tape on casting/riser measurement location and note depth.

Subsequent to the initial water level measurement, the weighted steel tape or water level indicator will be lowered to the bottom of the well and the total well depth, measured from the top of the riser pipe or protective casing, will be noted on the field form.

The steel tape and/or probe will be decontaminated between monitoring wells by wiping with a clean cloth or filter paper, washing with laboratory grade liquid detergent, and rinsing with deionized water.

Information to be recorded as follows:

- Surface measuring point (protective casing and riser pipe)
- For each water level measurement:
  - depth measured from surface measuring point
  - measurement of wetted portion of tape
- Depth to groundwater
- Depth to bottom of monitoring well below surface measuring point
- Any conditions observed which might have impacted water level measurements (e.g., pumping of nearby wells, drilling operations, well recently purged and may not have fully recovered, etc.)

6.1.5 Check for Multi-Phased/Multi-Layered Fluids

Observations of multi-phased/multi-layered fluids will be noted in the field log. If multi-phased/multi-layered fluids are encountered, the well will be purged of all phased/layered material to assure that this residual material is not detected in subsequent sampling events. A sample of the multi-phased/multi-layered material may be taken if required.

6.1.6 Well Purging

When conditions allow, monitoring wells shall be purged a minimum of three well volumes prior to the collection of analytical samples. In the event a well evacuates to dryness and recharges rapidly, well purging shall continue until well volumes are evaluated. If the well evacuates to dryness and is slow to recharge, it shall be sampled within 24 hours for as many samples as possible.

One well volume is defined as the volume of standing water in the well. Prior to purging, the volume of standing water will be calculated using the measured depth to groundwater and the depth of the well. The volume of water to be removed during purging can be calculated using the following formula:

$$V = r^2(h)(0.49)$$

Where: V = standing volume to be purged (in gallons)
r = inside radius of well in inches
h = linear feet of standing water in the well casing
(0.49) is a correction factor that includes conversion from inches to feet and the fact that three well volumes are to be purged.

Monitoring wells with groundwater levels less than 25 feet below ground surface may be purged using an ISCO peristaltic or equivalent pump. If this type of pump is used, new or dedicated polyethylene discharge and intake tubing (3/8 inch I.D. low density polyethylene) will be used for each well.

Monitoring wells with water levels greater than 25 feet will be purged using dedicated polyvinyl chloride (PVC) or stainless steel bailers. The bailers will be equipped with a bottom valve and will be constructed without the use of glue or welds.

In some cases, alternate methods may be employed for purging monitoring wells. Alternate methods may include, but are not limited to, dedicated gas-driven bladder, submersible, or air-lift pumps.

The volume of water purged from the monitoring wells will be collected in a 5-gallon bucket or other measuring device and the volume will be measured and recorded. All groundwater purged from the well shall be disposed of on the ground away from well head or collected and disposed of on-site.

6.1.7 Groundwater Sample Collection

After purging three well volumes of water from the monitoring well, groundwater samples will be collected using a dedicated or pre-cleaned stainless steel bailer or an approved sampling pump. Alternate sampling devices may be used if approved for use in site-specific sampling plan.

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TITLE: GROUNDWATER/SURFACE WATER SAMPLING

Supercedes: Revision 2

Sample collection will be performed in such a way as to minimize unnecessary agitation of the sample. Bailers will be slowly lowered into the water to minimize agitation of sample and sample pumps, if used, will be operated at low flow rates. The maximum allowable flow rate for the collection of volatile organic samples will be 100 ml/min. Sampling personnel will wear clean latex or other laboratory-grade gloves while handling sample containers and performing field measurements to minimize the potential for sample cross contamination. Gloves will be changed between wells and after handling or contacting anything which may result in sample cross contamination.

Sample containers will be filled directly from the bailer or pump tubing. Sample containers for volatile organic analyses will be completely filled to eliminate bubbles and air entrapment in the container.

If a well is evacuated to dryness and the well does not recharge at a rate sufficient to allow sampling, the well will be sampled within 24 hours. At that time, sampling personnel will obtain as many of the required sample aliquots as possible with the water which is available in the well. Wells with rapid recharge rates will be sampled within three hours of the completion of purging the well. In all cases, groundwater samples will be collected according to the prioritized schedule:

- Volatile organics
- Total Organic Carbon (TOC)
- Total Organic Halogen (TOX)
- Acid Extractable Organics
- Base/Neutral Organics
- Pesticides
- Metals
- Phenol
- Cyanide
- Sulfate and Chloride
- Nitrate and Ammonia
- Radionuclides
- Water Quality Parameters

In the event split samples are required as part of a sampling program, samples will be bailed from the well and the bailer equally split between the two sets of sample containers. Each type of sample container will be filled with equal volumes from the same bailer. When necessary, additional bailers may be required to completely fill each type of container. This method will limit the potential for differences between the analytical parameters measured by different laboratories. VOA bottles shall be completely filled during the initial pour.

At each well, an additional sample will be collected. The temperature, pH, and specific conductance (at a minimum) of this sample will be measured immediately. The field instruments used in the measurement of pH, and specific conductance will be calibrated prior to the first measurements of the day and will be recalibrated each time they are moved from location to location. Specific conductance, and pH measurements will be conducted in accordance with SOP# AWC-120.1-32.

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TITLE: GROUNDWATER/SURFACE WATER SAMPLING

Supercedes: Revision 2

Samples collected to be analyzed for soluble metals will be filtered immediately on-site before returning to the laboratory. Immediately is defined as within twenty minutes of sample collection except under adverse weather conditions when it may be necessary to exceed that twenty minute time period. The filtering will be accomplished using a GCA/Precision Scientific, Model PV-35 Vactorr vacuum pump in conjunction with a Nuclepore or Millipore 47 mm Filter solution and deionized water.

Preservatives should be added immediately after sample collection. All samples will be stored at 4°C (wet ice temperature) prior to delivery to the laboratory. The sample container will be stored and maintained to minimize the possibility of cross contamination.

6.2 Surface Water

Surface water samples will be collected by immersing the sample container below the water level and allowing the container to fill with water. If necessary, a smaller container (glass) may be used to collect and transfer surface water to the sample container or to completely fill the sample container and eliminate "headspace". Sampling personnel shall carefully collect the field samples and minimize the collection of sediment. Surface water shall always be collected from the furthest downstream location to the furthest upstream.

Surface water samples will be handled, preserved, stored and field measurements made as required.

7.0 REFERENCES N/A

8.0 FLOWCHARTS, TABLES & DIAGRAMS N/A

9.0 CHANGES FROM PREVIOUS REVISION

9.1 Update Safety Section, 5.1

Appendix E

Cover Page:

Quality Assurance Manual

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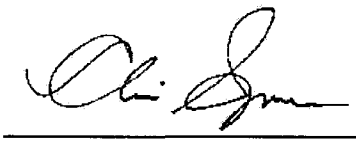
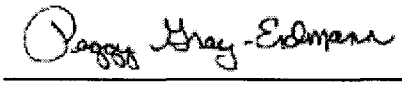


Laboratory Director – Chris Spencer — January 11, 2008 — Date



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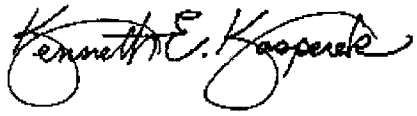

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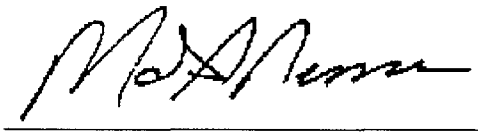

Customer Service Manager - Mark Nemec — January 11, 2008 — Date

Operations Manager – John Schove — January 11, 2008 — Date

SECTION 2

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SOPs AND POLICIES REFERRED TO IN THE QA MANUAL

SOP/Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-003	Management of Change Procedure
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-005	Calibration Curves (General)
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-L-P-001	Record Retention
CW-F-P-002	Authorization Matrix
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-004	Controlled Purchases Policy
BF-GP-001	Calibration of Autopipettes and Repipetters
BF-GP-002	Support Equipment: Maintenance, Record Keeping and Corrective Actions
BF-GP-005	Sample Homogenization and Subsampling
BF-GP-012	Technical Data Review
BF-GP-013	Manual Integration
BF-GP-015	Record Storage and Retention

SECTION 3

INTRODUCTION
(NELAC 5.1 - 5.3)

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica Buffalo's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. Each TestAmerica laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (1999). In addition, the policies and procedures outlined in this manual are compliant with the various accreditation and certification programs listed in Appendix 6. The relevant NELAC section is included in the heading of each QAM section.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- EPA SW-846, *Test Methods for the Evaluation of Solid Waste, 3rd Edition,* September 1986; Update I, July 1992; Update II, September 1994; and Update III, December 1996.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- USEPA Contract Laboratory Program. *Statement of Work for Inorganics Analysis. Multi-Media, Multi-Concentration.* Document ILM04.0/4.1/4.2
- USEPA Contract Laboratory Program. *Statement of Work for Inorganics Analysis. Multi-Media, Multi-Concentration.* Document ILM05.1/5.2/ 5.3.
- USEPA Contract Laboratory Program. *Statement of Work for Organics Analysis. Multi-Media, Multi-Concentration.* Document Number OLM03.1, August 1994.
- USEPA Contract Laboratory Program. *Statement of Work for Organics Analysis. Multi-Media, Multi-Concentration.* Document Number OLM04.0, August 1994 and updates
- New York State Analytical Services Protocol, July 2005
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th and 21st Edition.
- *U.S. Department of Energy* Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- *U.S. Department of Energy, Quality Systems for Analytical Services*, Revision 2.1, November 2005.
- Toxic Substances Control Act (TSCA).

3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by TestAmerica Buffalo conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 5 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

TestAmerica Buffalo analyzes thousands of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested water, air, industrial waste, and soil methodologies needed to provide analytical services in the United States, its territories and for foreign countries. The specific list of test methods used by the laboratory can be found in Appendix 4 of this QAM. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by TestAmerica Buffalo shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director, Technical Director, Quality Assurance (QA) Manager, Customer Service Manager or Operations Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process

The manual is reviewed annually by the QA Manager and laboratory personnel to assure that it reflects current practices and meets the requirements of TestAmerica Buffalo's clients and regulators. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. The updates will be reviewed by the QA Manager, Laboratory Director, Operations Manager, Technical Director, Department Managers, relevant operational staff and Corporate Quality Assurance (if a change is made to the Corporate template) and then formally incorporated into the document in periodic

updates. The QAM is based on a Corporate QAM Template that is prepared and approved by the Chief Operating Officers (COOs) and Corporate Quality Assurance. This template is reviewed annually by the COOs, Corporate Quality, and each laboratory. Necessary changes are coordinated by the Vice President of Quality and Environmental Health & Safety (EHS) and distributed to each laboratory for inclusion in the laboratory specific QA Manuals.

Policies in the QAM that require immediate attention may be addressed through the use of Corporate QA/QC Policy Memoranda. QA/QC Policy Memoranda are published from time to time to facilitate immediate changes to QA/QC Policy. QA/QC Policy Memoranda supersede the QAM and all other SOPs (refer to Section 5.3). All policy memoranda are dated, archived and distributed by their placement into the front of the QAM between the signature page and Section 2. At a minimum, each policy memorandum is approved by the same authorized signatories as shown on the cover page of the QA Manual. In addition, Corporate QA/QC Policy Memoranda are signed by the COOs and VP of Quality and EHS. The QA/QC Policy Memoranda are incorporated into the QAM during the periodic updates. Policy memorandum may also include an expiration date if appropriate. An example format can be found in Figure 3-1. A similar procedure is followed for local laboratory changes.

Laboratory-specific QAM changes are approved and documented through the Interim Change Procedure defined for controlled documents (SOP BF-QA-003, "Procedure for Writing, Reviewing and Revising Controlled Documents").

3.4.2 Control

This manual is considered confidential within TestAmerica and may not be altered in any manner by other than a duly appointed representative from TestAmerica. If the document has been provided to external users or regulators, it is for the exclusive purpose of reviewing **TestAmerica Buffalo**'s quality systems and shall not be used in any other way without the written permission of an appointed representative of TestAmerica. The procedure for control of distribution is incorporated by reference to SOP BF-QA-003, "Procedure for Writing, Reviewing and Revising Controlled Documents".

The order of precedence in the event of a conflict between policies is outlined in Section 5.3 of this Quality Assurance Manual.

Figure 3-1.

Format for a QA/QC Policy Memorandum

Corporate QA/QC Policy Memorandum # _____

Effective Date: _____ Expiration Date: When Appropriate QAM Section is Revised

Corporate: *(Only needed for Corporate Memorandum – Delete if Laboratory)*

COO - West	Date	Vice-President, QA and EHS	Date
COO - East	Date		

Local:

Laboratory Director Approval	Date	Quality Assurance Approval	Date
Operations Manager Approval	Date	Technical Director Approval	Date
Customer Service Manager Approval	Date		

1. **Purpose**

2. **Procedure**

3. **Attachments**

4. **References/Cross References**

SECTION 4

ORGANIZATION AND MANAGEMENT
(NELAC 5.4.1)

4.1 OVERVIEW

TestAmerica Buffalo is part of a national network of laboratories known as TestAmerica. This Quality Assurance Manual (QAM) is applicable to the ***TestAmerica Buffalo*** laboratory only.

TestAmerica Buffalo
10 Hazelwood Drive
Amherst, New York 14228
EPA ID #: NY00044

The Corporate organization chart can be found in Figure 4-1 and the laboratory's organization chart can be found in Appendix 2. The locations of other TestAmerica labs are as follows:

TestAmerica Anchorage
TestAmerica Austin
TestAmerica Burlington
TestAmerica Cedar Falls
TestAmerica Chicago
TestAmerica Connecticut
TestAmerica Corpus Christi
TestAmerica Dayton
TestAmerica Denver
TestAmerica Edison
TestAmerica Honolulu
TestAmerica Houston
TestAmerica Irvine
TestAmerica King of Prussia
TestAmerica Knoxville
TestAmerica Los Angeles
TestAmerica Mobile
TestAmerica Morgan Hill
TestAmerica Nashville
TestAmerica North Canton
TestAmerica Ontario
TestAmerica Orlando
TestAmerica Pensacola
TestAmerica Phoenix
TestAmerica Pittsburgh
TestAmerica Portland
TestAmerica Richland
TestAmerica San Francisco
TestAmerica Savannah
TestAmerica Seattle

TestAmerica Spokane
TestAmerica St. Louis
TestAmerica Tacoma
TestAmerica Tallahassee
TestAmerica Tampa
TestAmerica Valparaiso
TestAmerica Watertown
TestAmerica West Sacramento
TestAmerica Westfield

4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions define each role in its relationship to the Quality Assurance Program. More extensive job descriptions are maintained by laboratory management and TestAmerica Human Resources.

4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of ***TestAmerica Buffalo***. All employees have access to the QAM and are responsible for knowing the content of this manual and upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs.

4.2.2 Chairman/Chief Executive Officer (CEO)

The Chairman/CEO is the Chairman of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. Together with the President/CEO of the Analytical Division, the Chairman/CEO establishes the overall quality standard and data integrity program for the company, providing the necessary leadership and resources to assure that the standard and integrity program are met.

4.2.3 President/Chief Executive Officer (CEO)

The President/CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. Together with the Chairman/CEO, the President/CEO establishes the overall quality standard and data integrity program for the Analytical Division, providing the necessary leadership and resources to assure that the standard and integrity program are met.

4.2.4 Chief Operating Officer (COO) – East and West

The COOs serve as the ranking executives for all respective analytical laboratory operational functions and report to the President/CEO of the Analytical Division. They are responsible for the daily management of all analytical laboratories, long-term planning and development of technical policies and management plans. They ensure the attainment of corporate objectives through the selection, development, motivation, and evaluation of top management personnel. The COOs approve all operating budgets and capital expenditures. The COOs sign-off on the final QAM template that contains company policies for implementing the Quality Program.

4.2.5 General Manager (GM)

Each GM reports directly to a COO. Each GM has full responsibility for the overall administrative and operational management of their respective laboratories. The GM's responsibilities include allocation of personnel and resources, long-term planning, setting goals, and achieving the financial, business, and quality objectives of TestAmerica. The GM ensures timely compliance with corporate management directives, policies, and management systems reviews. The GM is also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

4.2.6 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President of QA/EHS reports directly to the Chairman/CEO. With the aid of the Analytical Division and Non-Analytical Division Senior Management Teams, Laboratory Director/ Managers, Quality Directors, EHS Directors, QA Managers and EHS Coordinators, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and Environmental, Health and Safety Program within TestAmerica. Additional responsibilities include:

- Review of QA/QC aspects of Corporate SOPs, national projects and expansions or changes in services.
- Coordination/preparation of the Corporate QAM Template that is used by each laboratory to prepare its own laboratory-specific QAM.
- Maintenance of Corporate Policies, Quality Memorandums and SOPs. Maintenance of data investigation records that are reported to Corporate Management.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation of a monthly report that includes quality metrics across the Analytical Division and a summary of any quality related initiatives and issues.
- With the assistance of the Corporate Senior Management Teams and the EHS Directors, development and implementation of the TestAmerica Environmental, Health and Safety Program.

4.2.7 Quality Directors (Corporate)

The Quality Directors report to the VP-QA/EHS. Together with the VP-QA/EHS, the Quality Directors have the responsibility for the establishment, general overview and maintenance of the Analytical Division's Quality Assurance Program within TestAmerica. The Quality Directors are responsible for:

- Oversight of the QA/QC programs within each laboratory. This includes a final review of each laboratory-specific QAM and receipt of each laboratory's QA monthly report.
- Review of QA/QC aspects of national projects.
- Assistance with certification activities.

4.2.8 Ethics and Compliance Officers (ECOs)

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – VP-QA/EHS and VP-Client and Technical Services. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the CEOs, COOs, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.9 Vice President of Client and Technical Services

The Vise President (VP) of Client and Technical Services is responsible for offerings to clients including risk management, technical assistance, legal compliance and contract administration. The VP of Client and Technical Services provides support and direction to the Managers of these areas, and supports the COOs in decisions regarding long term planning, resource allocation and capital expenditures.

4.2.10 Director of Technical Services

The Director of Technical Services is responsible for establishing, implementing and communicating TestAmerica's Analytical Division's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

4.2.11 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

4.2.12 Environmental Health and Safety Directors (EHSDs) (Corporate)

The EHSDs report directly to the VP-QA/EHS. The EHSDs are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.13 Laboratory Director

TestAmerica Buffalo's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

The Laboratory Director has the authority to affect those policies and procedures to ensure that only data of the highest level of excellence are produced. As such, the Laboratory Director is responsible for maintaining a working environment which encourages open, constructive problem solving and continuous improvement.

Specific responsibilities include, but are not limited to:

- Provides one or more department managers for the appropriate fields of testing. If the Department Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Department Manager to temporarily perform this function. If the absence

exceeds 65 consecutive calendar days, the primary NELAC accrediting authority must be notified in writing.

- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Leads the management team, consisting of the QA Manager, the Technical Director, Customer Service Manager, and the Operations Manager as direct reports.

4.2.14 Quality Assurance (QA) Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA department to accomplish specific responsibilities, which include, but are not limited to:

- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.

- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems, data authenticity and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken
- Monitoring standards of performance in quality control and quality assurance.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a subset of all final data reports for internal consistency.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Leads the QA team to enable communication and to distribute duties and responsibilities.

The QAM shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QAM is available to any employee at the facility to resolve data quality or ethical issues.

4.2.15 Quality Assurance Specialist

The QA Specialist is responsible for QA documentation and involvement in the following activities:

- Assist the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.
- Facilitate external audits, coordinating with the QA Manager and Laboratory Staff to address any deficiencies noted at the time of the audit and subsequently presented in the final audit report.
- Assist the QA Manager in the preparation of new SOP's and in the maintenance of existing SOPs, coordinating annual reviews and updates.
- Manages the performance testing (PT) studies, coordinates follow up studies for failed analytes and works with QA Manager and Laboratory Staff to complete needed corrective action reports.
- Document control maintenance.

- Assists the Quality Manager and Project Management Group in the review of program plans for consistency with organizational and contractual requirements. Summarize and convey to appropriate personnel anomalies or inconsistencies observed in the review process.
- Manages certifications and accreditations.
- Monitors for compliance the following QA Metrics: Temperature Monitoring of refrigeration units and incubators; thermometer calibrations; balance calibrations; eppendorf/pipette calibrations; and proper standard/reagent storage.
- Periodic checks on the proper use and review of instrument logs.
- Assists with authenticity audits
- Assists in the technical review of data packages which require QA review.

4.2.16 Technical Director

The Technical Director reports directly to the Laboratory Director and is responsible for assessing the construction and management of the facility design, maintaining environmental conditions, technical and financial evaluation of capital equipment and capital budgeting and asset valuation.

In addition, the Technical Director solves day to day technical issues, provides technical training and guidance to staff, project managers and clients, investigates technical issues identified by operations personnel or QA, and directs evaluation of new methods. Specific responsibilities include but are not limited to:

- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.

4.2.17 Operations Manager

The Operations Manager reports to the Laboratory Director and oversees the daily operations of the analytical laboratory, maintaining a working environment that encourages open, constructive problem solving and continuous improvement.

The Operations Manager is responsible for supervision of laboratory staff, setting goals and objectives for the laboratory, ensuring compliance with project/client requirements and ensuring on-time performance, supervises maintenance of equipment and scheduling of repairs. Responsibilities also include implementation of the quality system in the laboratory and ensuring timely compliance with audit and QA corrective actions.

In addition, the Operations Manager works with the Technical Director in evaluating technical equipment, assessing capital budget needs and determining the most efficient instrument utilization. More specifically he:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Director and QA Manager and in compliance with regulatory requirements.
- Works with the Preventive Maintenance Coordinator to ensure that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

4.2.18 Department Managers

Department Managers report to the Operations Manager. The Department Managers serve as the technical experts on assigned projects, provide technical liaison, assist in resolving any technical issues within the area of their expertise; and implement established policies and procedures to assist the Operations Manager in achieving section goals. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training, development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Human Resources Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.

- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Director, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CPAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Director, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.19 Environmental Health & Safety / Hazardous Waste Coordinator

The Health and Safety Coordinator is responsible for the safety and well-being of all employees while at the laboratory. This includes, but is not limited to, administering the Corporate Safety Manual that complies with federal regulations, MSDS training and review, conducting laboratory safety orientation and tours for all new employees, providing instructions on safety equipment, cleaning up laboratory spills, and instructing personnel of laboratory procedures for emergency situations. The Health and Safety Coordinator is on-call 24-hours a day, 7-days a week for all laboratory situations.

The Health and Safety Coordinator responsibilities additionally include waste management of laboratory generated hazardous waste in accordance with appropriate regulations. This includes maintenance of required documentation, such as waste manifests, segregation of waste in accordance with requirements, and training of personnel in proper segregation of waste and preparation of Safety related SOPs. The EHSC maintains overall EH&S program oversight, but may delegate specific day-to-day activities as necessary.

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.

- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.
- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.

4.2.20 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Director, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum

performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.

- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.21 Data Validation and Data Packaging Managers

The Data Validation and Data Packaging Specialists are responsible for coordinating receipt of all data from the various service groups within the laboratory, reviewing data for compliance to laboratory QC criteria and/or criteria in the LIMs Project Profile Specification, and ensuring that data are reported in a timely manner and in the proper format.

4.2.22 Sample Management

The Sample Custodian is designated as the Sample Management Coordinator for any work performed internally and responsible for the receipt and login of client samples. The sample custodian confirms the samples received against the Chain of Custody, transports the samples to the proper storage unit within the facility and tracks the disposal of client samples after the required holding time has expired. The Sample Management Manager reports to the Technical Director. The responsibilities are outlined below:

- Direct the logging of incoming samples into the LIMS.
- Ensure the verification of data entry from login.
- Waste management and disposal

4.2.23 Customer Service Manager

The Customer Service Manager reports to the Laboratory Director and serves as the primary interface between the laboratory and the Sales and Marketing staff. Responsibilities include:

- Coordinates marketing efforts with General Manager, Laboratory Director, Project Managers, and laboratory marketing group
- Coordinates proposal and contract review and response process
- Compiles and interprets receipts forecast to show near term business trends.
- Prepares proposals for new business opportunities.
- Provides general sales support to Account Executives for business development activities started in the field.
- Develops and maintains business materials and organized information resource files that include project descriptions, resumes, original proposals, boilerplates, and company qualifications materials.

4.2.24 Project Management Manager

The Project Management Manager reports to the Customer Service Manager and serves as the interface between the laboratory's technical departments and the laboratory's clients. The staff consists of the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Technical training and growth of the Project Management team.
- Technical liaison for the Project Management team.
- Human resource management of the Project Management team.
- Responsible to ensure that clients receive the proper sampling supplies.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

4.2.25 Project Manager

The laboratory recognizes the importance of efficient project management. The laboratory Project Managers (PM) are responsible for preparing the LIMs project technical specifications which summarize QA/QC requirements for the project, maintaining the laboratory schedule, communicating technical requirements to the laboratory, and advising the Operations Manager, QA and Department Managers of all variances. The laboratory Project Manager will provide technical guidance and the necessary laboratory-related information to the preparer of project-specific QAPPs and provide peer review of the final document to ensure accuracy of the laboratory information.

4.2.26 Bottle Kit Preparation and Shipping Manager

The Bottle Order Preparation Manager reports to the Customer Service Manager. He is responsible for ensuring the timely and correct shipment of sample containers, including proper preservatives and instructions, to clients. He maintains accurate records of sample container shipments.

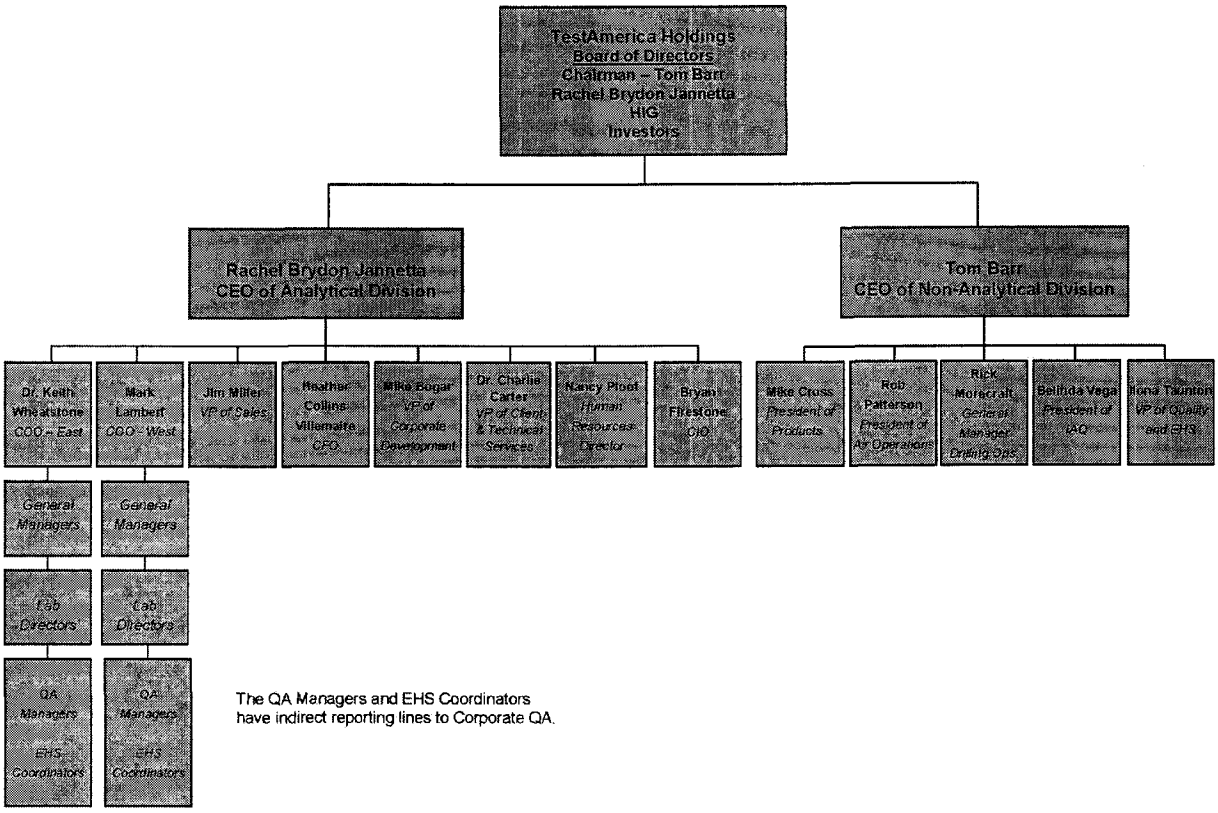
4.3 DEPUTIES

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy	Comment
Laboratory Director	Operations Manager (1) Technical Director (2)	
QA Manager	QA Specialist (1) Operations Manager (2)	
Technical Director	Laboratory Director (1) Operations Manager (2)	
Operations Manager	Department Manager (1) Department Manager (2)	Selected based on availability
Customer Service Manager	Project Mng't Manager (1) Laboratory Director (2)	
Project Management Manager	Customer Srv. Manager (1) Project Manager (2)	(2) Selected based on availability
Project Manager	Project Manager (1) Project Management Asst. (2)	(1) 2° team PM (2) Team PMA
Organic Department Manager	Analyst (1) Analyst (2)	Selected based on department, experience and availability
Inorganic Department Manager	Analyst (1) Analyst (2)	Selected based on department, experience and availability
Data Validation / Data Packaging Manager	Data Validation Specialist Data Packaging Specialist	Selected based on department and availability
EHS Coordinator	Safety Officer (1) Sample Mng't Manager (2)	
Sample Management Manager	Sample Custodian (1) EHS Coordinator (2)	
Bottle Preparation / Shipping Manager	Bottle Prep Technician (1) Sample Mng't Manager (2)	

Figure 4-1.

Corporate Organization Chart



SECTION 5

QUALITY SYSTEM
(NELAC 5.4.2)

5.1 QUALITY POLICY STATEMENT

The management of TestAmerica and ***TestAmerica Buffalo*** are committed to providing data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols described in this manual.

In all aspects of the laboratory and business operations, management is dedicated in maintaining the highest ethical standards. An Ethics Policy sign-off can be viewed in Appendix 1. Training on ethical and legal responsibilities is provided annually and each employee signs off annually on the policy as a condition of employment.

It is TestAmerica's Policy to continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. The company recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.

TestAmerica Buffalo strives to provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at ***TestAmerica Buffalo*** plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The 7 elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Policy No. CA-L-P-001) and employee ethics statements (Appendix 1).
- An Ethics and Compliance Officer (ECO).
- A training program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (SOP No. CA-L-S-001)
- Procedures and guidance for recalling data if necessary (SOP No. CA-L-S-001).
- An effective external and internal monitoring system that includes procedures for internal audits (Section 16).

As an American Council of Independent Laboratories (ACIL) member, all TestAmerica laboratories adhere to the following ACIL Code of Ethics:

- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the ethical and quality standards of our industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 QUALITY SYSTEM SUPPORTING DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents prepared by the laboratory and company management:

- Quality Assurance Manual (QAM) Template
- Quality Assurance Manual – Each laboratory has a lab specific quality assurance manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Corporate TestAmerica QA/QC Policy Memorandums (Refer to Section 3.4).
- Laboratory QA/QC Policy Memorandums (Refer to Section 3.4).

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- TestAmerica QA/QC Policy Memorandum - Corporate
- Laboratory QA/QC Policy Memorandum
- Quality Assurance Manual
- Corporate SOPs and Policies
- Laboratory SOPs and Policies

- Other (Work Instructions (WI), memos, flow charts, etc.)

5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples. The calculation of precision is described in Section 25.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery. The calculation of accuracy is described in Section 25.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories, and by the degree to which approval from the US EPA or other pertinent regulatory agencies is obtained for any procedure for which significant modifications have been made.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory prepares a *Quality Control Limit Summary that contains tables* that summarize the precision and accuracy acceptability limits for analyses performed at ***TestAmerica Buffalo***. This summary includes an effective date, is updated each time new limits are generated and is located within the laboratory LIMs system. The quality control limits are method, protocol, matrix and date sensitive and may be retrieved as needed after expiration. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, ***TestAmerica Buffalo*** has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in Section 25.

5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. *TestAmerica Buffalo* routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The procedure for determining the statistical limits may be found in SOP BF-QA-002, Quality Control Limits. The analysts are instructed to use the current limits in the laboratory (dated and approved the QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory through date sensitive tables within the LIMs System. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

As the QC limits are calculated, QC charts may be generated showing warning and control limits for the purpose of evaluating trends. The QA Manager periodically evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 17). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6

DOCUMENT CONTROL
(NELAC 5.4.3)

6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled at each laboratory Facility:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

The Corporate staff posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These are collectively termed "Official Documents" and encompass the Policies and Procedures that all facilities are required to employ. These official documents are only considered controlled when they are read on the company intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving official documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action notices. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports. Discussion on records control is described in Section 15.

The maintenance of purchasing data is discussed in Section 9.

The maintenance of sales and marketing contracts is discussed in Section 7.

6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a control system for each document include a unique name and number, the number of pages of the item, the effective date, revision number and the laboratory's name. The Quality Assurance Specialist and Document Control Officer are

responsible for the maintenance of the system and maintain the items in the QA Storage Room, On-Site Archive Storage and Off-Site Archive Storage.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a Department Manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain the official document on file. The official document is provided as needed to those using it. Controlled documents shall be available at all locations where the operational activity described in the document is performed (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years for the majority of procedures, every 1 year for Drinking Water programs and revised as appropriate. Changes to documents occur when a procedural change warrants a revision of the document.

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP No. BF-QA-003, "Writing, Reviewing and Revising Controlled Documents". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. A controlled electronic copy of the current version is maintained on the laboratory IntraNet site (BufNet) and is available to all personnel.

For changes to SOPs, refer to SOP No. BF-QA-003, "Writing, Reviewing and Revising Controlled Documents".

Forms, worksheets, work instructions and information are organized by department in the QA office. Electronic versions are kept in a controlled access electronic folder in the QA department. As revisions are required, a new version number and revision date is assigned and the document placed on the laboratory IntraNet (BufNet) for use.

6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived as described in Section 15.

SECTION 7

REVIEW OF WORK REQUEST

7.1 OVERVIEW

TestAmerica Buffalo has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is TestAmerica's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and TestAmerica's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the lab's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and TestAmerica's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the

contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.
All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The review process is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the National Account Director, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- General Manager
- Customer Service Manager
- Operations Manager
- Laboratory and/or Corporate Technical Directors
- Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The National Account Director, Legal Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts. The Customer Service Manager at the TestAmerica Buffalo facility also maintains copies of these documents.

7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Regional Account Manager. A copy of the contract and formal quote will be filed with the laboratory PM and the Customer Service Manager.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, ***TestAmerica Buffalo*** assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements. Specific information related to project planning may be found in SOP BF-PM-001, Project Information Requirements.

PM's are the direct client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the management staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager.

TestAmerica strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

SECTION 8

SUBCONTRACTING OF TESTS
(NELAC 5.4.5)

8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the corporate network. The phrase "work sharing" refers to internal transfers of samples between company laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When we must outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the SOP on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accredited work where required.

Project Managers (PMs), Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

Note: In addition to the client, some regulating agencies, such as the Department of Energy and the USDA, require notification prior to placing such work.

Approval may be documented through reference in a quote / contract or e-mail correspondence.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM, Regional Account Executive (RAE) or Customer Service Manager (CSM] becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified network laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be

as simple as placing a copy of an e-mail from the client in the project folder);

- Firms listed as pre-qualified and currently under a subcontract with the company (in JD Edwards): A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation for the requested tests prior to sending samples.
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All intra-company laboratories are pre-qualified for work-sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. Refer to SOP No. CA-C-S-001, Work Sharing Process.

When the potential sub-contract laboratory has not been previously approved, then to begin the process, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

8.2.1 The QA Manager must ensure that the Subcontracting Approval Form (Figure 8-1) has been completed and have supporting documentation on file prior to initiation of any work. A letter or e-mail is sent to the lab requesting the following information:

8.2.1.1 If a lab is NELAC or A2LA accredited,

8.2.1.1.1 Copy of necessary certifications verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accredit-able (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.

8.2.1.1.2 Insurance Certificate. This is required by TestAmerica's Chief Financial Officer

8.2.1.1.3 USDA soil permit if available**

8.2.1.2 For Laboratories accredited by other agencies with an auditing program:

8.2.1.2.1 Copy of necessary certifications verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accredit-able (if so, document). Certificate and scope of International Standard accreditation are

required, when applicable.

8.2.1.2.2 Insurance Certificate. This is required by TestAmerica's Chief Financial Officer

8.2.1.2.3 USDA soil permit if available**

8.2.1.2.4 Description of Ethics and Data Integrity Plan.

8.2.1.2.5 The most recent 2 sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action.

8.2.1.2.6 State Audit with Corrective Action Response

8.2.1.2.7 Example final report to confirm format is compliant and provides the necessary information. (minimally, it must be determined that Batch QC results are included in the laboratory reports and data is appropriately qualified.

8.2.1.2.8 A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review (it may need to be sent elsewhere for evaluation). This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously [minimum of 6 months] are grandfathered in.

8.2.1.3 For laboratories performing tests that are unaccredited or accredited by an agency without an audit program:

8.2.1.3.1 A copy of their Quality Assurance Manual (controlled if possible). Ensure data quality limits for relevant methods are acceptable and that training procedures are adequate.

8.2.1.3.2 Copy of necessary certifications (if available) verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accredit-able (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.

8.2.1.3.3 Insurance Certificate. This is required by TestAmerica's Chief Financial Officer.

8.2.1.3.4 USDA soil permit if available**

8.2.1.3.5 Evidence of a current SOP per method. A copy of the first page and signature page of the SOP is acceptable. A table of contents including effective dates may also be acceptable. The SOP can be examined if an on-site audit is performed.

8.2.1.3.6 Description of Ethics and Data Integrity Plan.

8.2.1.3.7 The most recent 2 sets of full proficiency testing (PT) results relevant to the analyses

of interest and any associated corrective action.

8.2.1.3.8 Example final report to confirm format is compliant and provides the necessary information. (minimally, it must be determined that Batch QC results are included in the laboratory reports and data is appropriately qualified.

8.2.1.3.9 Statement of Qualification (SOQ) or summary list of Technical Staff and Qualifications – position, education and years of experience.

8.2.1.3.10 A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review (it may need to be sent elsewhere for evaluation). This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously [minimum of 6 months] are grandfathered in.)

8.2.2 Once the information is received by the QA Manager, it is evaluated for acceptability and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site along with the associate documentation and notify the finance group for JD Edwards.

**USDA permit is required if soils less than three feet deep from New York, North Carolina, South Carolina, Georgia, Florida, Tennessee, Alabama, Mississippi, Louisiana, Arkansas, Texas, Oklahoma, New Mexico, Arizona, California, Hawaii, or outside the continental U. S. are to be analyzed. These samples require special shipping measures; check with the EHS Department. It may be necessary to heat-treat the samples before shipping if the subcontract laboratory does not have a USDA permit; however, some analytes/tests may be irrelevant after heat treatment.

8.2.3 The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. The company does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.4 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contract Department. Any problems identified will be brought to Corporate QA attention.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints must be posted using the Vendor Performance Report (Form No. CW-F-WI-009).
- Information must be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will

notify all network laboratories and Corporate QA and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Lab Directors/Managers, QA Managers and Sales Directors.

8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or RAE or CSM) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontract Laboratory Certification Verification Form (Figure 8-2) and the form is retained in the project folder. For network laboratories, certifications can be viewed on the company website.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within the network.

The PM will communicate with the subcontracted laboratory to monitor the status of the analyses, facilitate successful execution of the work and ensure the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data are incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a network work sharing laboratory may be transferred electronically and the results reported by the network work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, Corporate QA must be informed, and the QA Manager will be required to verify adequacy of proficiency scores and certifications. The laboratory must also request a copy of the raw data to support the analytical results for the first project submitted to the subcontract laboratory unless the laboratory has NELAC accreditation. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. The QA Manager will request full documentation and qualify the subcontractor under the provisions above. The approval process should be completed within 30 calendar days of subcontracting.

Figure 8-1.
Subcontracting Laboratory Approval Form (Initial / Renewal)

SUBCONTRACTING LABORATORY APPROVAL

Reference: Section 8 – Quality Assurance Manual

Date: _____
Laboratory: _____

Address: _____

_____

Contact and e-mail address: _____
Phone: Direct _____ Fax _____

Requested Item[3]	Date Received	Reviewed/ Accepted	Date
1. Copy of State Certification[1]			
2. Insurance Certificate			
3. USDA Soil Permit			
4. Description of Ethics Program[3]			
5. QA Manual[3]			
6. Most Recent (and relevant) 2 Sets of WP/WS Reports with Corrective Action Response[1,3]			
7. State Audit with Corrective Action Response (or NELAC or A2LA Audit)[3]			
8. Sample Report[3]			
9. SOQ or Summary list of Technical Staff and Qualifications [3]			
10. SOPs for Methods to Be Loadshifted[2,3]			
11. For DoD Work: Statement that Lab quality system complies with QSM.			
12. For DoD Work: Approved by specific DoD Component laboratory approval process.			

1 - Required when emergency procedures are implemented.
2 - Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable. This requirement may also be fulfilled by supplying a table of SOPs with effective dates.
3 – If the laboratory has NELAC accreditation, Item #s 4 through 10 are not required.

On Site Audit Planned: YES NO If yes, Date Completed: _____ By Whom: _____

Comments: _____

Lab Acceptable for Subcontracting Work: YES NO Limitations: _____

QA Manager (Signature): _____ Date: _____
(Printed Name)

□ Forwarded to Contract Coordinator, by: _____ Date: _____

Figure 8-2.

Subcontract Laboratory Certification Verification Form

TestAmerica Buffalo

Certification Verification

Subcontract Lab

To be completed by Project Manager:

Date: _____

Project Manager: _____ **PROJECT #:** _____ **TASK:** _____

Client: _____ **State:** _____ **Client Notified** **Yes** ☐ **No** ☐

☐ **- Drinking Water** ☐ **- Waste Water** ☐ **- Solid** ☐ **- Air**

Method : _____

Parameters : _____ _____ _____

List Attached for multi-analyte/project specified reporting limits **Yes** ☐ **No** ☐

Requested Subcontract Lab: _____

To be completed by Q/A:

APPROVED

☐ - **Lab is Certified – forward samples**

☐ - **Analysis is not certifiable by State – forward samples**

_____

Signature (QA Dept.) and Date

NOT APPROVED

☐ **Appropriate certification not in place at this time.**
DO NOT SEND SAMPLES TO THIS SUBCONTRACT LAB

_____

Signature (QA Dept.) and Date

SECTION 9

PURCHASING SERVICES AND SUPPLIES
(NELAC 5.4.6)

9.1 OVERVIEW

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Capital expenditures are made in accordance with the Controlled Purchases Procedure, CW-F-S-004. Only one quote is required where the item being purchased is a sole source product, Examples of sole source capital expenditures are laboratory test equipment, client specified purchases and building leases. A minimum of two quotes is required where the opportunity exists to source from more than one vendor. All documentation related to the purchase of capital items will be maintained in the individual CapEx files located in Corporate Purchasing. Data will be held in accordance with the record retention policy.

TestAmerica will enter into formal contracts with vendors when it is advantageous to do so. Contracts will be signed in accordance with the Authorization Matrix Policy, CW-F-P-002. Examples of items that are purchased through vendor contracts are laboratory instruments, consumables, copiers and office supplies. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

Non-capital expenditure items are purchased through the requisition and approval process in JD Edwards or through other TestAmerica authorized methods (approved web-sites, purchasing cards). Labs have the ability to select from the approved vendors in JD Edwards.

9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 REAGENTS, STANDARDS & SUPPLIES

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents must meet with the requirements of the specific method and testing procedures for which they are

being purchased. Solvents and acids are pre-tested in accordance with Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001 and TestAmerica Buffalo SOP on Solvent Purity, SOP BF-OP-013.

9.3.1 Purchasing

The nature of the analytical laboratory demands that all material used in any of the procedures is of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. Purchase requisitions are placed into the J.D. Edwards system by designated departmental personnel. The listing of items available in the J.D. Edwards system has been approved for use by the corporate purchasing staff. Each purchase requisition receives final approval by the laboratory Operations Manager or purchasing coordinator before the order is submitted.

The analyst may also check the item out of the on-site consignment system that contains items approved for laboratory use.

9.3.2 Receiving

It is the responsibility of the purchasing coordinator to receive the shipment. It is the responsibility of the department that ordered the materials to date the material when received. Once the ordered reagents or materials are received, the department that submitted the order compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are kept online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

There are many different grades of analytical reagents available to the analyst. All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method.

- An expiration date can not be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.

- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

- If the dry chemical is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained along with the calibration raw data for which the reagent was used.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 200 psig or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a conductivity of less than 1mmho/cm (or resistivity of greater than 1.0 megaohm-cm) at 25°C. The conductivity is checked and recorded daily. If the water's conductivity is outside the specified limit, the Technical Director, Operations Manager and/or QA Manager must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Table 9-1 details specific storage instructions for reagents and chemicals. Section 22 discusses conditions for standard storage.

9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Operations Manager and/or the Laboratory Director. If they agree with the request the procedures outlined in Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which

piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, it is given a short name, such as HP-20, added to the equipment list described in Section 21 that is maintained by the Technical Director and IT must be notified so that can be linked for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (see Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department as specified in the laboratory's procedure for software verification. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 21. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers, Operations Manager and/or Technical Director.

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). The level of control used in the selection process is dependent on the anticipated spend and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report (CW-F-WI-009).

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (CW-F-WI-007 – refer to Figure 9-2).

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Director are consulted with vendor and product selection that have an impact on quality.

Table 9-1.

Storage of Reagents and Chemicals

Chemical	Storage Requirements
Concentrated Acids and Bases	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Bulk Dry Chemicals	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Working Solutions containing Organic Compounds	Stored as per method recommendation/ requirement. They are generally stored refrigerated at 4°C± 2°C.
Working Solutions containing only Inorganics	Stored at room temperature; refrigeration is optional.
Flammable Solvents	Stored in solvent cabinets at room temperature.
Non-Flammable Solvents	Stored separately from the flammable solvents in cabinets at room temperature.

Figure 9-1
Example – JD Edwards Vendor Add Request Form

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JD Edwards Vendor Add Request Form

Vendor name:	Lab location and individual making request:
Vendor address (remit to):	Vendor phone:
Vendor address (remit to):	Vendor fax:
Contact name:	Product / service provided:

Reason for Vendor Addition: Check all reasons that apply

☐ Cost Reduction	Estimated Annual Savings $
☐ Replace Current Vendor	Reason?
	Vendor being Replaced?
☐ New Product / Service	Describe:
☐ ISO Approved (Required for Aerotech / P&K only)	

Small Business:

Does this vendor help us to meet our small business objectives: _____

If yes, which category: _____

Personal and Ethical Considerations:

Is there any personal conflict of interest with a TestAmerica employee and the vendor listed above? _____

Have ethical considerations been taken into account in your evaluation of this vendor?_____

Can this product be sourced from another TestAmerica facility?_____

Please complete form and email to NCPurchasing@testamericainc.com or fax to (330) 966-9275.

I approve the addition of this vendor:

_____ Purchasing Manager - Patrick Eckman

_____ Corporate Controller - Leslie Bowers

Form No. CW-F-WI-007

SECTION 10

SERVICE TO THE CLIENT
(NELAC 5.4.7)

10.1 OVERVIEW

TestAmerica Buffalo cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements discussed in Section 5. The laboratory has procedures to ensure confidentiality to clients (Section 16 and 26).

Note: ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

10.2 SPECIAL SERVICES

The laboratory's standard procedures for reporting data are described in Section 26. When requested the following special services are provided:

- The laboratory will provide the client or the client's representative reasonable access to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- The laboratory will work with client-specified third party data validators as specified in the client's contract.
- The laboratory will provide the client with all requested information pertaining to the analysis of their samples. An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

10.3 CLIENT COMMUNICATION

Project managers are an important communication link to the clients. The lab shall inform its clients of any delays in project completion as well as any non-conformances in either sample receipt (refer to Section 24) or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Department Managers, the Operations Manager, the Technical Director and the Quality Manager are available to discuss any technical questions or concerns that the client may have.

10.4 REPORTING

The laboratory will work with the client to produce any special communication reports required by the contract.

10.5 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica's Sales and Marketing teams periodically develop lab and client specific surveys to assess client satisfaction.

SECTION 11

COMPLAINTS
(NELAC 5.4.8)

11.1 OVERVIEW

TestAmerica Buffalo believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that helps to continually improve processes and improving client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, communications, responsiveness, data, reports, invoicing and other functions expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for dealing with both external and internal complaints.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 13 (Corrective Actions) and is documented following the laboratory SOPs related to Data Quality Review (BF-QA-006), Corrective Action (BF-QA-005) and also using the Web-based Customer Complaint database. It is the laboratory's goal to provide a satisfactory resolution to complaints in a timely and professional manner.

11.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process and the documentation of the complaint.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likely hood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

11.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 13. In addition, Corporate Management, Sales and Marketing and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 13.

11.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 17)

SECTION 12

CONTROL OF NON-CONFORMING WORK
(NELAC 5.4.9)

12.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 13).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the department manager for advice. The department manager may elect to discuss it with the Technical Director, QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's job exception and corrective action system described in Section 13. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 20. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Laboratory Director, Technical Director, Operations Manager or QA Manager, documented and included in the project folder. Deviations must also be noted on the final report with a statement that the compound is not reported in compliance with the analytical method requirements and the reason.

12.2 RESPONSIBILITIES AND AUTHORITIES

SOP No. CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall, outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of the company's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director, the Technical Director, the Operations Manager or the QA Manager may exceptionally authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature

of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc.. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's job exception and corrective action procedures described in Section 13. This information may also need to be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility senior laboratory management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, Technical Director, Operations Manager, QA Manager, Customer Service Manager, Human Resources Manager and Business Development Manager. Suspected misrepresentation issues may also be reported to any member of the Corporate staff as identified in Ethics Policy, CA-L-P-001. The data integrity hotline (1-800-736-9407) may also be used. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO) and Quality Director within 24 hours.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, COO's – East and West, General Managers and the Quality Directors – East and West have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

12.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

SOP No. CA-L-S-001 distinguishes between situations when it would be appropriate for the laboratory QA Manager and Laboratory Director (or his/her designee) to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting (Section 13) in lieu of the data recall determination form contained in SOP No. CA-L-S-001.

12.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system (Section 13).

On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

12.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 12.2, Paragraph 3 above.

Prior to suspension/restriction, confidentiality will be respected, and the problem and the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 13 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (i.e., Project Management, Log-in, etc...). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, Operations Manager, QA Manager, Department Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, the Customer Service Manager and Sales and Marketing should be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report as described in Section 13.

SECTION 13

CORRECTIVE ACTION
(NELAC 5.4.10)

13.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Job Exception Reports (JER) and Corrective Action Reports (CAR) (refer to Figure 13-1).

13.2 DEFINITIONS

- **Correction**: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions are contained in the method specific SOPs. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

- **Corrective Action**: The action taken is not only a correction made to the immediate event, but a change in process, procedure or behavior that is required to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

13.3 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc..

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigation.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify Systematic Problems before they become serious.
- Identify and track Client complaints and provide resolution (see more on client complaints in Section 11).

13.3.1 Job Exception Report (JER) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non matrix related)

- Isolated Reporting / Calculation Errors

13.3.2 Data Quality Review (DQR) - is used to document the following types of corrective actions:

- Client Concerns regarding analytical results
- Project Management concerns regarding specific analytical results

13.3.3 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of JERs.
- Issues found while reviewing JERs that warrant further investigation.
- Questionable trends that are found in the monthly review of DQRs or client complaints
- Internal and External Audit Findings Failed or Unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic Reporting / Calculation Errors

13.4 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

13.4.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. A JER or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 13-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Department Manager, Operations Manager, Technical Director, or QA Manager (or QA designee) is consulted.

13.4.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The JER or CAR is used for this documentation.

13.4.3 Monitoring of the Corrective Actions

- The Department Manager, Operations Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions will be documented and re-evaluated until acceptable resolution is achieved. Department Managers and the Operations Manager are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each JER and DQR are entered into a database and each CAR is entered into a spreadsheet for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly JERs, DQRs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 17). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

13.4.4 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements. (Section 16 includes additional information regarding internal audit procedures.)
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

13.5 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 12 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of a JER or CAR.

Table 13-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs.

Table 13-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, QAM Sections 20 and 21, and SOP CA-L-S-001 (Internal Investigation of Potential Data Discrepancies and Determination for Data Recall). All corrective actions are reviewed at a minimum monthly by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written JER and appropriate corrective action (e.g., reanalysis) is taken and documented.

13.6 BASIC CORRECTIONS

When mistakes occur in records, each mistake shall be crossed-out, and not erased, deleted, made illegible, or otherwise obliterated (e.g. no white-out), and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 13-1.
Example – Corrective Action Notice

TestAmerica
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CAN Statement
Revision 2
August 5, 2005

CORRECTIVE ACTION NOTICE

Date Issued:	**Issued By:**
Date Required:	**Responsible Party:**
Source of Issue:	

Explanation of Issue:

Investigation Summary:

Root Cause:

Impact on Client Data:

Corrective Action or Resolution:	
Timetable for Action:	
Means to Document Corrective Action:	
Completed By:	**Date:**
Approved By:	**Date:**

Follow-Up Comments:	
Follow-Up By:	**Date:**

Table 13-1.

Example – General Corrective Action Procedures

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Initial Instrument Blank *(Analyst)*	- Instrument response < MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Department Manager)*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP. (or refer to Appendix 4 or Section 21)	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Department Manager)*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in LIMs.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set.
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in LIMs.	- Batch must be re-prepared and re-analyzed. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.
Proficiency Testing (PT) Samples *(QA Manager, Department Manager)*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Department Manager, Operations Manager, Technical Director, Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors *(Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager, QA Manager, Corporate QA, Corporate Management)*	- SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CA-L-S-001.
Client Complaints *(Project Managers, Lab Director, Sales and Marketing, QA Manager)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
QA Monthly Report (Refer to Section 17 for an example) *(QA Manager, Lab Director, Operations Manager Department Managers)*	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation *(EH&S Coordinator, Lab Director, Operations Manager, Department Manager)*	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through EH&S office.

Note:

1. Except as noted below for certain compounds, the method blank should be below the reporting limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, acetone, 2-butanone and phthalates. This allowance presumes that the reporting limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur.

SECTION 14.0

PREVENTIVE ACTION
(NELAC 5.4.11)

14.1 OVERVIEW

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes ***TestAmerica Buffalo***'s commitment to its Quality Assurance (QA) program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc..

The monthly Quality Assurance Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's Corrective Action process (Section 13) is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

14.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review

- **Note:** There may be varying levels of formality and documentation during the preventive action process due to the simplicity/complexity of the action taken.

14.1.2 Any Preventive Actions undertaken or attempted shall be taken into account during the Annual Management Review (Section 17). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program will provide management a measure for evaluation.

14.2 MANAGEMENT OF CHANGE

Reserved

SECTION 15.0

CONTROL OF RECORDS
(NELAC 5.4.12)

TestAmerica Buffalo maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. TestAmerica Buffalo SOP BF-GP-015, Record Storage and Retention specifies additional storage, archiving and retention procedures.

15.1 OVERVIEW

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 15-1. Quality records are maintained by the Quality Assurance (QA) Manager in both hardcopy and electronic form. Hardcopy records are retained in the QA archive room and electronic records are retained in network folders which are backed up as part of the regular network backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Hardcopy technical records are maintained by the Data Deliverables Manager while electronic technical records are maintained by the IT Adminstrator.

Table 15-1. Record Index[1]

Technical Records	Official Documents	QA Records	Project Records	Administrative Records
Retention: 5 Years from analytical report issue*	5 Years from document retirement date*	5 Years from archival* **Data Investigation:** 5years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)	5 Years from analytical report issue*	**Personnel:** 7 Years (HR Records must be maintained as per Policy CW-L-P-001) **Finance:** See Accounting and Control Procedures Manual
Raw Data Logbooks[2] Standards Certificates Analytical	Quality Assurance Manual (QAM)	Internal and External Audits/ Responses	Sample receipt and COC Documentation	Finance and Accounting
	Work Instructions	Certifications	Contracts and Amendments	EH&S Manual, Permits, Disposal Records
	SOPs	Corrective/Preventive Action	Correspondence	Employee Handbook
	Manuals	Management Reviews	QAPP	Personnel files,

Technical Records	Official Documents	QA Records	Project Records	Administrative Records
Retention: 5 Years from analytical report issue*	5 Years from document retirement date*	5 Years from archival* **Data Investigation:** 5years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)	5 Years from analytical report issue*	**Personnel:** 7 Years (HR Records must be maintained as per Policy CW-L-P-001) **Finance:** See Accounting and Control Procedures Manual
Records Lab Reports		Method & Software Validation, Verification data	SAP	Employee Signature & Initials, Administrative Training Records (e.g., Ethics)
		Data Investigation	Telephone Logbooks	Administrative Policies
	Policies	Performance Evaluations	Lab Reports	Technical Training Records

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 15-2.

All records are legible and stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. Retention of records is maintained on-site at the laboratory for at least 3 months after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless other wise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 15-2 have lengthier retention requirements and are subject to the requirements in Section 15.1.3. Policy CW-L-P-001 (Record Retention) provides additional information on record retention requirements.

15.1.1 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 15-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data. Specific Information related to archival of data for greater than 5 years may be found in TestAmerica Buffalo SOP BF-GP-015.

Table 15-2. Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	5 years
NY Potable Water NYCRR Part 55-2	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements are noted with the archive documents or addressed in TestAmerica Buffalo facility-specific records retention procedure BF-GP-015.

15.1.2 All records are held secure and in confidence. Records maintained at the laboratory are located in the locked on-site storage room. Records archived off-site are stored in a secure location. Access to the off-site storage facility is controlled and logs are maintained for the documented removal/return of records

15.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, see section 20.12.1 'Computer and Electronic Data Related Requirements' for more information. TestAmerica Buffalo SOP BF-GP-015 also contains specific information for archival of scanned data.

15.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (records stored off site should be accessible within 2 business days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the chain of custody is stored with the project file and the

Job Number analytical service request form (ASRF) generated by the LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a Job Number, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set. Instrument data is stored sequentially by instrument. Calibration data for a given sequence are maintained in the order of the analysis. Sample data are stored on a job number basis in the project file or as part of the daily batch or sequence. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks, bench sheets or excel spreadsheets are used to record and file data. Standard and reagent information is recorded in logbooks or on the raw data for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in TestAmerica SOP BF-GP-015.

- Also refer to Section 20.13.1 'Computer and Electronic Data Related Requirements'.

15.2 TECHNICAL AND ANALYTICAL RECORDS

15.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement (refer to Section 15.1). The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and checking of results.

15.2.2 Observations, data and calculations are recorded at the time they are made and are identifiable to the specific task.

15.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis and time of analysis is required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in the method specific SOPs, in the instrument method detail records or the instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, temperatures, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

15.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

15.3.1 Sample Handling Records

Sample handling and tracking is discussed in Section 24. Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

15.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. See Table 15-1.

15.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

15.5.1 All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available to the accrediting body upon request.

15.5.2 All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

15.5.3 Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

15.5.4 ***TestAmerica Buffalo*** has a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per instrument or analysis basis, and are numbered sequentially as they are issued. No instrument or analysis has more than one active notebook at a time, so all data are recorded sequentially within a series of sequential notebooks. Bench sheets and raw data sequence files are filed sequentially by date. Standard and reagent information is maintained in logbooks which are maintained on a departmental basis and are numbered sequentially as they are issued or as they are archived by QA.

15.5.5 Records are considered archived when moved off-site. Access to archived hard-copy information is documented with an access log and in/out records is used to note data that is removed and returned. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to the data is limited to laboratory and company employees.

15.5.6 In the event that the laboratory transfers ownership or goes out of business, **TestAmerica Buffalo** shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

15.5.7 **Records Disposal**

15.5.7.1 Records are removed from the archive and disposed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration.

15.5.7.2 Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

15.5.7.3 If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required. [Refer to Policy No. CW-L-P-001 (Records Retention).]

SECTION 16

AUDITS
(NELAC 5.4.13)

16.1 OVERVIEW

Audits measure laboratory performance and insure compliance with accreditation/certification and project requirements. Audits specifically provide management with an on-going assessment of the quality of results produced by the laboratory, including how well the policies and procedures of the QA system and the Ethics and Data Integrity Program are being executed. They are also instrumental in identifying areas where improvement in the QA system will increase the reliability of data. There are two principle types of audits: Internal and External. Internal audits are performed by laboratory or corporate personnel. External audits are conducted by regulators, clients or third-party auditing firms. In either case, the assessment to program requirements is the focus.

Table 16-1. Audit Types and Frequency

Internal Audits	Description	Performed by	Frequency
	Analyst & Method Compliance	QA Department or Designee	- 100% of all methods over a two year period. - 100% of all analysts annually.
	Instrument	QA Department or Designee	100% of all organic instruments and any inorganic chromatography instruments over a two year period.
	Work Order/ Final Report	QA Department or Designee	- 1 complete report each month.
	Support Systems	QA Department or Designee	- Annual for entire labs support departments & equipment (e.g., thermometers, balances), can be divided into sub-sections over the course of the year.
	Performance Audits (Double-Blind PTs)	Corporate QA, Laboratory QA Department or Designee	- As needed.
	Special	QA Department or Designee	- As Needed
External Audits	**Description**	**Performed by**	**Frequency**
	Program / Method Compliance	Regulatory Agencies, Clients, accreditation organizations	- As required by program and/or clients needs
	Performance Audits	Provided by a third party.	- As required by a client or regulatory agency. Generally provided semi-annually through the analysis of PT samples.

16.2 INTERNAL AUDITS

Annually, the laboratory prepares a schedule of internal audits to be performed throughout the year. As previously stated, these audits verify and monitor that operations continue to comply with the requirements of the laboratory's QA Manual and the Corporate Ethics Program. A

schedule of the internal audits is maintained by the QA Manager in the *Internal Audit Workbook*. An example can be found in Attachment 1.

It is the responsibility of the QA Manager to plan and organize audits in consideration of the laboratory work load and the department personnel schedules so that all pertinent personnel and operations are thoroughly reviewed. When designees (other than QA department personnel & approved by the QA Manager), perform audits, the QA Manager shall insure that these persons do not audit their own activities except when it can be demonstrated that an effective audit will be carried out. In general, the auditor:

- is neither the person responsible for the process being audited nor the immediate supervisor of the person responsible for the project/process.
- Is free of any conflicts of interest.
- Is free from bias and influences that could affect objectivity.

Laboratory personnel (e.g., supervisors and analysts) may assist with both method and support system audits as long as the items listed in the above paragraph are observed. These audits are conducted according to defined criteria listed in the checklists of the *Internal Audit Workbook.* These personnel must be approved by the QA Manager; and must complete the audit checklists in their entirety. This process introduces analyst experience and insight into the laboratory's auditing program.

The auditor must review the previous audit report and identify all items for verification of corrective actions. A primary focus will be dedicated to the ability of the laboratory to correct root-cause deficiencies and that the corrective action has been implemented and sustained as documented.

16.2.1 Systems

An annual systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems and client and State requirements. This audit is performed in portions throughout the year through method, analyst, instrument, work order/final report and support system audits. Audits are documented and reported to management within 1 week of their performance. Systems audits cover all departments of the facility, both operational and support. When performed individually, the multiple audits are compiled into one systems audit package at the end of the year (*Internal Audit Workbook*). The systems audits may also be combined and performed as a single comprehensive internal systems audit during a 1-2 week time frame.

16.2.1.1 Method, Analyst, Instrument and Work Order/Final Report Audits

Procedures for the method compliance, analyst, instrument and work order/final report audits are incorporated by reference to SOP No. CA-Q-S-004, Method Compliance and Data Authenticity Audits. These audits are not mutually exclusive. For example, the performance of a method audit will also cover multiple analysts and instruments. The laboratory's goal is to review all analysts and instruments as described in SOP No. CA-Q-S-004. The laboratory will also audit all methods within a two year time period and audit a minimum of one Work Order/Final Report from receiving through reporting on a monthly basis.

16.2.1.2 Support Systems

Support system audits are performed to ensure that all departments & ancillary equipment are operating according to prescribed criteria. Support system audits include the review of both non-analytical and operational departments. Support equipment audits (e.g., metrology items) include the review of balance calibrations, weight calibrations; water quality testing, etc.. Non-analytical may include sample receiving and bottle preparation. These types of support audits ensure that the operations are being performed to support ethical data as well as ensuring the accuracy & precision of the utilized equipment.

These audits can be performed in portions throughout the year or in one scheduled session. However, the audit schedule must document that these aspects are reviewed annually. Many of the metrology systems are considered to be surveillance activities that can be monitored by QA personnel or delegated to specified department personnel. These surveillance activities are performed on a semi-annual basis unless issues warrant a greater frequency or previous audits continually showing no deficiencies allow the frequency to be reduced to once a year.

An example audit checklist can be found in Attachment 2. Instructions for reporting findings are included in the *Internal Audit Workbook*. In general, findings are reported to management within 1 week of the audit and a response is due from management within 30 days.

16.2.2 Performance Audits

Corporate QA may arrange for double blind PT studies to be performed in the laboratories. Results are given to Management and Corrective actions of any findings are coordinated at each facility by the QA Managers and Laboratory Directors/Managers. These studies are performed on an as needed basis. They may be performed when concerns are raised regarding the performance of a particular method in specific laboratories, periodically to evaluate methods that may not normally be covered in the external PT program or may be used in the process of developing best practices. The local QA Manager may also arrange for PT studies on an as needed basis. (Refer to Section 16.3.2 for additional information on Performance Audits.)

16.2.3 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

16.3 EXTERNAL AUDITS

TestAmerica facilities are routinely audited by clients and external regulatory authorities. External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. The laboratory department managers are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due

in the time allotted by the client or agency performing the audit. This time frame is generally 30 days.

TestAmerica Buffalo cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

16.3.1 Confidential Business Information (CBI) Considerations

During on-site audits, on-site auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

16.3.2 Performance Audits

The laboratory is involved in performance audits conducted semi-annually through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies:

Water Supply: 2 times per year
Water Pollution: 2 times per year
Soil: 2 times per year
DMR-QA: once per year
Client specific: as requested

- It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Further, where PT samples present special or unique problems in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

- PTs generally do not have holding times associated with them. In the absence of any holding time requirement, it is recommended that the holding time begin when the PT sample is prepared according to the manufacturers instructions. Holding times should apply to full volume PT samples only if the provider gives a meaningful "sampling date". If this is not provided, it is recommended that the date/time of opening of the full volume sample be considered the beginning of holding time.

- Login will obtain the COC information from the documentation provided with the PTs with review by QA or other designated staff.

- Vials will be prepared as required in the instruction set provided with the samples. After preparation to full volume the sample may be spiked, digested, concentrated, etc., as would be done for any normal sample requiring similar analysis.

- PT samples will not undergo multiple preps, multiple runs, multiple methods (unless being used to evaluate multiple methods), multiple dilutions, UNLESS this is what would be done to a normal client sample (e.g. if a client requests, as PT clients do, that we split VOA coeluters, then dual analysis IS normal practice).

- The type, composition, concentration and frequency of quality control samples analyzed with the PT samples shall be the same as with routine environmental samples.

- Instructions may be included in the laboratory's SOPs for how low level samples are analyzed, including concentration of the sample or adjustment of the normality of titrant. When a PT sample falls below the range of the routine analytical method, the low-level procedure may be used.

- No special reviews shall be performed by operation and QA, UNLESS this is what would be done to a normal client sample. To the degree that special report forms or login procedures are required by the PT supplier, it is reasonable that the laboratory WOULD apply special review procedures, as would be done for any client requesting unusual reporting or login processes.

- Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

16.4 AUDIT FINDINGS

Internal or External Audit findings should be documented using the corrective action process. (refer to Section 13). The laboratory is expected to prepare a response to audit findings within 30 days of receipt of an audit report unless the report specifies a different time frame. The response may include action plans that could not be completed within the 30 day timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Responsibility for developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

The procedures must be in accordance to SOP No. CA-L-S-001, Internal Investigations of Data Discrepancies and Determination of Data Recall.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

Figure 16-1.

Example - Internal Audit Workbook

Laboratory: TestAmerica Buffalo

Last Updated:
7/16/2007

Internal Audit Schedule 2008

Item No.	Area Audited	Audit Type	Audit Cycle	Scheduled	Date Audited	Date Closed	Tab No.	Comments
1	Balances	System	6 mo				3	
2	Temperature Logs/ Thermometers	System	6 mo				4	
3	Sample Storage and Disposal	System	1 yr				5	
4	Maintenance Logs	System	6 mo				6	
5	Holding Blanks for Volatile Ref/Freezers (where required)	System	6 mo				7	
6	Lab Water Quality Testing	System	6 mo				8	
7	Sample Control (Log In)	System	1 yr				9	
8	Shipping Procedures	System	1 yr				10	
9	Computer Operations (LIMS)	System	1 yr				11	
10	SOP Distribution System	System	1 yr				12	
11	Archiving of Paper Records	System	1 yr				13	
12	Statistical Process Control	System	1 yr				14	
13	Electronic Archiving	System	1 yr				15	
14	Data Review System	System	1 yr				16	
15	Final Report Generation	System	1 yr				17	
16	Standards/Reagents	System	6 mo				18	
17	Manual Integration	System	1 yr				19	
18	Corrective Action System	System	1 yr				20	
19	Training Records	System	6 mo				21	
20	MDLs	System	1 yr				22	
21	SOPs – Prep/Review/Update Process	System	1 yr				23	
22	Purchasing/Procurement	System	1 yr				24	
23	Pipette/Diluter/Dispenser Calibration Check	System	6 mo				25	
24	Subcontract Lab Approval	System	1 yr				26	
25	Customer Complaint System	System	1 yr				27	
26	Methods	Method	2 yr				28	
	Checklist Pending							

Figure 16-2.

Example – Internal Audit System Checklist: Corrective Actions

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

(Summary Page)

TestAmerica <Location>

INTERNAL AUDIT - Corrective Actions

[Printed Name(s) or Date(s)]

Area Audited:	
Auditor:	
Date:	
Persons Contacted During Audit:	
Date Reported to Department Manager:	
Reported To:	
Date Reported to Lab Director/Manager:	
Reported To:	
Date Response Due:	
Response Received and Accepted by QA Manager:	
Associated Corrective Action Report Number(s):	
Scheduled Follow-up:	

Item	Requirement	Ref.	Y	N	NA	Evidence/Comments	Follow Up
1	Does the laboratory have a corrective action program in place?	5.4.10.1					
2	Does the laboratory have a current corrective action SOP or is this information in the QA Manual?	5.4.10.1					
3	Do all laboratory personnel have documented training and access to initiate corrective actions?	5.4.10.1					
4	Are causes clearly identified by department, staff name, scope of *issue (how many reports affected)*?	5.4.10.6					
5	Is a root cause for the issue identified?	5.4.10.2					
6	Is a corrective action (plan) clearly described?						
7	Was the corrective action fully implemented?						
8	Is documentation (if applicable) completed as specifed by the corrective action (training, revised SOP, etc)						
9	Has a follow-up assessment been conducted to verify the corrective action was successful?						
10	Are corrective actions reviewed on a regular basis by management?	5.4.10.6a 5					
11	Is there a defined distribution flow for corrective action notification, review, closure, and follow-up?	5.4.10.6a					
12	Are non-conformances reviewed on a regular basis and used, if necessary, to initiate root cause corrective actions?						
13	Does the lab have a documented procedure for QC corrective action (i.e., documented within each method / parameter SOP or in the QA Manual)?	4.10.1					
14	***Verify Corrective Actions from previous systems audits. List Items:***						
15							
16							
17							

Auditor Signature:_____

Primary Reference(s): Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices
NELAC Standard, June 2003
DoD Quality Systems Manual, Version 3, January 2006
EPA Manual for the Certification of Laboratories Analyzing Drinking Water

SECTION 17

MANAGEMENT REVIEWS
(NELAC 5.4.14)

17.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director for review and comments. The final report shall be submitted to the Operation Manager as well as the appropriate Quality Director and General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. At a minimum, the report content will contain the items listed below. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

The TestAmerica QA Report template is comprised of a discussion of three key QA issues facing the laboratory and ten specific sections (Figure 17-1):

- **Metrics:** Describe actions or improvement activities underway to address any outlying quality metrics that have been reported in the monthly Quality Systems Metrics Table.
- **SOPs:** Report SOPs that have been finalized and report status of any outstanding SOP reviews.
- **Corrective Actions:** Describe highlights and the most frequent cause for report revisions and corrective/preventive action measures underway. Include a discussion of any recalls handled at the lab level as per Section 6.2.2 in the Investigation/Recall SOP (SOP: CA-L-S-001). Include a section for client feedback and complaints. Include both positive and negative feedback. Describe the most serious client complaints and resolutions in progress.
- **MDLs and Control Limits:** Report which MDLs/ MDL verifications are due. Report the same for Control Limits.
- **Audits:** Report Internal and External Audits that were conducted. Include all relevant information such as which methods, by whom, corrective actions needed by when and discuss unresolved audit findings.
- **Performance Testing (PT) Samples:** Report the PT tests that are currently being tested with their due dates, report recent PT results by study, acceptable, total reported and the month and year.
- **Certifications:** Report on any certification programs being worked on by due date, packages completed. Describe any issues, lapses, or potential revocations.
- **Regulatory Updates:** Include information on new state or federal regulations that may impact the laboratory. Report new methods that require new instrumentation, deletion of methods, changes in sampling requirements and frequencies etc...
- **Miscellaneous:** Include any issues that may impact quality within the laboratory.
- **Next Month:** Report on plans for the upcoming month.
- **Lab Director Comments Section:** This section gives the Laboratory Director the opportunity to comment on issues discussed in the report and to document plans to resolve

these issues. Unresolved issues that reappear in subsequent monthly reports must be commented on by the Laboratory Director.

- **Quality Systems Metrics Table:** The report also includes statistical results that are used to assess the effectiveness of the quality system. Effective quality systems are the responsibility of the entire laboratory staff. Each laboratory provides their results in a template provided by Corporate QA (Figure 17-2).

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The VP-QA/EHS prepares a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Analytical Division Senior Management Team and General Managers.

17.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Technical Director, Operations Manager, Customer Service Manager, QA Manager) conducts an annual review of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This review uses information generated during the preceding year to assess the "big picture" by ensuring that routine quality actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review (refer to Section 17.1) should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior Senior Management team meetings. Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources.
    - Adequacy of policies and procedures.
    - Future plans for resources and testing capability and capacity.

- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

The annual review includes the previous 12 months. Based on the annual review, a report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

The QA Manual is also reviewed at this time and revised to reflect any significant changes made to the quality systems.

17.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The Corporate Data Investigation/ Recall SOP shall be followed (SOP No. CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

The Chairman/CEO, President/CEO, COOs and Quality Directors receive a monthly report from the VP of Quality and EHS summarizing any current data integrity or data recall investigations as described in SOP No. CA-L-S-001. The General Manager's are also made aware of progress on these issues for their specific labs.

Figure 17-1.

Example - QA Monthly Report to Management

LABORATORY: x
PERIOD COVERED: Month/Year
PREPARED BY: x DATE: Month Day, Year
DISTRIBUTED TO: xx (Include LD, GM, QA Director, etc...)

THREE KEY ISSUES FOR MONTH:
Include a discussion of three key issues that were focused in on this month.
1. x
2. x
3. x

---

1. METRICS
Describe actions or improvement activities underway to address any outlying quality metrics.

2. SOPs

See Tab for SOP specifics.

The following SOPs were finalized (or reviewed for accuracy): (See Tab)

The following SOPs are due to QA: xx

In QA to complete: xx

3. CORRECTIVE ACTION

Highlights: xx

Revised Reports:
Describe the most frequent cause for report revisions and corrective/preventive action measures underway.

Data Investigations/Recalls (Corporate Data Investigation/Recall SOP) :
Include a discussion of any recalls handled at the lab level as Corp SOP.

Client Feedback and Complaints:
Include both positive and negative feedback.

Describe the most serious client complaints) and resolutions in progress.

4. MDLs AND CONTROL LIMITS

MDLs Due:

Control Limits Due:

5. AUDITS

INTERNAL AUDITS

Discuss Any Outstanding Issues (or Attach Summary):

EXTERNAL AUDITS
Discuss Any Outstanding Issues (or Attach Summary):

6. PT SAMPLES

The following PT samples are now in house (Due Dates):
xx

7. CERTIFICATIONS

Certification Packages Being Worked On (Include Due Date):
x

Describe any issues, lapses, or potential revocations.

8. REGULATORY UPDATE
Include information on new state or federal regulations that may impact the laboratory – new methods that require new instrumentation, deletion of methods, changes in sampling requirements or frequencies, …

9. MISCELLANEOUS
Include any issues that may impact quality within the laboratory.

10. NEXT MONTH
Items planned for next month.

LAB DIRECTOR COMMENTS AND PLANNED CORRECTIVE ACTIONS:	
LAB DIRECTOR REVIEW:	DATE:

Figure 17-2.

Example - Laboratory Metrics Categories

Reports for month
Reports revised due to lab error
% Revised Reports
# of Data Recall Investigations
# of Reports Actually Recalled
Corrective Action Reports
Corrective Action Reports still open
Total Number of Unresolved Open Corrective Action Reports
% of Unresolved Open Corrective Action Reports
# Reports independent QA reviewed
% QA Data Review: Reports
# Technical staff (Analysts/technicians, including Temps)
# of Analyst work product reviewed year-to-date
# of Analytical instruments w/electronic data file storage capability
# of Analytical instruments reviewed for data authenticity year-to-date
% Analyst/Instrument Data Authenticity Audits
 # Client Complaints
 # Client Compliments
# of planned internal audits
# of planned internal method audits performed year-to-date
% Annual Internal Audits Complete
# of Open Internal Audit Findings Past Due
Total Number of External Audit Findings
# of Open External Audit Findings Past Due
% External Audit Findings Past Due
# of PT analytes participated and received scores
# of PT analytes not acceptable
% PT Cumulative Score
 # PT Repeat Analyte Failures Cumulative (analyte failed more than once in 4 consecutive studies by PT Type) (only applies to failed analytes)
SOPs

SOPs Reviewed/revised within 24 months
Methods **or Administrative procedures** without approved SOPs
SOP Status
Method certification Losses due to performance/audit issues
Hold Time Violations due to lab error
Date of Last Comprehensive Ethics Training Session
 # Staff that haven't Received Comprehensive Ethics Training (>30 Days From Employment Date)
MDL Status (Good, Fair, or Poor) >90%, >70%, <70%
Training Documentation Records (Good, Fair, or Poor)
LQM Revision/review Date
QAM Updated to New Integrated Template
Last Annual Internal Audit Date (Opened, Closed)
Last Management QS Review Date
#SOPs required for 12 month review cycle (DOD *or drinking water)*
#SOPs for 12 month cycle/revised within 12 months *(Includes QS and Methods Listed in QSM)*
12 month % SOP Status *(Includes QS and Methods Listed in QSM)*

SECTION 18

PERSONNEL
(NELAC 5.5.2)

18.1 OVERVIEW

TestAmerica's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Appendix 2.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

18.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

TestAmerica makes every effort to hire analytical staff that possesses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. There are competent analysts and technicians in the industry who have not earned a college degree. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are

located in the TestAmerica Buffalo Human Resource office (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, pipette, quantitation techniques, etc. are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Directors/Department Managers – **General**	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

18.3 TRAINING

TestAmerica is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Refer to EH&S Manual	All
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 20.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- The Human Resource office maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in TestAmerica Buffalo SOP BF-QA-004, Laboratory Personnel Training.

18.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established an Ethics Policy No. CA-L-P-001 and an Ethics Statement/Agreement (Appendix 1). All initial and annual training is documented by signature on the signed Ethics Policy and Code of Ethical Conduct

demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy (Appendix 1)
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 19

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS
(NELAC 5.5.3)

19.1 OVERVIEW

TestAmerica Buffalo is a 32,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc.. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for field operations, bottle kit preparation, sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis and administrative functions.

19.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may effect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. Key equipment has been provided with back-up power supply in the event of a power outage.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels (refer to Section 12).

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

19.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

19.4 FLOOR PLAN

A floor plan can be found in Appendix 3.

19.5 BUILDING SECURITY

Building pass cards and alarm codes are distributed to all facility employees.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of TestAmerica Buffalo. [The reason for this is that it is important to know who is in the building in case of a safety emergency. The visitors logbook is used to ensure that everyone got out of the building safely.] In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 20.0

TEST METHODS AND METHOD VALIDATION
(NELAC 5.5.4)

20.1 OVERVIEW

TestAmerica Buffalo uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

20.2 STANDARD OPERATING PROCEDURES (SOPs)

TestAmerica Buffalo maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory (refer to Section 6 on Document Control):

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for preparation, review, revision and control are incorporated by reference to Corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP) and Laboratory SOP BF-QA-003, Procedure for Writing, Reviewing and Revising Controlled Quality Documents (QAM, SOP, etc)
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

20.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP. Refer to the corporate SOP CW-Q-S-002 "Writing a Standard Operating Procedure" for content and requirements of technical and non-technical SOPs.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from

the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

20.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

20.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

In general, TestAmerica Buffalo follows procedures from the referenced methods shown below in 20.4.1.1.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

20.4.1.1 The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999*
- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*

- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Statement of Work for Inorganics Analysis, ILM04.1, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- *Statement of Work for Inorganics Analysis, ILM05.2/5.3, USEPA Contract Laboratory Program Multi-media, Multi-concentration*
- *Statement of Work for Organics Analysis, OLM04.2/4.3, USEPA Contract Laboratory Program, Multi-media, Multi-concentration.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)* ***(DW labs only)***
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *New York State DEC Analytical Services Protocol, 2005*
- *New York State DOH Methods Manual*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

20.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

20.4.2.1 A demonstration of capability is performed whenever there is a significant change in instrument type, method or personnel.

20.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Department Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures (refer to Section 15, Control of Records).

20.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method).
- The reporting limit is set at or above the first standard of the curve for the analyte.
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*
- Refer to Section 12 (Control of Non-Conforming Work).

20.4.3 Initial Demonstration of Capability (IDOC) Procedures

Procedures for generation of IDOCs are detailed below and in laboratory SOP BF-QA-004, Laboratory Personnel Training.

20.4.3.1 The spiking standard used must be prepared independently from those used in instrument calibration.

20.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

20.4.3.3 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

20.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

20.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

20.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

20.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 20.4.3.3 above.
- Beginning with 20.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 20.4.3.1 above.

A certification statement (see Figure 20-1) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

20.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP/Methods Manual (Section 20.2) and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method. The information included in the checklist below (Figure 20-2) is needed before samples are accepted for analysis by a new method.

20.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled. (From 2003 NELAC Standard)

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to

confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

20.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

20.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

20.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. The laboratory determinations of MDLs are described in Section 20.7.

20.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

20.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

20.6.1.5 Determination of Range

Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

20.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

20.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

20.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

20.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 20.7.10). The analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used.

20.7.1 MDL's are initially performed for each individual instrument and non-microbiological method analysis. Unless there are requirements to the contrary, the laboratory will use the highest calculated MDL for all instruments used for a given method as the MDL for reporting purposes. This MDL is not required for methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report values to the MDL. For titration and gravimetric methods where there is no additional preparation involved, the MDL is based on the lowest discernable unit of measure that can be observed.

20.7.2 MDL's must be run against acceptable instrument QC, including ICV's and Tunes. This is to insure that the instrument is in proper working condition and falsely high or low MDL's are not calculated.

20.7.3 Use only clean matrix which is free of target analytes (e.g.: Laboratory reagent water, Ottawa Sand) unless a project specific MDL is required in a field sample matrix.

20.7.4 The Reporting Limit (also may be referred to as Limit of Quantitation or LOQ) should generally be between 2 and 5 times the MDL (or 10X the standard deviation of the MDL for Wisconsin projects). If the MDL is being performed during method development, use this guideline to determine the Reporting Limit for the analysis. If a sample is diluted, the reported MDL is adjusted according to the dilution factor.

20.7.5 The calculated MDL cannot be greater than the spike amount.

20.7.6 If the most recent calculated MDL does not permit qualitative identification of the analyte then the laboratory may use technical judgment for establishing the MDL (e.g., calculate what level would give a qualitative ID, compare with IDL (20.8), spike at a level where qualitative ID is determined and assign that value as MDL, minimum sensitivity requirements, standard deviation of method blanks over time, etc.). Alternate verification procedures have been included in laboratory SOP BF-QA-001, Determination of Method Detection Limits.

20.7.7 Each of the 7 spikes must be qualitatively identifiable (e.g., appear in both columns for dual column methods, characteristic ions for GCMS mass spectra, etc). Manual integrations to force the baseline for detection are not allowed.

20.7.8 The initial MDL is calculated as follows:

$$MDL = t_{(n-1,\ 1-a\ =\ 0.99)} \times (\text{Standard Deviation of replicates})$$

where $t_{(n-1,\ 1-a\ =\ 0.99)} = 3.143$ for seven replicates.

20.7.9 Subsequent to the initial MDL determination, periodic MDL verification, confirmation or determinations may be performed by the procedure in 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices (e.g., method blanks over time, single standard spikes that have been subjected to applicable sample prep processes, etc.). The alternate procedures utilized at TestAmerica Buffalo are detailed in SOP BF-QA-001, Determination of Method Detection Limits.

20.7.10 Because of the inherent variability in results outside of the calibration range, TestAmerica does not recommend the reporting of results below the lowest calibration point in a curve; however, it is recognized that some projects and agencies require the reporting of results below the RL. Any result that falls between the MDL and the Reporting limit, when reported, will be qualified as an estimated value.

20.7.11 Detections reported down to the MDL must be qualitatively identified.

20.7.12 MDLs and Reporting limits are adjusted in LIMs based on moisture content and sample aliquot size. The formatting of the MDL (adjusted or not adjusted) on the final report is based on the project specifications.

20.8 INSTRUMENT DETECTION LIMITS (IDL)

20.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

20.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation. (For CLP procedures, the IDL is determined using the standard deviation of 7 replicate spike analyses on each of 3 non-consecutive days.)

20.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

20.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

20.9.1 Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, CVAA, etc.) and 1-4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified or see section 20.7.9 for other options. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established (See 20.7.9). MDLs must be verified at least annually

20.9.2 When a Reporting limit is established, it must be initially verified by the analysis of a low level standard or QC sample (LCS at 1-2 the reporting limit) and annually thereafter. Unless there are requirements to the contrary the acceptance criteria is $\pm$ 50%. The annual requirement is waved for methods that have an annually verified MDL.

20.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes.

For GC, HPLC and IC methods, there must be sufficient separation between analyte peaks so as to not misidentify analytes. In the mid-level standard, the distance between the valley and peak height cannot be any less than 25% of the sum of the peak heights of the analytes. This also applies to GCMS in the case where the two compounds share the same quantitation ion.

Note: Some analytes do not separate sufficiently to be able to identify or quantitate them as separate analytes (e.g. m-xylene and p-xylene) and are quantitated and reported as a single analyte (e.g. m,p-xylenes).

Once the analyst has determined that the instrument is in optimum working condition through calibration and calibration verification procedures, he or she uses a mid-range calibration or calibration verification standard to establish the retention times for each of the individual analytes in a method. The analyst makes three injections of the same standard over a 72-hour (24 hr period for method 300.0) period, tabulating the retention times for each analyte for each of the three injections. The width of retention time window is normally the average absolute retention time $\pm$ 3 Standard Deviations. A peak outside the retention time window will not be identified by the computer as a positive match of the analyte of interest.

It is possible for the statistically calculated RT window to be too tight and need to be adjusted based on analyst experience. In these instances method default retention time windows may be

used (e.g., for 8000 series methods a default of 0.03 minutes may be used, and EPA CLP 0.05 minutes is used). The same concept is applied when any peak outside of that window will not be identified by the computer as a positive match.

The calibration verification standard at the beginning of a run may be used to adjust the RT for an analyte. This is essentially re-centering the window but the size of the window remains the same. The RTs are verified when all analytes are within their RT windows and are properly identified.

20.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical or atomic absorption profiles and specific electrode response factors.

20.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

20.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

20.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

20.12.3 The uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

20.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and

the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 +/- 0.5 mg/l.

20.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g. 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

20.13 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

20.13.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the 'Analytical Information Management System (AIMS)' which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes FoxPro-2.6x which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

20.13.1.1 Maintain the Database Integrity: Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

  Note: "Commercial off-the-shelf software in use within the designed application range is considered to be sufficiently validated." *From NELAC 2003 Standard.* However, laboratory specific configurations or modifications are validated prior to use.

- In order to assure accuracy, all data entered or transferred into the LIMS data system goes through a minimum of two levels of review.
- The QA department performs random data audits to ensure the correct information has been reported.
- Changes to reports are documented within the LIMs through audit trails, revision tracking and imposed case narrative comments.
- Analytical data file security is provided through three policies.
  - The first policy forbids unauthorized personnel from using laboratory data acquisition computers.
  - The second policy is the implementation of network passwords and login names that restrict directory access.
  - The third layer is maintained through the LIMS and includes the use of username/password combinations to gain access to the LIMS system, the fact that

all data in the LIMS is associated with the user to added/reviewed the data, and the restriction of review authority of data.

- All software installations will be in accordance with any relevant copyright licensing regulations.
- All software installed on any computer within the laboratory must be approved by the Information Technology Department regional support technician assigned to the laboratory Shrink-wrapped or otherwise sealed OEM software that is directly related to instrument usage does not need approval but the Information Technology department must be notified of the installation.
- Anti-virus software shall be installed on all servers and workstations. The anti-virus software shall be configured to check for virus signature file and program updates on a daily basis and these updates will be pushed to all servers and workstations. The anti-virus software will be configured to clean any virus-infected file if possible, otherwise the file will be deleted. Disks and CDs brought from any outside source that are not OEM software must be scanned for viruses before being accessed.

- **Interlab LIMS Permissions Policy**
  - PURPOSE - The purpose of this policy is to provide a mechanism for maintaining the integrity of information contained in each laboratory's LIMS while providing the necessary access for information sharing to staff at other laboratory facilities.
  - DEFINITIONS - Host Laboratory: The laboratory facility that 'owns' the LIMS system or 'hosts' a project/job.
  - POLICIES

    (a) All permissions for the laboratory's LIMS system must only be granted by a representative of that laboratory.
    - If someone outside of the host lab needs permissions for Project Management or other uses, they must go through the Lab Director or his/her designated representative.
    - Permissions must never be granted without the knowledge of the host laboratory.

    (b) Only laboratory analytical or QA staff from the home laboratory may have edit permissions for laboratory analysis data.

    (c) Any changes made in laboratory's LIMS system:
    - Must be documented and traceable.
    - If made by staff of an affiliate lab, written permission from the home lab to make the changes (email approval is sufficient) is required.
    - No corrections may be made in another laboratories system without their knowledge.

    (d) Data qualifiers in laboratory reports must only be corrected, edited, etc. by the staff at the host laboratory.

    (e) Full analytical data "View" only permissions may be granted to outside Project Management and Sales staff. Search permissions may also be granted so status may be checked.

    (f) All qualifiers must be approved by QA staff before adding to standard reference tables.

    (g) Please contact Corporate QA or IT staff if you have any questions regarding implementation or interpretation of this policy.

20.13.1.2 **Ensure Information Availability:** Protection against loss of information or service through scheduled back-ups, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

- Insured by timely backup procedures on reliable backup media, stable file server network architecture, and UPS protection
- UPS Protection:
  - Each fileserver is protected by an appropriate power protection/backup unit. In the event of a power outage, there is approximately 15-30 minutes of up-time for the servers prior to shutdown. This allows for proper shutdown procedures to be followed with the fileservers.
- File Server Architecture
  - All files are maintained on multiple Windows 2000 or newer servers which are secured physically in the Information Technology server room. Access to these servers is limited to members of the Information Technology staff.
  - All supporting software is maintained for at least 5 years from the last raw data generated using that software. [Length of time is dependent on local regulations or client requirements, e.g., NYS CLP requires 6 years
- System Back-up Overview and Procedures
  - Data from both servers and instrument attached PC's are backed up and purged in compliance with the corporate back-up policy.
  - A Maintenance Plan has been defined to create a daily archive of all data within the LIMS database to a backup location. This backup is initiated automatically by either the database or back-up system.
  - Backup tapes will be stored in compliance with the corporate Data Backup Policy. Backup verifications are carried out in accordance with the corporate Data Backup Policy.
  - Instrument data back-ups are verified on a periodic basis by the QA department when performing electronic data audits. The audit takes place on data that has been moved to a back-up location ensuring that it has been moved.

20.13.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls, and encryption of when electronically transmitting data.

- All servers are located in a secure area of the IT department offices. Access to the servers is limited to IT staff members, Lab Director, Technical Director, the President and Vice President of Operations.
- The reporting portion of the LIMS system requires a project manager to enter their unique password anytime they create a report that displays a signature on it (.PDF).
- If electronic documents are made available outside of the web site, the customer must sign an agreement in advance that states they will not alter the data in any way.

20.13.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The

analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The data review sheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices.*

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

20.13.2.1 All raw data must be retained in the project job folder, computer file, and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

20.13.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. The units "mg/l" and "mg/kg" are the same as "parts per million (ppm)". The units "µg/l" and "µg/kg" are the same as "parts per billion (ppb)." For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%.

- Several environmental methods, such as color, turbidity, conductivity, use very specific, non-concentration units to report results (e.g., NTU, umhos/cm etc).
- Occasionally, the client requests that results be reported in units which take into account the measured flow of water or air during the collection of the sample. Results for wipe sampling may be reported based on the area wiped. When the client provides this information, the calculations can be performed and reported.

20.13.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, final inorganic results are reported to 2 significant figures for values less than 10 and 3 significant figures for values greater than 10 on the final report. Organic results are generally reported to 1 significant figure for values less than 10 and 2 significant figures for values greater than 10 on the final report. The number of significant figures may be adjusted based on client or project requirements.

20.13.2.4 For those methods that do not have an instrument printout, an instrumental output or a calculation spreadsheet upload compatible with the LIMS System, the final results and dilution factors are entered directly into LIMS by the analyst, and the software formats the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

20.13.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is automatically transferred to the network server and, eventually, to a back-up tape file.

20.13.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 13.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Director/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

20.13.4 Review / Verification Procedures

Review procedures are out lined in several laboratory SOPs (e.g. BF-SR-002, "Receipt of Analytical Samples", BF-GP-012, "Technical Data Review", and BF-PM-001, "Project Information Requirements") to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (BF-GP-013, Manual Integration). The general review concepts are discussed below, more specific information can be found in the SOPs.

20.13.4.1 The data review process at ***TestAmerica Buffalo*** starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Project Managers perform review of the chain-of-custody forms and inputted information and approve the

input in LIMs to make the samples available to the laboratory departments for batching and processing.

20.13.4.2 The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add any manual data qualifiers or dilution codes if applicable (see Appendix 7 for list of common data qualifiers). To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Approximately 10% of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range
- Results deviate from historical trends (if history available)

20.13.4.3 Unacceptable analytical results may require reanalysis of the samples. Any unusual or uncharacteristic circumstances are brought to the attention of the Department Manager. The Department Manager may involve the Project Manager, the Technical Director and/or the QA Manager for further investigation depending on the issue. Corrective action is initiated whenever necessary.

20.13.4.4 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

20.13.4.5 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The following are some examples of chemical relationships that are reviewed (if data are available):

- Total Results are ≥ Dissolved results (e.g. metals)
- Total Solids (TS) ≥ TDS or TSS
- TKN ≥ Ammonia
- Total Phosphorus ≥ Orthophosphate
- COD ≥ TOC
- Total cyanide ≥ Amenable Cyanide
- TDS ≥ individual anions

20.13.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report and creates the invoice. (*Also see section 26 on Reporting Results*). When complete, the report is issued to the client.

20.13.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines.

20.13.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

20.13.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

20.13.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

20.13.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters

(calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 20-1.
Example - Demonstration of Capability Documentation

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

DOC Cert. Statement
Revision 7
Dec. 14, 2007

TESTAMERICA LABORATORIES, INC.

TRAINING & DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

Employee: _____ Page _____of_____

Method Number: _____ Date: _____

Parameters or Analytes: _____

Initial Demonstration of Capability: ☐

SOP Number: _____Revision # _____ Date Read_____

Trained By:_____

Date training began:_____ Date training completed:_____

Continued Demonstration of Capability: ☐

SOP Number: _____Revision # _____ Date Read_____

I CERTIFY that I have read and understand the SOP identified above. I have also submitted data associated with the demonstration of capability.

_____ _____
Employee Signature Date

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific Sops are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at this facility, and that the associated information is well organized and available for review by authorized assessors.

John Schove _____ _____
Operations Manager Signature Date

Peggy Gray-Erdmann _____ _____
Quality Assurance Manager Signature Date

Figure 20-2.

Example - New Method / Additional Analyte Checklist

New Method / Additional Analyte Checklist

The following items are **required** to be completed prior to the acceptance of client samples. Fill in any blanks that do not apply with "NA". Provide associated instrument QC when samples or QC samples are analyzed (includes run log).

New Method _____ Added Analytes _____

1_____ Standard Operating Procedure
- Note: For additional analytes, a **ROMD [or whatever an internal communication memo is named in your lab]** can be used to add the analytes, include RL and matrix.
  - _____ Analysis SOP
  - _____ Preparation SOP
  - _____ SOP for any other relevant process
  - _____ Pages from any applicable logbooks (instrument, standards, etc)

2_____Evaluation of Selectivity. As applicable: e.g. Retention Time Window Study, second column confirmation, Interelement correction checks, spectral or fluorescence profiles, etc.

3_____ Initial Calibration Curve (Include Tune verification or similar (e.g. degradation checks) if applicable)

4_____ Method Detection Limit (MDL) Study (summary and raw data)
- _____ Water
- _____ Soil
- _____ Other

5_____ Reporting Limit Verification standard
- Spike a blank matrix at the RL and process through the entire method. MDL study should be able to be used if recovery is good. Note the spike level(s) and recovery(yies)

6_____ Demonstration of Capability (DOC) per analyst (Precision and Accuracy (P&A) verification)
- 4 LCS for each matrix – most acceptance criteria are in the methods. The MDL study may be used if DOC criteria are met.
- Non-Standard methods – 3 x (1 LCS at LOQ-25%, 50%, 75% of the calibration range + Blank) prepared each day. (see NELAC Chpt 5, appendix C.3.3 (b))

7_____ Acceptable PT sample(s) if available

Notes: PT sample required for all new methods
PT sample required for all new analytes under NELAP

Submitted by _____ Date _____

8_____ Certification/Approval from Regulatory Agency where available.

QA Review / Acceptance _____ **Date** _____

SECTION 21

EQUIPMENT (AND CALIBRATIONS)
(NELAC 5.5.5)

21.1 OVERVIEW

TestAmerica purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 21-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

21.2 PREVENTIVE MAINTENANCE

21.2.1 ***TestAmerica Buffalo*** follows a well-defined program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

21.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

21.2.2.1 Calibrations, routine maintenance, and adjustments are part of the analysts' and Department Managers' responsibilities. However, service contracts may be in place for some instruments to cover any major repairs.

21.2.2.2 High purity gases, reagents, and spare parts are kept on hand to minimize repair time and optimize instrument performance.

21.2.3 Table 21-2 summarizes the schedule for routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

21.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all

major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

21.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

21.2.4.2 Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.).

21.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

21.2.5 In addition, the maintenance records contain:

- The identification of the instrument/equipment (instrument's Serial Number and Model Number)
- The date the instrument/equipment was put into use.
- If available, the condition when the instrument was received (e.g. new, used, reconditioned).

21.2.6 If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses (refer to Sections 12 and 13).

21.2.7 In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted using the procedures outlined in Section 8.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

21.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance. Laboratory SOPs BF-GP-001,"Calibration of Autopipettes and Repipetters" and BF-GP-002, "Support Equipment: Maintenance, Record Keeping and Corrective Actions of Analytical Balances, Temperature Control Devises and Reagent Water" provide additional detail on the monitoring and record keeping for support equipment.

21.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

21.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to $\pm$ 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

21.3.3 Thermometers

All reusable thermometers are calibrated on an annual basis with a NIST-traceable thermometer. Disposable glycol thermometers are discarded upon expiration and replaced with newly purchased thermometers. IR thermometers are verified daily and calibrated annually. Digital probes and thermocouples are calibrated quarterly.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer has increments of 0.2 °C, and has a range applicable to all method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the laboratory SOP BF-GP-020, "Thermometer Calibration".

21.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0°C and $\leq$ 6 °C.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

21.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are checked for accuracy at least quarterly. Glass micro-syringes are considered the same as Class A glassware.

The laboratory maintains a sufficient inventory of autopipettors, and dilutors of differing capacities that fulfill all method requirements.

These devices are given unique identification numbers, and the delivery volumes are verified each day of use and calibrated gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

21.3.6 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated monthly (or if not utilized monthly, immediately prior to its usage) by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 10%, the procedure is repeated and if the result is still greater than 10%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

Additional calibration and use information is detailed in laboratory SOP BF-FS-006, "Calibration of Field Meter".

21.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 13).

Note: Instruments are calibrated initially and as needed after that and at least annually.

21.4.1 CALIBRATION STANDARDS

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. However, the general procedures are described below.

21.4.1.1 For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods. If a reference or mandated method does not specify the number of calibration standards, the minimum number is three, not including blanks or a zero standard. All of the standard solutions are prepared using Class A volumetric glassware, calibrated pipettes, and/or microsyringes and appropriate laboratory quality solvents and stock standards.

21.4.1.2 Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to NIST whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. A standard preparation log is maintained for each department, containing concentration, date of receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.

21.4.1.3 The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

21.4.1.4 The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The lowest calibration standard must be at or below the reporting limit. The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

21.4.1.5 Given the number of target compounds addressed by some of the organic methods, it may be necessary to prepare several sets of calibration standards, each set consisting of the appropriate number of solutions at different concentrations. The initial calibration will then involve the analysis of each of these sets of the appropriate number of standards.

21.4.1.6 All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be

considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

21.4.2 CALIBRATION FOR ORGANIC METHODS (GC, HPLC, GC/MS)

21.4.2.1 Many of the organic analytical methods utilize an internal standard calibration (GCMS and some GC). Because of the complex nature of the multipeak chromatograms produced by the method, some instruments necessitate the use of external standard calibration (most GC and HPLC). Surrogate compounds are included in the calibration processes for all appropriate organic analyses. For more details on the calibration types listed below, refer to SOP No. CA-Q-S-005, Calibration Curves.

21.4.2.2 Once the operating parameters have been established according to the method, each instrument is calibrated for the appropriate method. The analyst prepares five or more standard solutions at various concentrations containing all of the analytes of interest, internal standards, and surrogates that are appropriate for the method. Note: There are several EPA methods that have different requirements and are exceptions (e.g. EPA 625) where a minimum of 3 calibration standards are prepared and analyzed.

21.4.2.3 The standard solutions are introduced into the instrument in the same manner as samples are; whether by direct injection, by headspace analysis, or by purge and trap. The calibration factor (CF) for methods that use external standards, and the response factor (RF) for methods that use internal standards are calculated for the five standards.

- External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the response to the amount of analyte in the calibration standard is defined as the Calibration factor (CF).

- Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. The use of specific internal standards is available in the method SOP.

Whichever internal standards are employed, the analyst needs to demonstrate that the measurement of the internal standard is not affected by method analytes and surrogates or by matrix interferences. In general, internal standard calibration is not as useful for GC and HPLC

methods with non-MS detectors because of the inability to chromatographically resolve many internal standards from the target compounds. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

When preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes will vary. The internal standard solution will contain one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution (e.g., not all internal standards need to be at the same concentration in this solution). The mass of each internal standard added to each sample extract immediately prior to injection into the instrument or to each sample prior to purging must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts should be such that minimal dilution of the extract occurs (e.g., 10 uL of solution added to a 1 mL final extract results in only a negligible 1% change in the final extract volume which can be ignored in the calculations).

An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard should produce an instrument response (e.g., area counts) that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This should result in a minimum response factor of approximately 0.01 for the least responsive target compound. Refer to SOP No. CA-Q-S-005, Calibration Curves, for specific calculations.

21.4.2.4 Policies regarding the use of calibration standard results for creating the calibration curve are as follows:

- A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting level must be elevated to be the lowest calibration standard used for calibration.

- The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.

- Mid-calibration standards may not be excluded unless an obvious reason is found, i.e., cracked vial, incorrectly made, etc. The failed standard should be re-run immediately and inserted into the initial calibration. If not useful, recalibration is required.

21.4.2.5 Percent RSD Corrective Action

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

21.4.2.5.1 The first step is generally to check the instrument operating conditions. This option will apply in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance, changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.

21.4.2.5.2 If the RSD for any analyte is greater than the applicable acceptance criteria in the applicable analytical method SOP, the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

21.4.2.5.3 A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

Note: When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the method quantitation limit is at least as low as the regulatory limit or action level.

21.4.2.6 Alternatively, the least squares regression may be used to determine linearity. A five point line must result in a correlation coefficient (r) of 0.990 or better using the least squares method to be considered acceptable. In many cases it may be preferred that the curves be forced through zero (not to be confused with including the origin as an additional data point, which is not allowed). **Note:** EPA method 8000B does not allow forcing through zero however the agency has revaluated this position and has since changed this stance to allow forcing through zero. In addition, from EPA Method 8000C: "However, the use of a linear regression or forcing the regression through zero may NOT be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards.").

21.4.2.7 Instead of a linear curve model (either Average RF or least squares regression), a second order curve (Quadratic) may be used (and preferred) as long as it contains at least six data points. As a rule of thumb, if there is a consistent trend

in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (COD or r^2) for the quadratic curve must be at least 0.99 for it to be considered acceptable. For more details on the calculations see Calibration Curve SOP CA-Q-S-005. Some limitations on the use of Quadratic Curve fits:

21.4.2.7.1 Care MUST be exercised to assure that the results from this equation are real, positive, and fit the range of the initial calibration.

21.4.2.7.2 They **may not** be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).

21.4.2.7.3 They **may not** be used to compensate for detector saturation. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve and try a 1st order fit or average RF.

21.4.3 Calibration for Inorganic Analyses

EPA Method 7000 from EPA SW-846 is a general introduction to the quality control requirements for metals analysis. For inorganic methods, quality control measures set out in the individual methods and in the *Standard Methods for the Examination of Water and Wastewater* (20th Edition) may also be included. Standard Operating Procedures for the analysis and the quality control documentation measures are maintained on the laboratory intranet (BufNet).

In general, inorganic instrumentation is calibrated with external standards. Some exceptions would be Inductively Coupled Plasma (ICP) and Inductively Coupled Plasma Mass Spec (ICPMS). These analyses may use an internal standard to compensate for viscosity or other matrix effects. While the calibration procedures are much the same for inorganics as they are for organics, CF's or RF's are not used. The calibration model in 21.4.2.6 is generally used for most methods, however in some instances the model from section 21.4.2.7 may be used. A correlation coefficient (r) of 0.995 or greater must be used to accept a calibration curve generated for an inorganic procedure. Correlation coefficients are determined by hand-held scientific calculators, excel spreadsheets or by computer programs and documented as part of the calibration raw data. Coefficients of calibration curves used for quantitation must be documented as part of the raw data. Curves are not allowed to be stored in calculator memories and must be written on the raw data for the purposes of data validation.

21.4.3.1 "Calibrations" for titrimetric analyses are performed by standardizing the titrants against a primary standard solution. See specific methods in *Standard Methods for the Examination of Water and Wastewater* (20th Edition) for more information.

21.4.3.2 Spreadsheets that are used for general chemistry calculations must have all cells containing calculations locked to prevent accidental changes to the calculations.

21.4.4 Calibration Verification

The calibration relationship established during the initial calibration must be verified at periodic intervals as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration, and is not appropriate nor permitted in SW-846 chromatographic procedures for trace environmental analyses.

21.4.4.1 Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.

21.4.4.2 A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements (see specific SOPs). Most Inorganic methods require the CCV to be analyzed after ever 10 samples.

21.4.4.3 The acceptance limits for calibration verifications can be found in each method SOP. As a rule of thumb: GCMS $\pm$ 20%, GC and HPLC $\pm$ 15%, Inorganics: $\pm$ 10 or 15%. Actual methods may have wider or tighter limits; see the method SOP for specifics.

21.4.4.4 If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF, RF or % drift values from the initial calibration to quantitate sample results.

21.4.4.5 If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new initial instrument calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

21.4.4.5.1 When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

21.4.4.5.2 When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

21.4.4.6 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

The Percent Difference is calculated as follows:

$$\% \text{ Difference} = \frac{(CF(v) \text{ or } RF(v)) - (\text{Avg. CF or RF})}{(\text{Avg. CF or RF})} \times 100$$

Where: CF(v) or RF(v) = CF or RF from verification standard
Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

$$\% \text{ Drift} = \frac{\text{Result} - \text{True Value}}{\text{True Value}} \times 100$$

The Percent Recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100$$

21.4.4.7 Verification of a Non-Linear Calibration

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations described in 21.4.4.6 above.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard,

then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

21.5 POLICY ON TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

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 purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it will not be reported as a TIC. If the compound is reported on the same form as true TICs, it must be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. See laboratory SOP's BF-MB-005 and BF-MV-007 for guidelines for making tentative identifications

Note:
For general reporting if TICs are requested, the ten (10), largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard will be termed "Tentatively Identified Compounds" (TICs). More or fewer TICs may be identified based on client requirements.

21.5.1 TIC Reporting Limits

In general Reporting limits cannot be specified because of the unknown nature of the TIC. Any reporting limit that is reported can only be evaluated as an estimate as the quantitation is based on the assumption that the TIC responds exactly as the IS responds which is most likely not the case. In general, it is not recommended to set a Reporting limit at too low of a concentration as it gives a false impression.

TICs that meet the required identification criteria at 10% area of the IS: The RL would be 10% of the concentration of the internal standard used for quantitation. (e.g. 2.5 ug/L for 8260B, 4.0 ug/L for 8270C). In general, if the 10% area criterion is not met, the TIC RLs should be set at a level approximately 5x the level of the poorest performer in the analysis.

If a compound meets the TIC criteria, the reporting limit will reflect the ratio between the TIC and the IS or 5x the level of the poorest performer whichever is lower.

21.6 POLICY ON GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

21.6.1 The concentration of the BFB or DFTPP must be at or below the concentrations that are referenced in the analytical methods. Part of the purpose of the tune is to demonstrate sensitivity and analyzing solutions at higher concentrations does not support this purpose. Tune failures may be due to saturation and a lower BFB/DFTPP concentration may be warranted.

21.6.2 Tune evaluations usually utilize the "Autofind" function and are set up to look at the apex +/- 1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction cannot include any part of the target peak.

21.6.3 Other Options or if Auto Tune Fails:

21.6.3.1 Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. In this case, manually identify and average the apex peak +/- 1 scan and background correct as in 21.6.2 above. This is consistent with EPA 8260 and 8270.

21.6.3.2 Or the scan across the peak at one half peak height may be averaged and background corrected. This is consistent with Standard Methods 6200, EPA 624 and EPA 625.

21.6.3.3 Adjustments such as adjustments to the repeller and ion focus lenses, adjusting the EM Voltage, etc. may be made prior to tune verification as long as all of the subsequent injections in the 12 hour tune cycle are analyzed under the same MS tune settings and it is documented in the run sequence log and/or maintenance log that an adjustment was made. Excessive adjusting (more than 2 tries) without clear documentation is not allowed. Necessary maintenance is performed and documented in instrument log.

21.6.3.4 A single scan at the Apex (only) may also be used for the evaluation of the tune. For SW 846 and EPA 600 series methods, background correction is still required.

21.6.3.5 Cleaning the source or other maintenance may be performed and then follow steps for tune evaluation above. Note: If significant maintenance was performed, see methods 8000B or 8000C then the instrument may require recalibration prior to proceeding.

21.6.4 Tune evaluation printouts must include the chromatogram and spectra as well as the Tune evaluation information. In addition, the verifications must be sent directly to the printer or pdf file (no screen prints for DFTPP or BFB tunes). This ability should be built into the instrument software.

21.6.5 Since the limits are expressed in whole percentages, the results may be rounded to whole percentage before comparing to criteria when assessing the tune verification against the tune requirements. However, the comparison to the criteria is usually done automatically by the software and if the printout says "Fail" then there would have to be documentation of the hand calculation on the raw data and comparison to the criteria if the lab intends to still accept the tune. In most cases the analyst is better off performing an adjustment and rerunning the tune standard.

21.6.6 All MS tune settings must remain constant between running the tune check and all other samples. It is recommended that a separate tune method not be used, however a separate method may be used as long as the MS conditions between the methods are the same as the sample analysis method and tracked so any changes that are made to the analysis method are also made to the tune method.

Table 21-1.

Laboratory Equipment and Instrumentation – TestAmerica Buffalo

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS	Hewlett Packard	5973	US44621446	2005	new
	Hewlett Packard	5973	US52420646	2005	new
	Hewlett Packard	5973	US41720721	2004	new
	Hewlett Packard	5973	US35120354	2004	new
	Hewlett Packard	5973	US41720707	2004	new
	Hewlett Packard	5973	US10241053	2003	new
	Hewlett Packard	5973	US30965634	2003	new
	Hewlett Packard	5973	US03965692	2003	new
	Hewlett Packard	5973	US05060076	2001	new
	Hewlett Packard	5973	US05060084	2001	new
	Hewlett Packard	5973	US03950346	2001	new
	Hewlett Packard	5973	US82321636	2001	new
GC	Hewlett Packard	6890 dual uECD	CN10520009	2005	new
	Hewlett Packard	6890 dual uECD	CN10520010	2005	new
	Hewlett Packard	6890 dual uECD	CN10448015	2005	new
	Hewlett Packard	5890II dual ECD	3336A53126	1994	new
	Hewlett Packard	5890II dual ECD	3336A63465	1994	new
	Hewlett Packard	5890II dual ECD	3336A53464	1994	new
	Hewlett Packard	5890II dual ECD	3336A53463	1994	new
	Hewlett Packard	5890II dual ECD	3336A54409	1994	new
	Hewlett Packard	5890II dual ECD	3336A54408	1994	new
	Hewlett Packard	5890II FID/FID	3115A34892	1994	new
	Hewlett Packard	5890II PID/FID	3336A60622	1994	new
	Hewlett Packard	5890II Hall/PID	3235A54089	1994	new
	Hewlett Packard	5890II PID/FID	3336A53465	1994	new
	Hewlett Packard	5890II dual FID	3336A53727	1994	new
	Hewlett Packard	5890II dual ECD	3310A47661	1993	new
	Hewlett Packard	5890II dual ECD	3336A53325	1993	new
	Hewlett Packard	5890II PID/FID	3133A37157	1993	new
	Hewlett Packard	5890II dual ECD	3203A42206	1992	new
	Hewlett Packard	5890II dual FID	3019A28433	1991	new
	Hewlett Packard	5890II Hall/PID	3121A35782	1990	new
LC	Hewlett Packard	1100 HPLC Fluor./DAD	DE92001578	2000	new
Metals	Perkin Elmer	Elan 9000 ICP-MS	P0230202	2002	new
	Leeman	PS200 II	HG9045	2000	new
	Leeman	PS200 II	HG0033	2000	new
	Thermo Jarrell Ash	ICP61E Trace	334490	1995	new
	Thermo Jarrell Ash	ICP61E Trace	382590	1995	new

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Water Quality	Konelab	20XT	E3719731	2005	new
	Thermo ECA	1200 TOX/AOX	2004.901	2004	new
	Dionex Ion Chromatograph	DX-120	20126	2004	new
	Konelab	20	S5019455	2004	new
	Glastron	CN Midi-distillation	2502	2003	new
	Glastron	Phenol Midi-distillation	2069	2003	new
	Glastron	Phenol Midi-distillation	2053	2003	new
	Labtronics	BOD Magic - Autoanalyzer	270H3XB531	2004	new
	Labtronics	BOD Magic - Autoanalyzer	270J2XB669	2003	new
	ManTech	PC Titrator	MS-OK2-607	2003	new
	HACH Spectrophotometer	DR/2500	30200004886	2003	new
	Dionex Ion Chromatograph	DX-120	2060196	2002	new
	OI Carbon Analyzer	1010 #2	H014710903	2000	new
	Spectronic Genesis	4001/4	3SGC199091	2000	new
	Lachat Quickchem	8000 Autoanalyzer	A83000-1527	2000	new
	OI Carbon Analyzer	1010 #1	H92170411	1999	new
	Lachat Quickchem	8000 Autoanalyzer	A83000-1439	1999	new
	Dionex Ion Chromatograph	DX-120	99010157	1999	new
	Orion	Ion Meter 230A	2229	1999	new
	VWR Ion	Meter 2100	1063	1997	new
	YSI	Oxygen Meter 57	93J09826	1995	new
	Lab-Line	Hi-Lo BOD chamber	391-010	1994	new
	Fischer	Accumet Ion Meter 925	860	1991	new
Sample Preparation	J2 ACCUPREP GPC		03F-10723	2003	new
	TurboVap II	TurboVap II	TV9445N5816	1996	new
	TurboVap II	TurboVap II	TV9427N4133	1996	new
	TurboVap II	TurboVap II	TV944N5819	1996	new
	TurboVap II	TurboVap II	TV944N5820	1996	new
	TurboVap II	TurboVap II	TV0024N9623	2000	new
	TurboVap II	TurboVap II	TV0022N9604	2000	new

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Sample Preparation (Continued)	TurboVap II	TurboVap II	TV0312N11592	2003	new
	TurboVap II	TurboVap II	TV0312N11591	2003	new
	Organomation	Rot-X-Tractor	16902	1999	new
	Organomation	Rot-X-Tractor	16907	1999	new
	Organomation	Rot-X-Tractor	16913	1999	new
	Heat Systems Sonicator	#XL-2020	G1647/C5659	1994	new
	Heat Systems Sonicator	#XL-2020	G2665/C5674	1994	new
	Heat Systems Sonicator	#XL-2020	G2620/C5660	1994	new
	Heat Systems Sonicator	#XL-2020	G2245/C6328	1995	new
	Heat Systems Sonicator	#XL-2020	G2621/C6733	1995	new
	Heat Systems Sonicator	#XL-2020	G2713/C6732	1995	new
	Heat Systems Sonicator	#XL-2020	G1643/C6837	1995	new
	Heat Systems Sonicator	#XL-2020	G2742/C6842	1995	new

Table 21-2.

Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check tubing for wear	Daily
	Fill rinse tank with 10% HCl	Daily
	Change dryer tube	As Needed
	Fill reductant bottle with 10% Stannous Chloride	Daily
ICP & ICP/MS	Check pump tubing	Daily
	Check liquid argon supply	Daily
	Check fluid level in waste container	Daily
	Check re-circulator levels	Monthly
	Clean or replace filters	As required
	Check torch	Daily
	Check sample spray chamber for debris	Monthly
	Clean and align nebulizer	Monthly
	Change pump oil	Monthly
	Change Cones	As required
	Change printer cartridge	As required
	Replace pump tubing	As required
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Precision check/alignment of flow cell	As required
	Wavelength verification check	Annually
Auto Analyzers	Clean sampler	Daily
	Check all tubing	Daily
	Clean inside of colorimeter	Daily
	Clean pump well and pump rollers	Quarterly
	Clean wash fluid receptacle	Weekly
	Oil rollers/chains/side rails	Weekly
	Clean optics and cells	Quarterly
Agilent GC/MS	Pump oil-level check	Monthly
	Pump oil changing	Annually
	Analyzer bake-out	As required
	Analyzer cleaning	As required
	Resolution adjustment	As required
	COMPUTER SYSTEM AND PRINTER:	
	Air filter cleaning	As required
	Change data system air filter	As required
	Printer head carriage lubrication	As required
	Paper sprocket cleaning	As required
	Drive belt lubrication	As required

Instrument	Procedure	Frequency
Gas Chromatograph	Compare standard response to previous day or since last initial calibration	Daily
	Check carrier gas flow rate in column	Daily via use of known compound retention
	Check temp. of detector, inlet, column oven	Daily
	Septum replacement	As required
	Glass wool replacement	As required
	Check system for gas leaks with SNOOP	W/cylinder change as required
	Check for loose/frayed power wires and insulation	As Required
	Bake injector/column	As Required
	Change/remove sections of guard column	As Required
	Replace connectors/liners	As Required
	Change/replace column(s)	As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63)	Semi-annually
	Detector cleaning	As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings	As required
	Clean lamp window	As required
HPLC	Change guard columns	As required
	Change lamps	As required
	Change pump seals	Semi-annually or as required
	Replace tubing	As required
	Change fuses in power supply	As required
	Filter all samples and solvents	Daily
	Change autosampler rotor/stator	As required
Vacuum Pumps/ Air Compressor	Drained	Weekly
	Belts checked	Monthly
	Lubricated	Semi-annually
Centrifuge	Check brushes and bearings	Every 6 months or as needed

Table 21-3.

Periodic Calibration

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Analytical Balance	Accuracy determined using "S" NIST traceable weights. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by A2LA accredited person annually.	Daily, when used Annual	± 0.2%	Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service.
Top Loading Balance	Accuracy determined using "S" NIST traceable. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by A2LA accredited person annually.	Daily, when used Annual	± 0.5%	Clean. Replace.
NIST Certified Weights	Accuracy determined by accredited weights and measurement laboratory.	1 year	As per certificate.	Replace.
NIST-Traceable Thermometer	Accuracy determined by accredited measurement laboratory.	5 years	As per certificate.	Replace.
Thermometer	Against NIST-traceable thermometer	Yearly at appropriate temperature range for intended use	± 1.2°C	Replace
Minimum-Maximum Thermometers	Against NIST-traceable thermometer	Yearly	± 1.5°C	Replace
InfraRed Temperature Guns	Against NIST-traceable thermometer Accuracy determined by accredited measurement laboratory.	Daily at appropriate temperature range for intended use. Annual	± 1.5°C	Repair/replace
Dial-type Thermometers	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 1.5°C	Replace

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Refrigerator	Temperature checked using NIST-traceable thermometer.	Daily. If out of range, check again in two hours.	0-6°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Freezer	Temperature checked using NIST-traceable thermometer	Daily. If out of range, check again in two hours.	(-10)-(-20)°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Oven	Temperature checked using NIST-traceable thermometer.	When in use.	104 ± 1°C (drying) 180 ± 2°C (TDS)	Adjust. Replace.
Water Bath	Temperature checked using NIST-traceable thermometer.	When in use.	± 2°C	Adjust. Replace.
Volumetric Dispensing Devices (Eppendorf ® pipette, automatic dilutor or dispensing devices)	One delivery by weight. Using DI water or solvent of use, dispense into tared vessel. Record weight with device ID number. Calibrate using 4 replicate gravimetric measurements	Each day of use Quarterly	± 2% Calculate accuracy by dividing weight by stated volume times 100 for percent.	Adjust. Replace.
Glass Microliter Syringes	None	Accuracy must be initially demonstrated if syringe was not received with a certificate attesting to established accuracy.	± 1%	Not applicable.
Deionized Water	Check in-line conductivity meter on system with conductivity meter in Inorganics Department.	Daily	<1.0 µmho at 25°C	Record on log. Report discrepancies to QA Manager, Operations Manager or Technical Director.

SECTION 22

MEASUREMENT TRACEABILITY
(NELAC 5.5.6)

22.1 OVERVIEW

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. The following definitions are provided by the American Association for Laboratory Accreditation (A2LA):

"Traceability is the property of a measurement result whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, each step in the chain having stated uncertainties." There are six essential elements:

- An unbroken chain of comparison
- A calculated measurement uncertainty for each step in the chain to allow for an overall uncertainty calculation
- Documentation of each step in each calibration report
- All steps in the chain are performed by individuals with evidence of technical competence and accredited by a recognized accreditation body
- Reference to International Standard (SI) units
- Recalibration at appropriate intervals to preserve traceability

Calibration is defined as "determining and documenting the deviation of the indication of a measuring instrument (or the stated value of a material measure) from the conventional 'true' value of the measurand."

Uncertainty is defined as "a parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measurand." Measurement of Uncertainty is discussed is Section 20 of this QA Manual.

22.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), (APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

The calibration report or certificate submitted to ***TestAmerica Buffalo*** contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

22.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA or NVLAP with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. (Refer to Section 9 for additional information on purchasing). The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for

use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to Table 9-1 in Section 9 for general storage requirements and Table 22-1 for additional storage information. Method specific information may also be found in the laboratory method SOPs in the "Standards and Reagents" sections. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

22.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to SOP No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained by each department in bound or electronic folders. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer laboratory SOP BF-GP-019, "Standard Traceability and Preparation" and also to the method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

22.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory department's chemical history log and are assigned a unique identification number. Preparation of working standards or reagents prepared from the stock is documented in the laboratory Department's Standard Preparation Log. The following information is typically recorded:

- Standard ID
- Description of Standard
- Department
- Preparer's name

- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment section

Records are maintained for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

22.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date
- Standard ID
- Special Health/Safety warnings if applicable

22.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods; and 3) according to Table 22-1.

Table 22-1.

Standard Sources and Preparation

Instrument	Source	How Received	Stock Storage	Preparation	Intermediate & Working Standard Storage	Frequency
ICP	SPEX; Environmental Express; CPI	1000 ppm Solutions	Room Temperature	Working standards from stock	Room Temperature	Daily
GC	Supelco; Restek; Ultra	Solutions	Freezer (-10°C)	Working standards from stock	Refrigerate	Monthly
TOX	Chemservice; ERA	Solutions	Refrigerate	Working standards from stock	Refrigerate	Monthly
TOC	Ricca; ERA	Solutions	Refrigerate	As received	Refrigerate	N/A
Volatile Organics	Restek; Supelco; Ultra	Ampoule/ Solutions	Freezer (-10°C)	Working standards from stock	Refrigerate	Monthly; Gas, weekly
Semi-Volatile Organics	Restek; Supelco; Ultra	Ampoule/ Solutions	Refrigerate or Room temp.	Working standards from stock	Refrigerate	Monthly
Infrared Spec-trophotometry	Hach; Accustandard	Pure Reagent	Room Temperature	Working standards from stock	Refrigerate	Weekly
Ion Chromatography	Ultra; Accustandard	Solutions	Refrigerate	Working standards from stock	Refrigerate	Monthly
Lachat; Konelab	ERA; Ricca;Accustandard; Ultra	Solutions	Refrigerate	Working standards from stock	Refrigerate	Weekly, monthly
BOD Magic	Ricca, ERA	Solution	Refrigerate	As received	Refrigerate	Daily

SECTION 23.0

SAMPLING
(NELAC 5.5.7)

23.1 OVERVIEW

TestAmerica Buffalo provides sampling services. Sampling procedures are described in the following SOPs:

BF-FS-001	Chain of Custody Documentation
BF-FS-002	Sample Packaging and Shipment Off-Site
BF-FS-003	Groundwater Sampling Field Data Collection
BF-FS-004	Equipment Decontamination
BF-FS-005	Groundwater/Surface Water Sampling
BF-FS-006	Calibration of Field Meter
BF-FS-007	Low Flow Sampling Procedures
BF-FS-008	Surface and Subsurface Soil/Sediment Sampling

23.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

23.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

23.2.2 Preparing Container Orders

When new containers arrive at the laboratory, the date of receipt is recorded on the packing list received with them for retained documentation. Upon request or based on scheduled monitoring events, the containers are then sent to clients for use in collecting samples. The

shipping date, type and number of containers are maintained on file by the lab. Shipping personnel insure that container stock is rotated so that "first in" is "first out." When a client requests containers, a project management representative creates a container request in LIMS; it is then stored permanently in LIMS with a unique container order number. Copies of the container request are printed by the bottle kit preparation department. One copy goes to the client with the containers; one copy is filed in the bottle kit preparation department. For additional detail refer to laboratory SOPs BF-SR-001, "Cooler Shipping – Bottle Kits and Samples" and BF-PM-003, "Bottle Order Set-up".

The laboratory also provides EnCore, TerraCore or other soil sampling devices when requested.

If containers are provided directly to the client from the manufacturer or from other sources, the laboratory will not be responsible for any of the above records.

23.3 FIELD QUALITY CONTROL (QC)

Common field quality control samples are defined in the following paragraphs. The frequency of field quality control samples should be specified in the site specific Quality Assurance Project Plan (QAPP) or by the client. TestAmerica provides trip blanks for VOC analysis with the sample containers for all volatile organic analyses. Blanks generated in the field will be analyzed along with the field samples (exception soil samples where the blank is aqueous).

23.3.1 Equipment Blank / Rinsate Blank - The equipment blank, sometimes referred to as a rinsate blank, is a sample of the water used to decontaminate sampling equipment. The source water should be as free of target analytes as possible. An aliquot of this water is poured over or through the sample collection device after decontamination, collected in a sample container, preserved with appropriate reagents, and returned to the laboratory. This serves as a check on sampling device cleanliness, and will also be affected by the site and sample handling conditions evaluated by the other types of blanks. The sampling time for the equipment blank should begin when the equipment is rinsed and the water is collected.

23.3.2 Field Blank - The field blank is water that is as free of target analytes as possible and from the same source as the equipment blank. The water is poured into a sampling container at the sampling site, preserved with the appropriate reagents, and returned to the laboratory. This serves as a check on reagent and environmental contamination. The sampling time for the field blank should be when the blank is prepared in the field.

23.3.3 Trip Blank - The trip blank pertains to volatile analysis only. This serves as a check on sample contamination originating from sample transport, sample container contamination, shipping and storage, or from certain site conditions. Trip blanks are often referred to as travel blanks. They are prepared using pre-cleaned sample containers. They are filled with organic-free water (the source of the organic free water is the same source of water used to prepare volatile standards, method blanks, LCS and sample dilutions), sealed and taken into the field with the empty containers which will be used for sampling. The recommended frequency is one trip blank per cooler (in duplicate or triplicate), per volatiles method. Unless otherwise specified, the sampling time for the trip blank is the time of receipt at the laboratory (When the "Trip" ends).

23.3.4 Field Duplicates - Field duplicates are replicate samples collected from the same sampling point or location during a field collection event. This control sample is used to demonstrate the ability of both the sampling and analytical process to generate data of acceptable precision.

23.4 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g. 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time for time critical parameters ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is. These programs will be addressed on a case-by-case basis.

23.4.1 Semi-Volatile - Holding times for sample preparation for semi-volatile organics are measured from the sampling date until the day solvent contacts the sample. Holding times for analysis are measured from the date of initiation of extraction to the date/time of injection into the gas chromatograph.

23.4.2 Volatiles - Holding times for volatile organics are measured from the date (and time where applicable) of sampling to the date and time of injection into the gas chromatograph. The time of initiation of purging is considered the injection time, but data systems record the start of the chromatographic run rather than the start of purging. Hence, if a sample is so near expiration that the start-of-purging time rather than the chromatographic run time is needed to document the integrity of the sample; the analyst must observe and record the start-of-purging time in the instrument log. Medium-level extractions, e.g. for high level soils, must be completed in time to allow for analysis to be initiated within the maximum allowable holding time.

23.4.3 Inorganics - For inorganic and metals analysis, the preparation/digestion/distillation must be started within the maximum holding time as measured from the sampling date (and time where applicable).

23.5 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times (refer to Tables 23-1 to 23-7) or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

23.6 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to

take a representative subsample or aliquot of the sample provided for analysis. In that regard the following guidelines apply to analysts:

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

The following information provides general guidance for homogenization and subsampling. For laboratory specific procedures refer to SOP BF-GP-005, "Sample Homogenization and Subsampling".

23.6.1 For water samples, before taking each aliquot for analysis, invert the sample container end-over-end three times and immediately pour off the aliquot. Especially when suspended solids are present, adequate mixing of the sample is extremely important.

23.6.2 For solid samples, when volatile organics are not requested, if the solid can be mixed, stir before removing the aliquot. Mix more than is needed for the analysis to be performed (e.g. if 30 g are needed, mix 50-100 g, if 1 g is needed, mix 20 g, etc...).

- If the solid cannot be easily mixed: After thoroughly mixing the sample within the sample container or, for non-organic methods, the sample can be transferred to a clean glass container, Teflon boat or other suitable container for manual mixing, a sub-sample from various quadrants and depths of the sample are taken to acquire the required sample weight.
- For soil samples, avoid debris in the subsample aliquot as much as possible (e.g. gravel, sticks, roots and grass); note this information in the sample preparation record.
- If the solid is extremely heterogeneous, and the client has given no instructions, utilize the following technique: separate the like materials into groups on a clean surface and take portions of masses from each group, proportional to their contribution to the original sample, to make a composite. Record in detail exactly how the composite was created. For very unusual samples, consult with the Technical Director or Department Manager.

23.6.3 For solid samples, when volatile organics analysis is requested, the sample should be manipulated as little as possible. In most cases, the sample will arrive already preserved or in an EnCore™ sampler of the correct mass (requiring quick preservation of the entire amount). If the client requests volatiles on a solid sample which has been collected in a jar and is in a common container from which aliquots for other test methods must be taken, sample management personnel should deliver the container to the volatiles department for preparing a proper aliquot prior to any other aliquots being taken out.

23.6.4 For multiphasic samples, the client should instruct the laboratory as to the intent of the testing and how to handle the sample. If the entire sample is to be accounted for, and the phases do not mix easily with inversion/stirring, such that a representative aliquot can be taken, the analyst should record the percent by volume of each phase. The analysis must be conducted on each phase separately; the final results are combined mathematically, weighting the individual phase results by volume. One exception to this procedure is the situation addressed in the TCLP and SPLP methods for wastes containing free liquids. However, if the leachate and final filtrate are not miscible, it is necessary to combine mathematically the concentrations of the two (or more) solutions by volume.

Tables 23-1 to 23-7 detail holding times, preservation and container requirements, and sample volumes for SDWA and NPDES methods. The sample volumes are intended to be a minimal amount to perform the method, the containers that are used may be of larger size. TestAmerica Buffalo does not currently retain capability for all listed methods or parameters however the additional details are included for reference purposes. ***Please note**: the holding times are program specific and different programs may have different holding times for equivalent methods (e.g., there are difference in Holding times for many Organic analytes between SDWA and NPDES. RCRA methods may also be different.)*

Table 23-1.
Holding Times, Preservation and Container Requirements: Drinking Water (SDWA)

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.[23]	Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Asbestos	Plastic/Glass	4°C	None	48 hours[5]	1 L
Coliforms (Total and Fecal)	Plastic/Glass[20]	10°C	$Na_2S_2O_3$	30 hours[21]	120 mL
Cyanide	Plastic/Glass	4°C	NaOH to pH >12 Ascorbic acid[9] or Sodium arsenite[9]	14 days	500 mL
Fluoride	Plastic/Glass	None	None	None	250 mL
Heterotrophic Plate Count	Plastic/Glass[20]	10°C	$Na_2S_2O_3$	8 hours (24 hours[22])	120 mL
Mercury	Plastic/Glass	None	HNO_3 to pH<2	28 days	250 mL
Metals[4]	Plastic/Glass	None	HNO_3 to pH<2[24]	6 months	250 mL
Nitrate	Plastic/Glass	4°C	None	48 hours[6]	250 mL
Nitrate-Nitrite	Plastic/Glass	None	H_2SO_4 to pH<2	28 days	250 mL
Nitrite	Plastic/Glass	4°C	None	48 hours	250 mL
THMs Only	Glass[8]	4°C	$Na_2S_2O_3$[9] HCl to pH <2 may also be used	14 days	3 X 40 mL
Volatile Organic Compounds	Glass[8]	4°C	HCl to pH <2 $Na_2S_2O_3$ or Ascorbic acid[9]	14 days	3 X 40 mL
EDB, DBCP, 1,2,3-TCP (EPA 504.1)	Glass[8]	4°C	$Na_2S_2O_3$	14 days	3 X 40 mL
Organochlorine Pesticides/PCBs (EPA 505)[10]	Glass[8]	4°C	$Na_2S_2O_3$	14 days[11]	3 X 40 mL
Nitrogen and Phos. Pesticides (EPA 507)	Glass-Amber[8]	4°C	$Na_2S_2O_3$	14 days[12]	1 L
Total PCBs (EPA 508A)	Glass-Amber[8]	4°C	None	14 days[13]	1 L
Pesticides and PCBs (EPA 508.1)[14]	Glass-Amber[8]	4°C	HCl to pH <2 $Na_2S_2O_3$[9]	14 days[13]	1 L

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.[23]	Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Chlorinated Acids (EPA 515.1)	Glass-Amber[8]	4°C	$Na_2S_2O_3$	14 days[12]	1 L
Semivolatiles (EPA 525.2)	Glass-Amber[8]	4°C	HCl to pH <2 $Na_2S_2O_3$[9]	14 days[13]	1 L
N-Methylcarbamoyloxamines and N-Methcarbamates (EPA 531.1)	Glass[8]	4°C	$Na_2S_2O_3$, Monochloroacetic Acid buffer to pH<3	28 days	3 X 60 mL
Glyphosate (EPA 547)	Glass[8]	4°C	$Na_2S_2O_3$	14 days	3 X 60 mL
Endothall (EPA 548)	$Na_2S_2O_3$	4°C	None	7 days[15]	1 L
Diquat/Parquat (EPA 549.1)	Glass-Amber[8] (Silanized or PVC amber)	4°C	H_2SO_4 to PH <2 $Na_2S_2O_3$[9]	7 days[16]	1 L
Chlorinated Disinfection Byproducts, Chlorinated Solvents, and Halogenated Pesticides/Herbicides (EPA 551)	Glass[8]	4°C	Phosphate Buffer and Ammonium Chloride[19]	14 days[17]	3 X 60 mL
Haloacetic Acids (EPA 552.1)	Glass-Amber[8]	4°C	Ammonium Chloride	28 days[18]	250 mL

Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
4. All metals except Hg.
5. Instructions for containers, preservation procedures and holding times as specified in Method 100.2 must be adhered to for all compliance analysis including those conducted with Method 100.1.
6. If the sample is chlorinated, the holding time for an un-acidified sample kept at 4°C is extended to 14 days.
7. Nitrate-Nitrite refers to a measurement of total nitrite.
8. With Teflon lined septum.
9. If chlorinated add reagent prior to acidification (for Cyanide, add before NaOH).

Key to Table

10. Heptaclor has a 7 day hold time
11. 14 days until extraction. 24 hours after extraction.
12. 14 days until extraction. 28 days after extraction.
13. 14 days until extraction. 30 days after extraction.
14. For cyanazine, cool to 4°C only.
15. 7 days until derivitization. 1 day after derivitization.
16. 7 days until extraction. 21 days after extraction.
17. 14 days until extraction. 14 days after extraction.
18. 28 days until extraction. 48 hours after extraction.
19. Sodium Sulfite may be used as a dechlorinating agent in some instances. Verify with laboratory prior to sampling.
20. Sterilized. Plastic must be Polypropylene.
21. 40 CFR part 141.74 regulations to avoid filtration or disinfection state 8 hours (DW compliance testing). Most facilities are using either disinfection or filtration so the 8 would not apply in most cases.
22. 40 CFR part 141.74 regulations for Disinfection By-Product rule state 8 hours (DW compliance testing) where SM 9215 allows up to 24 hours if sample is stored between > 0 and $\leq 4^{\circ}$ C
23. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to $\leq 6^{\circ}$C is acceptable.
24. Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.

Table 23-2
Holding Times, Preservation and Container Requirements: NPDES – Bacteria, Protozoa, Toxicity Tests

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp.	PRESERVATION[2,3] Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Total, Fecal, and E.coli Coliforms	Plastic/Glass	10°C	0.0008 % $Na_2S_2O_3$[6]	6 hours	100 mL
Fecal Streptococci	Plastic/Glass	10°C	0.0008 % $Na_2S_2O_3$[6]	6 hours	100 mL
Enterococci	Plastic/Glass	10°C	0.0008 % $Na_2S_2O_3$[6]	6 hours	100 mL
Cryptosporidium	LPDE Plastic	0-8°C	None	96 Hours	500 mL
Giardia	LPDE Plastic	0-8°C	None	96 Hours	500 mL
Toxicity – Acute/Chronic	Plastic/Glass	≤ 6°C[5]	None	36 Hours	2 L

Key to Table

1. Plastic should be Polypropylene or other sterilizable plastic.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
5. Samples must not be frozen. Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present, when samples arrive, it is necessary to measure the temperature of the samples and confirm that the ≤ 6°C temperature has not been exceeded.
6. Should only be used in the presence of residual chlorine.

Table 23-3
Holding Times, Preservation and Container Requirements: NPDES - Inorganic

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp[14].	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Acidity	Plastic/Glass	≤ 6ºC	None	14 days	100 mL
Alkalinity	Plastic/Glass	≤ 6ºC	None	14 days	100 mL
Ammonia	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH<2	28 days	400 mL
BOD 5 Day	Plastic/Glass	≤ 6ºC	None	48 hours	1000 mL
Boron	Plastic[5]	None	HNO_3 to pH<2	6 months	200 mL
Bromide	Plastic/Glass	None	None	28 days	100 mL
CBOD 5 Day	Plastic/Glass	≤ 6ºC	None	48 hours	1000 mL
COD	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH<2	28 days	100 mL
Chloride	Plastic/Glass	None	None	28 days	50 mL
Chlorine, Residual	Plastic/Glass	None	None	15 min.[6]	200 mL
Color	Plastic/Glass	≤ 6ºC	None	48 hours	50 mL
Cyanide –Total[16,17]	Plastic/Glass	≤ 6ºC	NaOH to pH >12, 0.6 g ascorbic Acid[7]	14 days	100 mL
Cyanide – Amenable[16,17]	Plastic/Glass	≤ 6ºC	NaOH to pH >12, 0.6 g ascorbic Acid[7]	14 days	100 mL
Fluoride	Plastic	None	None	28 days	300 mL
Hardness	Plastic/Glass	None	HNO_3 to pH<2[8]	6 months	100 mL
Hexavalent, Chromium	Plastic/Glass	≤ 6ºC	Ammonium sulfate buffer pH = 9.3 - 9.7	28 dys / 24 hrs[15]	200 mL
Hydrogen Ion (pH)	Plastic/Glass	None	None	15 min.[6]	200 mL
Kjeldahl and organic Nitrogen	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH <2	28 days	500 mL
Mercury[11]	Plastic/Glass	None	HNO_3 to pH<2	28 days	200 mL
Metals[9,10]	Plastic/Glass	None	HNO_3 to pH<2[18]	6 months	200 mL
Nitrate	Plastic/Glass	≤ 6ºC	None	48 hours	100 mL
Nitrate-Nitrite	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH <2	28 days	100 mL
Nitrite	Plastic/Glass	≤ 6ºC	None	48 hours	100 mL
Oil and Grease	Glass	≤ 6ºC	H_2SO_4 or HCl to pH <2	28 days	1 L

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp[14].	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Organic Carbon (TOC)	Plastic/Glass	≤ 6°C	H_2SO_4 or HCl to pH <2[12]	28 days	250 mL
Orthophosphate	Plastic/Glass	≤ 6°C	Filter within 15 min.	48 hours	250 mL
Oxygen, Dissolved Probe	Glass[13]	None	None	15 min.[6]	200 mL
Oxygen, Winkler	Glass[13]	None	Fix on site and store in dark.	8 hours	300 mL
Phenols	Glass	≤ 6°C	H_2SO_4 to pH <2	28 days	500 mL
Phosphorus, Elemental	Glass	≤ 6°C	None	48 hours	250 mL
Phosphorus, Total	Plastic/Glass	≤ 6°C	H_2SO_4 to pH <2	28 days	250 mL
Residue, Total	Plastic/Glass	≤ 6°C	None	7 days	1 L
Residue, Filterable	Plastic/Glass	≤ 6°C	None	7 days	1 L
Residue, Non-Filterable	Plastic/Glass	≤ 6°C	None	7 days	1 L
Residue, Settleable	Plastic/Glass	≤ 6°C	None	48 hours	1 L
Residue, Volatile	Plastic/Glass	≤ 6°C	None	7 days	1 L
Silica	Plastic[5]	≤ 6°C	None	28 days	250 mL
Specific Conductance	Plastic/Glass	≤ 6°C	None	28 days	250 mL
Sulfate	Plastic/Glass	≤ 6°C	None	28 days	250 mL
Sulfide	Plastic/Glass	≤ 6°C	Zinc acetate plus NaOH to pH>9	7 days	500 mL
Sulfite	Plastic/Glass	None	None	15 min.[6]	200 mL
Surfactants	Plastic/Glass	≤ 6°C	None	48 hours	1 L
Temperature	Plastic/Glass	None	None	N/A	100 mL
Turbidity	Plastic/Glass	≤ 6°C	None	48 hours	1 L

Key to Table

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at ≤6°C until compositing and sample splitting is completed.

Key to Table

3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
5. May also be collected in quartz or PFTE Plastic.
6. For compliance testing, the analysis must be performed in the field at the time of analysis. If transported to the laboratory for analysis, the analysis will be performed as soon as practical and reported qualified.
7. Should only be used in the presence of residual chlorine. (Alternatively, sodium arsenite may be used)
8. H_2SO_4 to a pH <2 is also acceptable.
9. Except Mercury and Hexavalent Chromium.
10. For dissolved metals, samples must be filtered on site before adding HNO_3 preservative (or before shipping to laboratory).
11. Samples collected for determination of trace level mercury (100 ng/L) using EPA 1631 must be collected in tightly capped fluoropolymer or glad bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipping, samples should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. Samples that been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.
12. Phosphoric acid (H_3PO_4) may also be used.
13. Should have glass lid or top.
14. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of "≤ °C" is used in place of the "4 °C" and "<4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).
15. Holding time is 24 hours if pH adjustment is not performed.
16 In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH. If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered (with sulfide treatment by laboratory) and qualify the results in the final report.
17 It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.
18 Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.

Table 23-4
Holding Times, Preservation and Container Requirements: NPDES - Organic

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.[15]	PRESERVATION[1,2] Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Purgeable Halocarbons	Glass[4]	≤ 6°C	0.0008 % $Na_2S_2O_3$[5]	14 days	40 mL
Purgeable Aromatic Hydrocarbons	Glass[4]	≤ 6°C	0.0008 % $Na_2S_2O_3$[5], HCl to pH<2	14 days[6]	40 mL
Acrolein and Acrylonitrile	Glass[4]	≤ 6°C	0.0008 % $Na_2S_2O_3$[5], adjust pH to 4-5[7]	14 days	40 mL
Phenols[9]	Glass[4]	≤ 6°C	0.0008 % $Na_2S_2O_3$[5]	7 days[8]	1 L
Benzidines[9]	Glass[4]	≤ 6°C	0.0008 % $Na_2S_2O_3$[5]	7 days[8, 11]	1 L
Phthalate esters[9]	Glass[4]	≤ 6°C	None	7 days[8]	1 L
Nitosamines[9,12]	Glass[4]	≤ 6°C	0.0008 % $Na_2S_2O_3$[5,13]	7 days[8]	1 L
PCBs[9]	Glass[4]	≤ 6°C	None	1 year[8]	1 L
Nitroaromatics and Isophorone[9]	Glass[4]	≤ 6°C	0.0008 % $Na_2S_2O_3$[5,13]	7 days[8]	1 L
Polynuclear Aromatic Hydrocarbons[9]	Glass[4]	≤ 6°C	0.0008 % $Na_2S_2O_3$[5,13]	7 days[8]	1 L
Haloethers[9]	Glass[4]	≤ 6°C	0.0008 % $Na_2S_2O_3$[5]	7 days[8]	1 L
Chlorinated Hydrocarbons[9]	Glass[4]	≤ 6°C	None	7 days[8]	1 L
CDD/CDFs[9] – Aqueous: Field/Lab Preservation	Glass	≤ 6°C	pH <9, 0.0008 % $Na_2S_2O_3$[5]	1 year	1 L
CDD/CDFs[9] – Solids/Mixed Phase/ - Field Preservation	Glass	≤ 6°C	None	7 days	1 L
CDD/CDFs[9] – Tissue – Field Preservation	Glass	≤ 6°C	None	24 hours	
CDD/CDFs[9] – Solids/Mixed Phase/Tissue - Lab Preservation	Glass	< -10°C	None	1 year	1 L
Pesticides[9]	Glass	≤ 6°C	pH 5-9 [14]	7 days[8]	1 L

Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at $\leq 6^{\circ}C$ until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
4. With Teflon lined septum.
5. Should only be used in the presence of residual chlorine. (Ascorbic acid may be used instead)
6. Samples receiving no pH adjustments must be analyzed within 7 days. If 2-chlorovinylethylether is a target analyte, the sample should not be acidified.
7. The pH adjustment is not required if acrolein is not being measured. Samples for acrolein receiving no pH adjustment must be analyze within three days of sampling.
8. 7 days until extraction, 40 days after extraction. (PCB only – 1 year after extraction)
9. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more categories, the sample may be preserved by cooling to $\leq 6^{\circ}C$ reducing residual chlorine with 0.0008 % sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9. Samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine) and footnotes 10 and 11(re the analysis of Benzidine).
10. If 1,2-diphenylhydrazine is likely to be present, adjust pH to of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.
11. Extracts may be stored up to 30 days before analysis if storage temperature is $< 0^{\circ}C$.
12. For the analysis of diphenylnitrosamine, add 0.008 % $Na_2S_2O_3$ and ajust pH to 7-10 with NaOH within 24 hours of sampling.
13. Store in dark.
14. The pH adjustment may be performed upon receipt in the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin , add 0.0008 % $Na_2S_2O_3$.
15. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of "≤ °C" is used in place of the "4 °C" and "<4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

Table 23-5.
Holding Times, Preservation and Container Requirements: NPDES - Radiological

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.	PRESERVATION[1,2] Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Alpha, Beta, Radium	Plastic/Glass	None	HNO_3 to pH<2	6 months	1 L

Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

Table 23-6.
Holding Times, Preservation and Container Requirements: RCRA - Aqueous

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp.[12]	PRESERVATION[2,3] Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Chloride	Plastic/Glass	4°C	None	28 days	100 mL
Cyanide -Total	Plastic/Glass	4°C	NaOH to pH >12[5]	14 days	250 mL
Cyanide -Amenable	Plastic/Glass	4°C	NaOH to pH >12[5]	14 days	250 mL
Hydrogen Ion (pH)	Plastic/Glass	4°C	None	24 hours[11]	100 mL
Nitrate	Plastic/Glass	4°C	None	48 hours	100 mL
Oil and Grease	Glass	4°C	HCl	28 days	1 L
Organic carbon (TOC)	Plastic/Glass	4°C	pH to <2[6] Store in dark	28 days	2 X 40 mL
Sulfate	Plastic/Glass	4°C	None	28 days	400 mL
Sulfide	Plastic/Glass	4°C	Add Zn Acetate	7 days	400 mL
Chromium VI	Plastic/Glass	4°C	None	24 hours	250 mL
Mercury	Plastic/Glass	None	HNO_3 to pH<2	28 days	250 mL
Other Metals	Plastic/Glass	None	HNO_3 to pH<2	6 months	250 mL
Acrolein and Acrylonitrile	Glass[10]	4°C	0.0008 % $Na_2S_2O_3$[7] Adjust pH to 4-5[13]	14 days	2-40 ml VOA
Benzidines	Glass[10]	4°C	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L
Chlorinated Hydrocarbons	Glass[10]	4°C	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L
Dioxins and Furans	Glass[10]	4°C	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L
Haloethers	Glass[10]	4°C	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L
Nitroaromatics and cyclic ketones	Glass[10]	4°C	0.0008 % $Na_2S_2O_3$[7], store in dark	7 days[8]	1 L
Nitrosomines	Glass[10]	4°C	0.0008 % $Na_2S_2O_3$[7], store in dark	7 days[8]	1 L
Organochlorine Pesticides	Glass[10]	4°C	None	7 days[8]	1 L
Organophosphorus Pesticides	Glass[10]	4°C	Adjust pH[9]	7 days[8]	1 L
PCBs	Glass[10]	4°C	None	7 days[8]	1 L
Phenols	Glass[10]	4°C	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp.[12]	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Phthalate Esters	Glass[10]	4°C	None	7 days[8]	1 L
Polynuclear Aromatic Hydrocarbons	Glass[10]	4°C	0.0008 % $Na_2S_2O_3$[7], store in dark	7 days[8]	1 L
Purgeable Hydrocarbons	Glass[10]	4°C	0.0008 % $Na_2S_2O_3$[7] Adjust pH <2[2]	14 days	40 mL
Purgeable Halocarbons	Glass[10]	4°C	0.0008 % $Na_2S_2O_3$[7]	14 days	40 mL
Total Organic Halides (TOX)	Glass[10]	4°C	Adjust pH to <2 with H_2SO_4	28 days	2 X 250 mL
Radiological Tests (Alpha, Beta, Radium)	Plastic/Glass	None	HNO_3 to pH<2	6 months	250 mL

Key to Table

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
5. If oxidizing agents are present, add 5 mL 0.1 N $NaAsO_2$ or 0.06 g of ascorbic acid per L. See Cyanide SOP for additional information about other interferences.
6. Adjust pH to <2 with H_2SO_4, HCl, or solid $NaHSO_4$. Free Chlorine must be removed prior to adjustment.
7. Free Chlorine must be removed by the appropriate addition of $Na_2S_2O_3$.
8. 7 days until extraction. 40 days after extraction.
9. Adjust pH to 5-8 using NaOH or H_2SO_4.
10. With Teflon lined septum.
11. Holding Time is listed as "As Soon as Possible" in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
12. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to $\leq$ 6°C is acceptable.
13. Based on guidance from EPA MICE, if samples are received without pH adjustment, the holding time is 7 days.

Table 23-7.
Holding Times, Preservation and Container Requirements: RCRA – Non-Aqueous

PARAMETER	CONTAINER [1]	PRESERVATION Temp.[7]	PRESERVATION Chemical	HOLDING TIME[2]	SAMPLE WEIGHT
Chloride	Glass	4°C	None	28 days	50 g
Cyanide -Total	Glass	4°C	None	14 days	50 g
Cyanide -Amenable	Glass	4°C	None	14 days	50 g
Hydrogen Ion (pH)	Glass	4°C	None	7 days[6]	50 g
Nitrate	Glass	4°C	None	N/A	50 g
Oil and Grease	Glass	4°C	None	28 days	50 g
Sulfide	Glass	4°C	Add Zn Acetate, zero headspace	7 days	50 g
Chromium VI	Glass	4°C	None	30 days[8]	50 g
Mercury	Plastic/Glass	None	None	28 days	50 g
Other Metals	Plastic/Glass	None	None	6 months	50 g
Acrolein and Acrylonitrile	Glass[4]	4°C	None	14 days	100 g
Benzidines	Glass[4]	4°C	None	14 days[3]	100 g
Chlorinated Hydrocarbons	Glass[4]	4°C	None	14 days[3]	100 g
Dioxins and Furans	Glass[4]	4°C	None	14 days[3]	100 g
Haloethers	Glass[4]	4°C	None	14 days[3]	100 g
Nitroaromatics and cyclic ketones	Glass[4]	4°C	None	14 days[3]	100 g
Nitrosomines	Glass[4]	4°C	None	14 days[3]	100 g
Organochlorine Pesticides	Glass[4]	4°C	None	14 days[3]	100 g
Organophosphorus Pesticides	Glass[4]	4°C	None	14 days[3]	100 g
PCBs	Glass[4]	4°C	None	14 days[3]	100 g
Phenols	Glass[4]	4°C	None	14 days[3]	100 g
Phthalate Esters	Glass[4]	4°C	None	14 days[3]	100 g
Polynuclear Aromatic Hydrocarbons	Glass[4]	4°C	None	14 days[3]	100 g

PARAMETER	CONTAINER [1]	PRESERVATION Temp.[7]	PRESERVATION Chemical	HOLDING TIME[2]	SAMPLE WEIGHT
Purgeable Hydrocarbons	Glass[4]	4°C	None	14 days[5]	100 g
Purgeable Halocarbons	Glass[4]	4°C	None	14 days[5]	100 g
Total Organic Halides (TOX)	Glass[4]	4°C	None	28 days	50 g

Key to Table

1. Plastic should be Polyethylene.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
3. 14 days until extraction. 40 days after extraction.
4. With Teflon Lined Septum
5. See Volatile SOP for more detailed preservation requirements.
6. Holding Time is listed as "As Soon as Possible" in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
7. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to $\leq$ 6°C is acceptable.
8. 30 days to digestion, 7 days from digestion to analysis.

Table 23-8.
Holding Times, Preservation and Container Requirements: Air Samples

PARAMETER	CONTAINER [1]	PRESERVATION		HOLDING TIME[2]	SAMPLE WEIGHT
		Temp.	Chemical		
Volatile Organics	Summa Cannister	None	None	30 days	6L or 1L
Volatile Organics	Tedlar Bag	None	None	72 hrs[3,4]	1 L

Key to Table

1. Plastic should be Polyethylene.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
3. Holding Time is based on SW 846 Method 0040 "SAMPLING OF PRINCIPAL ORGANIC HAZARDOUS CONSTITUENTS FROM COMBUSTION SOURCES USING TEDLAR® BAGS". Some states specifically enforce this holding time (e.g. Florida, New Jersey) and others have not specified this information in their regulatory requirements.
4. The holding time is 72 hours unless the laboratory has a documented validation study that indicates a longer HT is acceptable for the analytes of interest.

SECTION 24

HANDLING OF SAMPLES
(NELAC 5.5.8)

Sample management procedures at ***TestAmerica Buffalo*** ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

24.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and can be initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 24-1.

24.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 24-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The shipping documents are retained with the project files.

24.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC or in the project notes, sample management will initiate Strict Chain of Custody procedures as defined in SOP BF-GP-018, "Strict Internal Chain-of-Custody".

24.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

24.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Login – Analytical Receipt Resolution Form (Figure 24-6). and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

24.2.1.1 Inspection of samples include a check for:

- Complete documentation to include sample identification, location, date and time of collection, collector's name, preservation type, sample type and any additional comments concerning the samples.
- Complete sample labels to include unique identification in indelible ink.
- Use of appropriate sample containers (see Section 23)
- Adherence to holding times as specified in the test method and/or summarized in Section 23.
- Adequate sample volume for required analyses (see Section 23).

- Damage or signs of contamination to sample container. Volatile vials are also inspected for headspace

24.2.1.2 Using an IR temperature gun, check and record the temperature of the samples that require thermal preservation. On a project specific basis, temperature blanks may also be required.

- Samples shall be deemed acceptable if arrival temperature is just above freezing and less than or equal to 6.0° C. Samples that are hand-delivered immediately after collection may not be at the required temperatures; however, if there is evidence that the chilling process has begun, such as the arrival on ice, the samples shall be considered acceptable. This will be documented on the COC and in the LIMs job comments.

- If the samples were shipped in ice and solid ice is still present and in direct contact with samples, report the samples as "received on ice." Direct contact means samples must be surrounded by ice cubes or crushed ice. Ice present in a plastic bottle or other container does not constitute direct contact. Samples shipped with only "blue ice" may not be reported as "received on ice".

24.2.1.3 Verify sample preservation as specified in the test method. Check for correct pH as specified in the test method. The results are documented in the LIMs sample preservation tables. In the case of volatiles (including TOC and TOX) the pH is recorded after analysis in the analysis logbook or on the raw data instrument printout. At the time of analysis, chlorine is checked on samples requiring extractable organics, BOD, TOX, cyanide, fluoride, ammonia, TKN, CBOD and Nitrate; presence or absence is recorded

24.2.1.4 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

24.2.1.5 If samples are received without a COC, TestAmerica will provide a generic COC form to be completed by the client when the samples are brought to the laboratory. The client is always provided with a copy of the completed COC form for their records.

24.2.1.6 If analyses with short holding times are requested, the dates and times are inspected to ensure that holding times have not already expired.

24.2.1.7 TestAmerica Buffalo maintains routine sample acceptance for both 1st and 2nd shift. However, samples received after normal working hours are left in their coolers and placed in a cold storage location. The person receiving the samples must record the date and time received and sign the CoC. Chains of custody are placed in the sample management office and associated samples are addressed by sample management personnel during the next working shift.

24.2.1.8 Any deviations from the checks described in Section 24.2.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance criteria (Section 24.3) are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples

or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

24.2.2 Sample Log-in

All samples that are received by the laboratory are logged into the LIMS to allow the laboratory to track and evaluate sample progress. Each group of samples that are logged in together (typically one project from a given client/sampling event/sample receipt date) is assigned a unique job number. Within each job, each sampling point (or sample) receives a unique number. Sample numbers are generated sequentially over time, and are not re-assigned. A sample may be composed of more than one bottle since different preservatives may be required to perform all analyses requested. Even if multiple containers are received for a single sample, each container is uniquely identified with an alphabetic letter added to the sample number. The LIMS generates sample labels that are attached to each bottle for a given sample.

Each job/set of samples is logged into LIMS with a minimum of the following information:

- Client Name, Project Name, Address, Phone, Fax, Report to information, invoice to information (most of this information is "default information" that is stored in the LIMS).
- Date and time sampled;
- Date and time received;
- Job and/or project description, sample description;
- Sample matrix, special sample remarks;
- Reporting requirements (i.e., QC level, report format, invoicing format);
- Turn-around-time requirements;
- Parameters (methods and reporting limits or MDLs are default information for a given parameter)

24.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (Figure 24-5) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method;
- sample holding times must be adhered to;

- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

24.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. Aqueous samples designated for metals analysis are stored at ambient temperature. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed at a minimum of every two weeks.

Analysts and technicians provide a request form to the cooler custodian who then retrieves the requested samples. In the absence of the cooler custodian, the analysts may personally retrieve the sample containers allocated to their analysis from the designated refrigerator. The samples are placed on carts, transported the analytical area and analyzed. Following analysis the remaining sample is returned to the refrigerator from which it originally came. All unused portions of samples are returned to the secure sample control area. All samples are kept in the refrigerators for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to dry room temperature, sample archive area where they are retained a minimum of 2 weeks after the final report has been issued to the client at which time disposal occurs. Special arrangements may be made to store samples for longer periods of time. Extended archival periods allow additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

24.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, samples which are known or suspected to be hazardous are segregated and a notification is issued to all laboratory personnel. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm. All soil samples, including foreign soil samples are heat treated or incinerated in accordance with USDA permit requirements and are transported / disposed by USEPA approved facilities.

Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

24.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). For sample shipments which include water/solid volatile organic analyses, a trip blank is enclosed when required by method specifications or state or regulatory programs. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

24.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 2 weeks after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: BF-WM-001, "Waste Management".) All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than six weeks from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample may request to participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal and nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client). All disposal of sample containers is accomplished through incineration. A Waste Disposal Record (Figure 24-4) should be completed.

Figure 24-1.

Example: Chain of Custody (COC)

[redacted]

Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL-4142 (0907)

Client	Project Manager	Date	Chain of Custody Number 388703
Address	Telephone Number (Area Code)/Fax Number	Lab Number	Page _____ of _____
City / State / Zip Code	Site Contact / Lab Contact	Analysis (Attach list if more space is needed)	
Project Name and Location (State)	Carrier/Waybill Number		Special Instructions/ Conditions of Receipt
Contract/Purchase Order/Quote No.	Matrix / Containers & Preservatives		

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Air	Aqueous	Sed.	Soil	Unpres.	H_2SO_4	HNO_3	HCl	NaOH	ZnAc/ NaOH

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months
(A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other_____

QC Requirements (Specify)

1. Relinquished By	Date	Time	1. Received By	Date	Time
2. Relinquished By	Date	Time	2. Received By	Date	Time
3. Relinquished By	Date	Time	3. Received By	Date	Time

Comments

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

Figure 24-2.

Example: Strict Internal Chain of Custody (COC)

JOB#_____ Received by:_____ Date &Time_____

Parameter	Samples	Analyst	Date & Time OUT	Date & Time IN	Disposal Date
Small Walk-in cooler (SC#2)					

VOA cooler (SC#1)					

VOA Freezer (SC#3)					

Extract cooler (SC#4)		Received by:_____		Date & Time_____	
		Relinquish by:_____		Date & Time_____	

Figure 24-3

Example: Sample Disposal Record

Date: 12/26/2007 — TestAmerica Laboratories Inc. — Page: 1
Time: 09:58:59 — Sample Disposal Inventory — Rept: AN1468
For ASRF Dates 09/01/2007 -> 09/02/2007 AND Preservation Code "00"

Test Matrix: SOILS

Sampled	Received	Client Sample ID	Lab Smp ID	Bottles	Parameters
08/30/2007	09/01/2007	SB-07-01 (7.5-10)	A7983901	1-4ozGW	HSL PAH
08/30/2007	09/01/2007	SB-07-01 (7.5-10)	A7983901	1-4ozGW	T AS
08/30/2007	09/01/2007	SB-07-01 (7.5-10)	A7983901	3-ENCORE	BTEX
08/30/2007	09/01/2007	SB-07-02 (6-8)	A7983902	1-4ozGW	HSL PAH
08/30/2007	09/01/2007	SB-07-02 (6-8)	A7983902	1-4ozGW	T AS
08/30/2007	09/01/2007	SB-07-02 (6-8)	A7983902	3-ENCORE	BTEX
08/30/2007	09/01/2007	SB-07-03 (6-8)	A7983903	1-4ozGW	HSL PAH
08/30/2007	09/01/2007	SB-07-03 (6-8)	A7983903	1-4ozGW	T AS
08/30/2007	09/01/2007	SB-07-03 (6-8)	A7983903	3-ENCORE	BTEX
08/30/2007	09/01/2007	SB-07-04 (6-8)	A7983904	1-4ozGW	HSL PAH
08/30/2007	09/01/2007	SB-07-04 (6-8)	A7983904	1-4ozGW	T AS
08/30/2007	09/01/2007	SB-07-04 (6-8)	A7983904	3-ENCORE	BTEX
08/30/2007	09/01/2007	SB-07-05 (6-8)	A7983905	1-4ozGW	HSL PAH
08/30/2007	09/01/2007	SB-07-05 (6-8)	A7983905	1-4ozGW	T AS
08/30/2007	09/01/2007	SB-07-05 (6-8)	A7983905	3-ENCORE	BTEX
08/30/2007	09/01/2007	DUP-01	A7983906	1-4ozGW	HSL PAH
08/30/2007	09/01/2007	DUP-01	A7983906	1-4ozGW	T AS
08/30/2007	09/01/2007	DUP-01	A7983906	3-ENCORE	BTEX
08/27/2007	09/01/2007	SB-38-15/16-03	A7984801	1-8ozGW	8260/8270 STARS
08/27/2007	09/01/2007	SB-36-15/16-03	A7984802	1-8ozGW	8260/8270 STARS
08/27/2007	09/01/2007	SB-36-25/30-03	A7984803	1-8ozGW	8260/8270 STARS
08/28/2007	09/01/2007	SB-35-19/20-03	A7984804	1-4ozGW	8260 STARS
08/28/2007	09/01/2007	SB-35-19/20-03	A7984804	1-4ozGW	8270 STARS
08/29/2007	09/01/2007	SB-33-17/18-03	A7984805	1-4ozGW	8260 STARS
08/29/2007	09/01/2007	SB-33-17/18-03	A7984805	1-4ozGW	8270 STARS
08/29/2007	09/01/2007	SB-32-20/21-03	A7984806	1-4ozGW	8260 STARS
08/29/2007	09/01/2007	SB-32-20/21-03	A7984806	1-4ozGW	8270 STARS
08/30/2007	09/01/2007	SW-20	A7984901	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	SW-34	A7984902	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	SW-35	A7984903	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	SW-36	A7984904	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	SW-37	A7984905	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	SW-38	A7984906	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	SW-39	A7984907	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	B-22	A7984908	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	B-23	A7984909	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	B-24	A7984910	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	B-25	A7984911	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	B-26	A7984912	1-8ozGW	8260;8270;LPH
08/29/2007	09/01/2007	SW-09	A7985001	1-8ozGW	8260;8270;LPH
08/29/2007	09/01/2007	SW-10	A7985002	1-8ozGW	8260;8270;LPH
08/29/2007	09/01/2007	SW-11	A7985003	1-8ozGW	8260;8270;LPH
08/29/2007	09/01/2007	SW-12	A7985004	1-8ozGW	8260;8270;LPH
08/29/2007	09/01/2007	SW-13	A7985005	1-8ozGW	8260;8270;LPH
08/29/2007	09/01/2007	SW-14	A7985006	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	SW-15	A7985007	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	SW-16	A7985008	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	SW-17	A7985009	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	SW-18	A7985010	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	SW-19	A7985011	1-8ozGW	8260;8270;LPH
08/30/2007	09/01/2007	B-21	A7985012	1-8ozGW	8260;8270;LPH

Figure 24-4.

Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - *Client name, address, phone number and fax number (if available)*
   - *Project name and/or number*
   - *The sample identification*
   - *Date, time and location of sampling*
   - *The collectors name*
   - *The matrix description*
   - *The container description*
   - *The total number of each type of container*
   - *Preservatives used*
   - *Analysis requested*
   - *Requested turnaround time (TAT)*
   - *Any special instructions*
   - *Purchase Order number or billing information (e.g. quote number) if available*
   - *The date and time that each person received or relinquished the sample(s), including their signed name.*
   - ***The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.***
   - **Information must be legible**

2) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - **Information must be legible**

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

4) Samples must be preserved according to the requirements of the requested analytical method or in accordance with the instructions provided by TestAmerica.
   Note: Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

- Chemical preservation (pH) will be verified prior to analysis and the project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

- For Volatile Organic analyses in drinking water (Methods 502.2 or 524.2). Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
  - 1. Test for residual chlorine in the field prior to sampling.
    - If no chlorine is present, the samples are to be preserved using HCl as usual.
    - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCl.
  - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.
- **FOR WATER SAMPLES TESTED FOR CYANIDE – for NPDES samples**
  - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH.

    - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered and qualify the results in the final report.

  - It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.

  - The laboratory must test the sample for oxidizing agents (e.g. Chlorine) prior to analysis and treat according to the methods prior to distillation. (ascorbic acid or sodium arsenite are the preferred choice).

5) Sample Holding Times
   - TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time to ensure analysis.

   - Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis.

6) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply this blank with the bottle order.

7) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.

8) Recommendations for packing samples for shipment.

   - Pack samples in Ice rather than "Blue" ice packs.
   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
   - Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
   - Fill extra cooler space with bubble wrap.

Figure 24-5.

Example: Cooler Receipt Form

TestAmerica Buffalo

Doc. Login/ARRF - Side A
Rev 5
November 6, 2007

SAMPLE LOGIN **JOB #**

Shipment ID_____ **Strict Internal COC:** YES / NO

Residual Chlorine Check: ☐

Radiation Check <0.02 mR/hr: YES / NO

AC _____ **Project / Task** _____/_____

TAT_____BD/_____CD # OF SAMPLES _____TRIP BLANK Y/N # _____

SHIPPED BY	ATTACH SHIPPING TAGS
RECEIVED DATE / TIME:	_____/_____/____ _____:____

COOLER TEMP	°C (<6 °C)	OK	NO

Cooler Custody Seal intact? YES/NO NONE SEAL # _____

If NO to cooler temp or seal, PM notified? YES_____ (PM Name)

SUBCONTRACT YES/NO **LAB**_____ **SM #** _____

COMMENTS: SAMPLE TIME **ACTUAL** **+1HR** **+2 HR** **+3 HR** **NONE**

Sample received outside hold time_____

Headspace in VOA vials_____

Problems with bottle labels_____

OTHER SAMPLE RECEIPT COMMENTS (Fill out ARRF, see reverse)

_____

_____

PRESERVATION CHECKED YES___ NO___ NA___ **Initials**_____

ARE SAMPLE DATES AND TIMES CORRECT? **Initials**_____

WERE ALL THE APPROPRIATE TESTS ASSIGNED? **Initials**_____

Temp.Cert.Loss:_____

SECTION 25.0

ASSURING THE QUALITY OF TEST RESULTS
(NELAC 5.5.9)

25.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 21, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

25.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

25.3 NEGATIVE CONTROLS

25.3.1 **Method Blanks** are used to assess preparation and analysis for possible contamination during the preparation and processing steps.

25.3.1.1 The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.

25.3.1.2 The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).

25.3.1.3 The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.

25.3.1.4 Evaluation criteria and corrective action for method blanks is defined in the specific standard operating procedure for each analysis. Generally, corrective action is taken if the concentration of a target analyte in the blank is at or above the reporting limit as established by the method or regulation:

- The source of contamination is investigated

- Measures are taken to minimize or eliminate the source of the contamination
- Affected samples are reprocessed or the results are qualified on the final report.

25.3.2 **Calibration Blanks** are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

25.3.3 **Instrument Blanks** are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

25.3.4 **Trip Blanks** are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples. Trip Blanks are also sometimes referred to as Travel Blanks.

25.3.5 **Field Blanks** are sometimes used for specific projects by the field samplers. A field blank is prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

25.3.6 **Equipment Blanks** are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

25.3.7 **Holding Blanks**, also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory (refer to section 24.4).

25.3.8 **Field blanks, equipment blank and trip blanks**, when received, are analyzed in the same manner as other field samples. When known, blanks should not be selected for matrix QC, as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB".

25.4 POSITIVE CONTROLS

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)),

which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

25.4.1 Method Performance Control - Laboratory Control Sample (LCS)

25.4.1.1 The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

25.4.1.2 The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard may be reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

25.4.1.3 Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

25.4.1.4 As stated in the opening of this section, the LCS goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).

25.4.1.5 The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

25.4.1.6 If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected

components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

25.4.1.6.1 For methods that have 1-10 target analytes, spike all components.

25.4.1.6.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

25.4.1.6.3 For methods with more than 20 target analytes, spike at least 16 components.

25.4.1.6.4 Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

25.4.1.6.5 Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

25.4.1.7 **Accuracy Calculation**: Percent Recovery (%R) Calculation (applies to LCS, CCV, Surrogates, and Matrix Spikes.

$$\%R = \frac{AV}{TV} \times 100$$

Where: AV = Analyzed Value
TV = True Value

25.5 SAMPLE MATRIX CONTROLS

25.5.1 Matrix Spikes (MS)

25.5.1.1 The Matrix spike is used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used.

25.5.1.2 An MS is essentially a sample fortified with a known amount of the test analyte(s). At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.

25.5.1.3 If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number of the listed components (see LCS analytes 25.4.1.6 above) may be used to control the test method. The selected components of each spiking mix shall represent all

chemistries, elution patterns and masses, permit-specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

25.5.1.4 The percent recovery calculation for matrix spikes is essentially the same as the calculation shown in 25.2.1.7 except that:

AV = Sp – Sa

Where: Sp = Spike result
Sa = Sample result

25.5.2 Surrogate Spikes

25.5.2.1 Surrogate Spikes are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.

25.5.2.2 Surrogate compounds are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method (also refer to Section 25.5). Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.

25.5.3 Duplicates

25.5.3.1 For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure. Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

25.5.3.2 Precision Calculation (Relative Percent Difference - RPD)

$$RPD = \frac{|S - D|}{\frac{(S + D)}{2}} \times 100$$

Where: S=Sample Concentration
D=Duplicate Concentration

25.5.4 Internal Standards

25.5.4.1 In most organic analyses, internal standards are spiked into all environmental and quality control samples (including the initial calibration standards). An internal standard is also used with some metals analyses. It is added to sample extracts after the extraction (post-prep). The acceptance criteria in most methods are 50% to 200% of the responses in the mid-point of the corresponding calibration curve. Consult the method-specific SOPs for details on the internal standard compounds, calculations and acceptance criteria.

25.5.4.2 When the internal standard recoveries fall outside these limits, if there are not obvious chromatographic interferences, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets internal standard recovery criteria, the second run is reported (or both are reported if requested by the client).

25.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

25.6.1 Each individual analyte in the LCS, MS, or Surrogate Spike are evaluated against the control limits as published in the test method. Where there are no established acceptance criteria, the laboratory calculates control limits with the use of statistical evaluations or, in some cases, utilizes client project specific or regulatory mandated control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

25.6.2 Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating (e.g. EPA SW846 8000 series methods). Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

25.6.2.1 The lab should consider the effects of the spiking concentration control limits, and to avoid censoring of data. The acceptance criteria for recovery and precision are often a function of the spike concentration used. Therefore, caution must be used when pooling data to generate control limits.

25.6.2.2 Not only should the results all be from a similar matrix, but the spiking levels should also be approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results should all be generated using the same set of extraction, cleanup and analysis techniques.

25.6.2.3 The laboratory should try and avoid discarding data that do not meet a preconceived notion of acceptable performance. This results in a censored data set, which, when used to develop acceptance criteria, will lead to unrealistically narrow criteria. For a 99% confidence interval, 1 out of every 100 observations likely will still fall outside the limits. For methods with long analyte lists this may mean occasional failures every batch or two. While professional judgment is important in evaluating data to be

used to develop acceptance criteria, specific results are not discarded simply because they do not meet one's expectations. However, data points shall be discarded if they were the result of human or mechanical error or sample concentration exceeded spike level by > 4x.

25.6.3 Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

25.6.3.1 Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

25.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method.

25.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable.

25.6.3.4 The maximum acceptable recovery limit will be 150%.

25.6.3.5 The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

25.6.3.6 If either the high or low end of the control limit changes by $\leq$ 5% from previous, the data points are inspected and, using professional judgment, the limits may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

25.6.4 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

25.6.4.1 The control limits are maintained in the laboratory LIMs system. The limits for each analyte/method/matrix combination are assigned effective and expiration dates. The QA department is able to query the LIMs sytem and print an active list of control limits based on this database. The most current laboratory limits (based on the effective/expiration dates) are reflected on the laboratory worksheets and final reports unless superseded by project specific limits.

25.6.5 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

25.6.5.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

25.6.5.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

25.6.5.3 Or, for NELAC and Department of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME). This information may be found in the laboratory specific SOP BF-QA-005, "Preventative and Corrective Action".

- <11 analytes – 0 marginal exceedances are allowed.
- 11 – 30 Analytes – 1 marginal exceedance is allowed
- 31-50 Analytes – 2 marginal exceedances are allowed
- 51-70 Analytes – 3 marginal exceedances are allowed
- 71-90 Analytes – 4 marginal exceedances are allowed
- > 90 Analytes – 5 marginal exceedances are allowed

25.6.5.3.1 Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).

25.6.5.3.2 Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken.

25.6.5.3.3 Though marginal excedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

25.6.6 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the method specific SOPs and in Section 13.

25.6.7 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

25.7 METHOD DETECTION LIMITS (MDLs)

MDLs, calculated as described in Section 20.7, are updated or verified annually or more often if required by the method.

25.8 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

25.8.1 The laboratory has written procedures to assure the accuracy of the test method including calibration (see Section 21), use of certified reference materials (see Section 22) and use of PT samples (see Section 16).

25.8.2 A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 20.

25.8.3 Use of formulae to reduce data is discussed in the method standard operating procedures and in Section 21.

25.8.4 Selection of appropriate reagents and standards is included in Section 9 and 22.

25.8.5 A discussion on selectivity of the test is included in Section 5.

25.8.6 Constant and consistent test conditions are discussed in Section 19.

25.8.7 The laboratories sample acceptance policy is included in Section 24.

25.8.8 A listing of the type of test result correlations that are looked at during report review (e.g. Total Chromium should be greater or equal to Hexavalent Chromium) is included in Section 20.13.4.5 and in laboratory specific SOP BF-PM-005, "Correctness of Analysis".

SECTION 26.0

REPORTING RESULTS
(NELAC 5.5.10)

26.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is a conflict between the client requested formats and accreditation requirements or data usability information, accreditation requirements and data usability information will take precedence over client requests. A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 20.

26.2 TEST REPORTS

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

26.2.1 A report title (e.g. Analytical Report) with a "sample results" column header.

26.2.2 The report cover page is printed on company letterhead which includes the laboratory name, address and telephone number.

26.2.3 A unique identification of the report (e.g. job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as # / ##. Where the first number is the page number and the second is the total number of pages.

26.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.
- In most cases, the applicable COC is paginated and is an integral part of the report.

- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (eg. Sampling information).

26.2.5 The name and address of client and a project name/number, if applicable.

26.2.6 Client project manager or other contact

26.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

26.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

26.2.9 Date reported or date of revision, if applicable.

26.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

26.2.11 Practical quantitation limits or client reporting limit.

26.2.12 Method detection limits (if requested)

26.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

26.2.14 Sample results.

26.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits (if requested).

26.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 26.2.4 – Item 3 regarding additional addenda). Sample temperatures are recorded in the report case narrative and on the CoC. Deviations from normal conditions (e.g., preservation, breakage) are recorded in the report case narrative.

26.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

26.2.18 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

26.2.19 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

26.2.20 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

26.2.21 The laboratory includes a cover letter.

26.2.22 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

26.2.23 When Soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

26.2.24 Appropriate laboratory certification number for the state of origin of the sample if applicable.

26.2.25 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report and that a complete report will follow once all of the work has been completed.

26.2.26 Any out of network subcontracted analysis results are provided as an addendum to the report on the official letterhead of the subcontractor. All in-network subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

26.3 REPORTING LEVEL OR REPORT TYPE

TestAmerica Buffalo offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 26.2 above.
- Level II is a Level I report plus summary information, including results for the method blank, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 26.7.

26.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. ***TestAmerica Buffalo*** offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific

electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

26.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report. Refer to Appendix 7 for a list of the laboratory's standard footnotes and qualifiers.

26.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

26.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

26.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

26.4.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

26.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If ***TestAmerica Buffalo*** is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

26.6 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

26.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. It is our policy that facsimiles are intended for and should be used for business purposes only. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify the sender.

26.7 FORMAT OF REPORTS

The format of reports are designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

26.8 AMENDMENTS TO TEST REPORTS

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 13).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R". The revised report will have the word "revised" appended to the cover letter.

When the report is re-issued, a notation of "revised" is placed on the cover/signature page of the report. A brief explanation of reason for the re-issue is included in the report case narrative.

26.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

26.9.1 Sample Reanalysis Policy

Because there is a certain level of uncertainty with any analytical measurement a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g. sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client specific arrangements for reanalysis protocols can be established.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Department Manager, Operations Manager, Technical Director, QA Manager or Laboratory Director if unsure.

26.9.2 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).

- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

26.9.3 Multiple Reports

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1.

TESTAMERICA
ETHICS POLICY No. CA-L-P-001

Refer to CA-L-P-001 for complete policy.

TestAmerica
EMPLOYEE ETHICS STATEMENT

I understand that TestAmerica is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

- *With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:*
- *I will not intentionally report data values that are inconsistent with the actual values observed or measured.*
- *I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations.*
- *I will not intentionally misrepresent another individual's work as my own or represent my own work as someone else's.*
- *I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers; I shall not modify data (either sample or QC data) unless the modification can be technically justified through a measurable analytical process, such as one deemed acceptable to the laboratory's Standard Operating Procedures, Quality Assurance Manual or Technical Director. All such modifications must be clearly and thoroughly documented in the appropriate laboratory notebooks/worksheets and/or raw data and include my initials or signature and date.*
- *I shall not make false statements to, or seek to otherwise deceive, members of Management or their representatives, agents, or clients/customers. I will not, through acts of commission, omission, erasure, or destruction, improperly report measurement standards, quality control data, test results or conclusions.*
- *I shall not compare or disclose results for any Performance Testing (PT) sample, or other similar QA or QC requirements, with any employee of any other laboratory, including any other TestAmerica laboratory, prior to the required submission date of the results to the person, organization, or entity supplying the PT sample.*
- *I shall immediately inform my supervisor or other member of management regarding any intentional or unintentional reporting of my own inauthentic data. Such report shall be given both orally and in writing to the supervisor or other member of management contacted and to the local Quality Assurance Manager. The Quality Assurance Manager will initial and date the information and return a copy to me. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and will immediately report its occurrence. If I have actual knowledge of such acts committed by any other employees, and I do not report such information to designated members of Management, it shall be considered as serious as if I personally committed the offense. Accordingly, in that event, I understand that I may be subject to immediate termination of employment.*
- *I understand that if any supervisor, manager, or representative of TestAmerica management instructs, requests, or directs me to perform any of the aforementioned improper laboratory practices, or if I am in doubt or uncertain as to whether or not such laboratory practices are proper, I will not*

comply. In fact, I must report such event to all appropriate members of Management including, but not limited to, the Lab Director, all supervisors and managers with direct line reporting relationship between me and the Lab Director, and the local Quality Assurance representative, excluding such individuals who participated in such perceived improper instruction, request, or directive. In addition, I may contact Corporate Quality Assurance / Ethics Compliance Officer(s) for assistance.

- *I understand the critical importance of accurately reporting data, measurements, and results, whether initially requested by a client, or retained by TestAmerica and submitted to a client at a later date, or retained by TestAmerica for subsequent internal use;*
- *I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the TestAmerica family of companies.*
- *I shall not accept gifts of a value that would adversely influence judgment.*
- *I shall avoid conflicts of interest and report any potential conflicts to the management (e.g. employment or consulting with competitors, clients, or vendors).*
- *I shall not participate in unfair competition practices (e.g. slandering competitors, collusion with other labs to restrict others from bidding on projects).*
- *I shall not misrepresent certifications and status of certifications to clients or regulators.*
- *I shall not intentionally discharge wastes illegally down the drain or onto the ground.*
- *I understand that any attempt by management or an employee to circumvent these policies will be subject to disciplinary action.*

As a TestAmerica employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

I understand that if my job includes supervisory responsibilities, I shall not instruct, request, or direct any subordinate to perform any laboratory practice which is unethical or improper. Also, I shall not discourage, intimidate, or inhibit an employee who may choose to appropriately appeal my supervisory instruction, request, or directive which the employee perceives to be improper, nor retaliate against those who do.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination of my employment. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE _____ Date _____

Supervisor/Trainer: _____ Date _____

Work Instruction No. CA-WI-005

TestAmerica
CONFIDENTIALITY AND PROPRIETARY INFORMATION AGREEMENT

TestAmerica and their predecessors, in their businesses, have developed and use commercially valuable technical and non-technical information and to guard the legitimate interests of TestAmerica and its clients, it is necessary to protect certain information as confidential and proprietary.

I, _____ , understand and acknowledge that during the term of my employment by TestAmerica, I will be privy to and entrusted with certain confidential information and trade secrets of TestAmerica and its clients.

Confidential information and trade secrets include, but are not limited to: customer and client lists; price lists; marketing and sales strategies and procedures; operational and equipment techniques; standard operating procedures; business plans and systems; quality control procedures and systems; special projects and technological research, including projects, research and reports for any government entity or client; client's plans and processes; client's manner of operation; the trade secrets of clients; client's data; vendor or supplier pricing; employee lists and personal information, and any other records, data, files, drawings, inventions, discoveries, applications, or processes which are not in the public domain.

I agree as follows:

1. I will not in any way, during the term of my employment, or at any time thereafter, except as authorized in writing by the Legal Department of TestAmerica or the client where client data is involved, disclose to others, use for my own benefit, remove from TestAmerica's premises (except to the extent off-site work is approved by my supervisor), copy or make notes of any confidential information and/or trade secrets of TestAmerica or its clients, excepting only that information which may be public knowledge. Technical and business information of any previous employer or other third party which I may disclose to TestAmerica shall be limited to that which was acquired legitimately and disclosed to me without restriction as to secrecy.

2. I agree that all inventions (whether or not patentable) conceived or made by me during the period of my employment by TestAmerica shall belong to TestAmerica, provided such inventions grow out of my work for TestAmerica and are related to the business of TestAmerica. I agree to disclose and assign such inventions to TestAmerica. In California, this provision shall not apply to any invention which qualifies fully under Section 2870 of the California Labor Code.

3. On termination of my employment from TestAmerica, I will deliver to TestAmerica all documents, records, notes, data, memoranda, files, manuals, equipment and things of any nature which relate in any way to confidential information and/or trade secrets of TestAmerica or its clients and which are in my possession or under my control.

4. I agree that during the period of my employment and for one (1) year from and after the termination (for any reason) of my employment with TestAmerica, I shall not directly or indirectly (without first obtaining the written permission of TestAmerica), recruit for employment, or induce to terminate his or her employment with TestAmerica, any person who is an active employee of TestAmerica on the last day of my employment with TestAmerica.

5. I acknowledge that if I were to breach any provision of this Confidentiality Agreement, money damages will be inadequate, and I hereby agree that TestAmerica shall be entitled, where appropriate, to specific performance and/or injunctive relief (i.e. to require me to comply with this Agreement). I further acknowledge that the willingness of TestAmerica to hire me or to continue my employment constitutes full and adequate consideration for the agreements, and obligations to which I have agreed as set forth in this document.

I have executed this Agreement, intending to be legally bound.

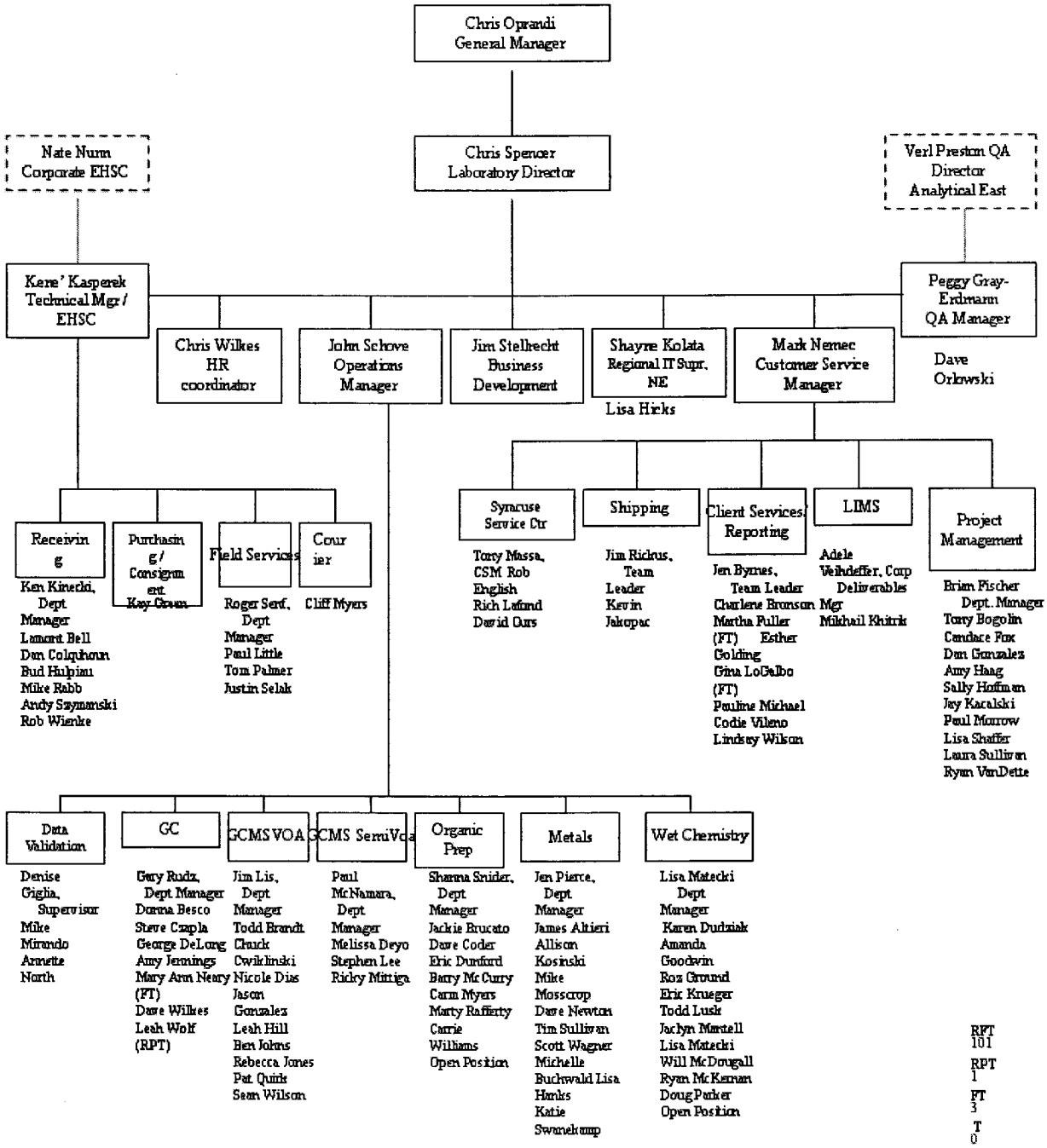
_____	_____	_____
Printed Name	*Signature*	*Date*

Work Instruction No. CA-WI-006

Appendix 2.

TestAmerica Buffalo Laboratory Organization Chart

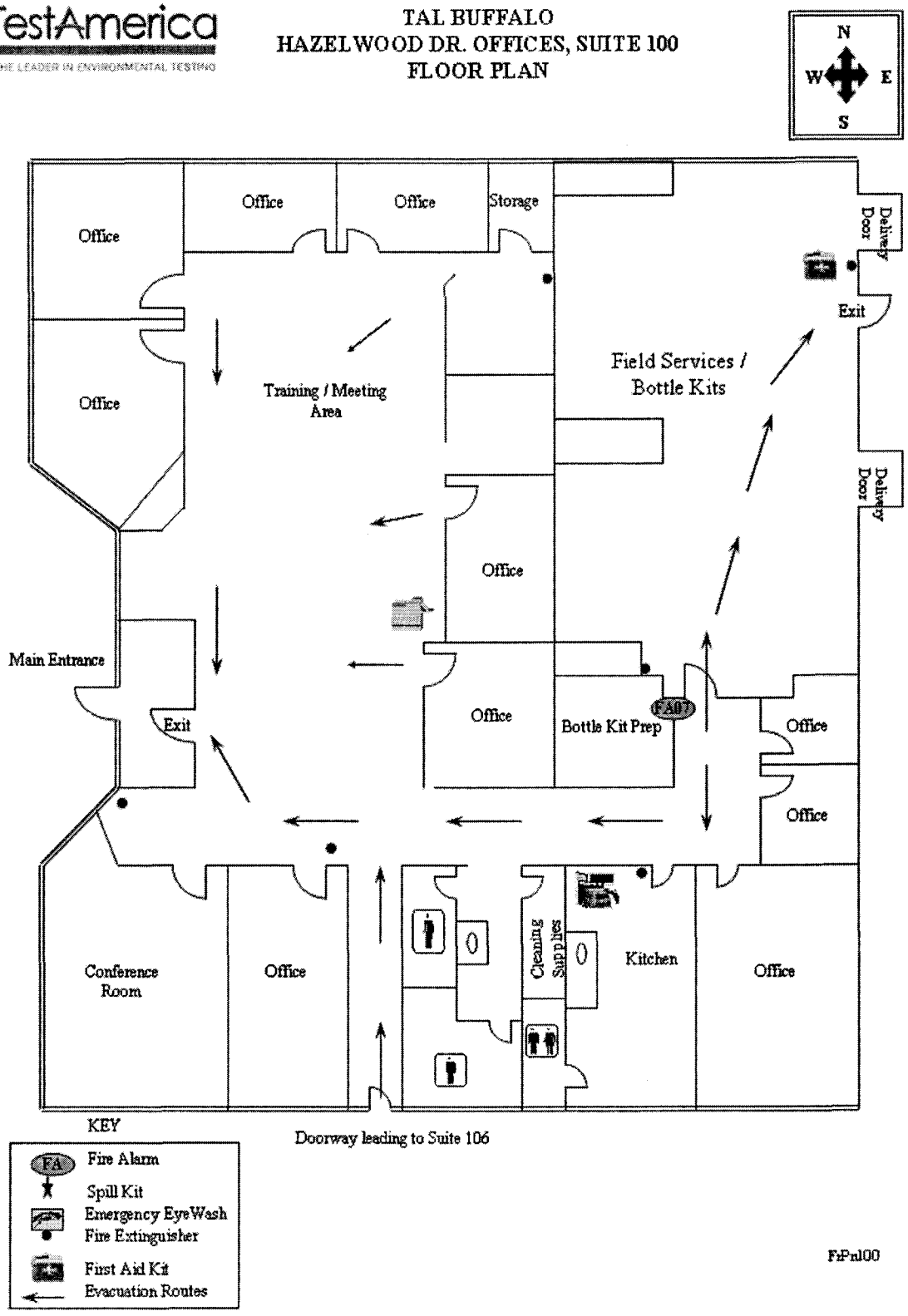
(The most current chart can be obtained from the QA Manager or Lab Director/Manager)



Appendix 3.

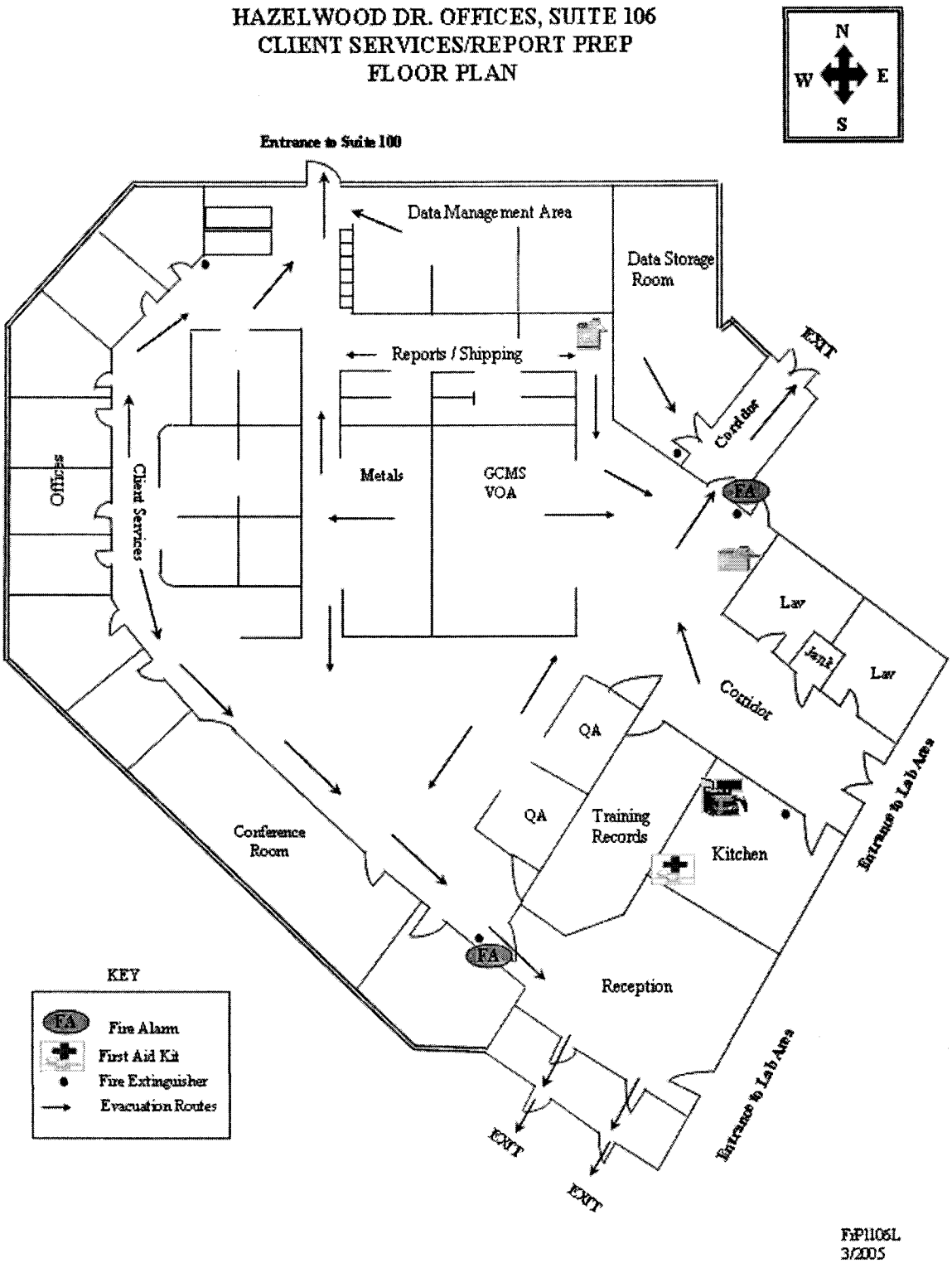
TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 100
FLOOR PLAN



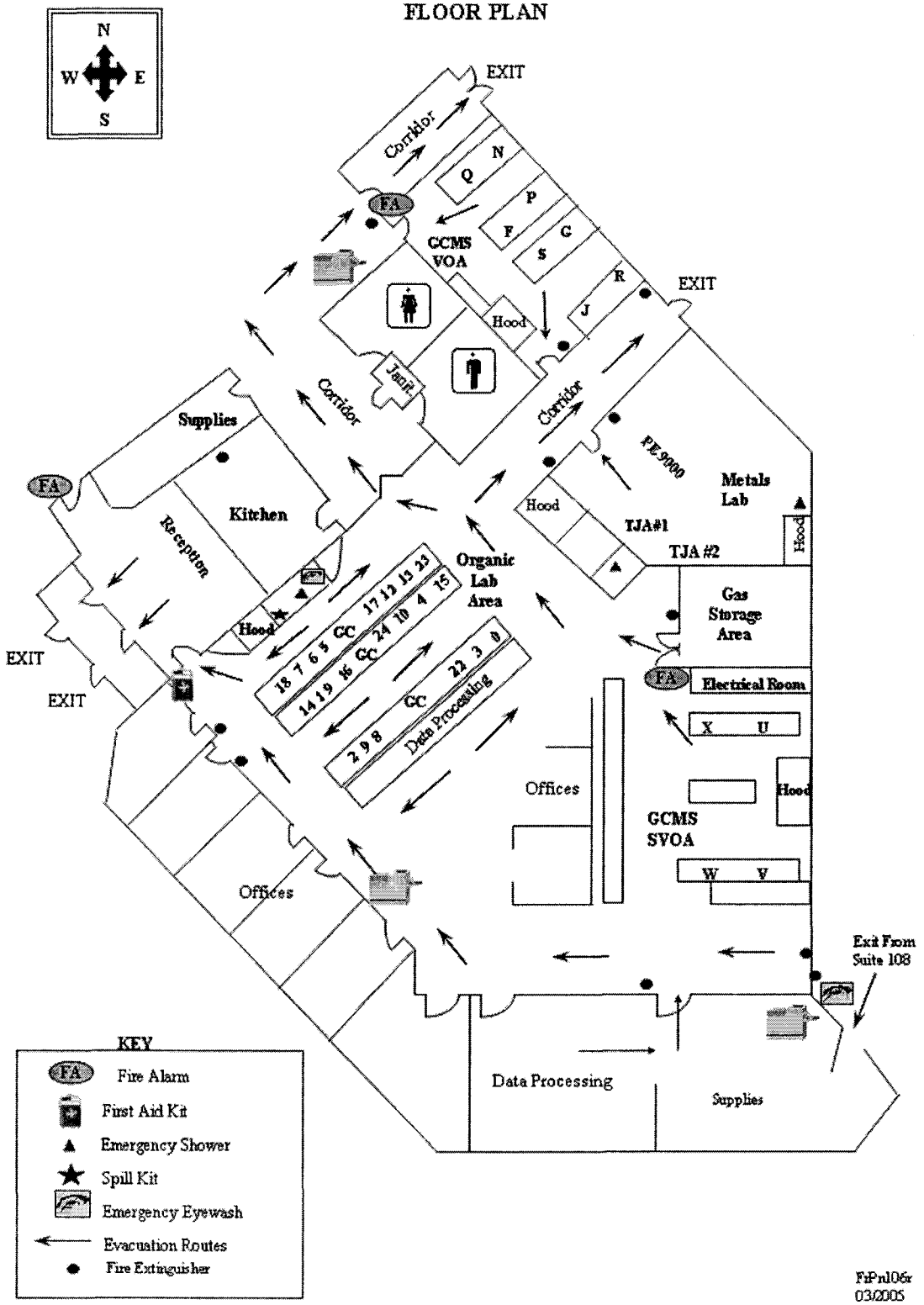
TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 106
CLIENT SERVICES/REPORT PREP
FLOOR PLAN



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL BUFFALO
HAZELWOOD DR. NY OFFICES, SUITE 106
LABORATORY AREA
FLOOR PLAN



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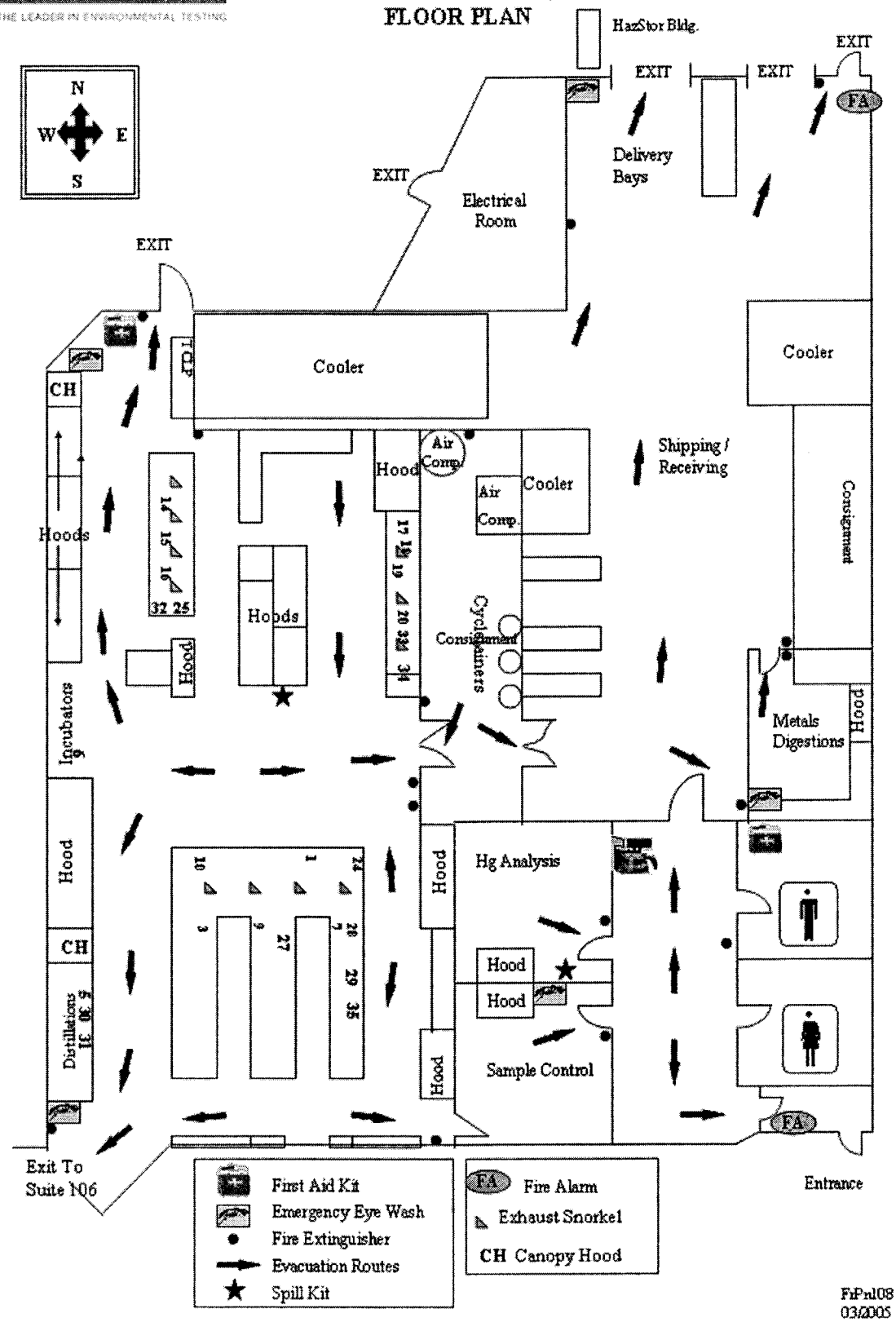
03/2005

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 108
FLOOR PLAN



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03/2005

Appendix 4: Reserved

Appendix 5. Glossary/Acronyms

Glossary:

Acceptance Criteria:
Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation:

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority:
The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

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. (QAMS)

Analyst:
The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Assessment:
The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

Assessment Criteria:
The measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

Assessment Team:
The group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (NELAC)

Assessor:
One who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (NELAC)
Audit:

A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

Batch:
Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank:
A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample:
A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration:
To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve:
The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method:
A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard:
A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):
A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

Chain of Custody:
An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

Clean Air Act:
The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):
The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

Compromised Samples:
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 a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

Confidential Business Information (CBI):
Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:
Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation
Alternate wavelength
Derivitization
Mass spectral interpretation
Alternative detectors or
Additional Cleanup procedures

(NELAC)

Conformance:
An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Corrective Action:
The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit:
A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction:
The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency:
An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Detection Limit:
The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Document Control:
The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses:
The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Environmental Detection Limit (EDL):
The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

Equipment Blank:
Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

External Standard Calibration:
Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA):
The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (NELAC)

Federal Water Pollution Control Act (Clean Water Act, CWA):
The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field Blank:
Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Testing:
NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

Finding:
An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (NELAC)

Holding Times (Maximum Allowable Holding Times):
The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Inspection:
An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Internal Standard:
A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Internal Standard Calibration:
Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank:
A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Response:
Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

Laboratory:
A defined facility performing environmental analyses in a controlled and scientific manner. (NELAC)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):
A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in

aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:
Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Least Squares Regression (1st Order Curve):
The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD):
An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Manager (however named):
The individual designed as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

Matrix:
The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

> Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.
>
> Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.
>
> Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
>
> Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample):

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):
A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

Method Blank:
A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit:
The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

National Environmental Laboratory Accreditation Conference (NELAC):
A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP):
The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Negative Control:
Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

NELAC Standards:
The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (NELAC)

Performance Audit:
The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS):
A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control:
Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision:
The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation:
Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing:
A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Proficiency Testing Program:
The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT):
A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Quality Assurance:
An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance [Project] Plan (QAPP):
A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control:
The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample:
An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual:
A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System:
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

Quantitation Limits:
The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range:
The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Reagent Blank (method reagent blank):
A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Reference Material:
A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method:
A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard:
A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Replicate Analyses:
The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Requirement:
Denotes a mandatory specification; often designated by the term "shall". (NELAC)

Resource Conservation and Recovery Act (RCRA):
The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

Safe Drinking Water Act (SDWA):
The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample Duplicate:
Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Second Order Polynomial Curve (Quadratic): The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity:
(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity:
The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike:
A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.. (NELAC)

Standard:
The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs):
A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM):
A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named):
The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate:
A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit):
A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director:
Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test:
A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method:
An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Toxic Substances Control Act (TSCA):
The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability:
The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

Uncertainty:
A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

United States Environmental Protection Agency (EPA):
The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation:
The process of substantiating specified performance criteria. (EPA-QAD)

Verification:
Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell:
A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Acronyms:

BS – Blank Spike
BSD – Blank Spike Duplicate
CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
CRS – Change Request Form
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DU – Duplicate
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
MDL – Method Detection Limit
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
NELAC - National Environmental Laboratory Accreditation Conference
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP: Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 6.

Laboratory Certifications, Accreditations, Validations

TestAmerica Buffalo maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

State	Program	Certification Number
Arkansas	SDWA, CWA, RCRA, SOIL	88-0686
California*	NELAP CWA, RCRA	01169CA
Connecticut	SDWA, CWA, RCRA, SOIL	PH-0568
Florida*	NELAP CWA, RCRA	E87672
Georgia*	SDWA,NELAP CWA, RCRA	956
Illinois*	NELAP SDWA, CWA, RCRA	200003
Iowa	SW/CS	374
Kansas*	NELAP SDWA, CWA, RCRA	E-10187
Kentucky	SDWA	90029
Kentucky UST	UST	30
Louisiana*	NELAP CWA, RCRA	2031
Maine	SDWA, CWA	NY0044
Maryland	SDWA	294
Massachusetts	SDWA, CWA	M-NY044
Michigan	SDWA	9937
Minnesota	SDWA,CWA, RCRA	036-999-337
New Hampshire*	NELAP SDWA, CWA	233701
New Jersey*	NELAP,SDWA, CWA, RCRA,	NY455
New York*	NELAP, AIR, SDWA, CWA, RCRA,CLP	10026
Oklahoma	CWA, RCRA	9421
Pennsylvania*	Registration, NELAP CWA,RCRA	68-00281
Tennessee	SDWA	02970
USDA	FOREIGN SOIL PERMIT	S-41579
USDOE	Department of Energy	DOECAP-STB
Virginia	SDWA	278
Washington	CWA,RCRA	C1677
West Virginia	CWA,RCRA	252
Wisconsin	CWA, RCRA	998310390

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, and in the QA office.

Claims of Accreditation Status

TestAmerica Buffalo has agreed to make only valid claims as to its accreditation/certification status by any authority by ensuring that the expiration dates are not exceeded and the method-specific scope or parameter lists are

supportable, as required by each. Any false claims would be reported to that authority. The agreement covers the use of the authority's name, such as "Authority-Accredited," logo, or certificate number. The only valid proof of accreditation/certification is the current certificate and scope of the authority. It is the responsibility of the laboratory to make these documents available to all staff, and it is the staff's duty to reference only the current documents.

A report with scope and non-scope analytes may only be presented on the same report if the non-accredited results are clearly and unambiguously identified. No report with non-scope analytes may be associated with the logo, "Authority accredited" phrase, or the certificate number. Only the analytes specified by a unique method are valid within the scope. There shall be no intentional misleading of the users of the laboratory's services in this regard.

No opinions and/or interpretations based on results outside the laboratory's scope may be presented on a document referenced by "Authority-accredited, the logo, or the certificate number. If these are made, they must be written in a separate letter which is not endorsed by the authority.

The "Authority-accredited" logo may only be affixed to equipment calibrated by a laboratory that is accredited by the authority. If calibration labels contain the logo, they must also show the calibration laboratory's name or its certificate number, the instrument's unique identification, the date of the last calibration, and a cross-reference to the last calibration certificate.

Should the company decide to use the "Authority-accredited" logo in marketing activities, no misrepresentation may occur. Only reference to the accredited scope at a specific laboratory site is allowed. If any "Authority-accredited" language is used in proposals or quotations, any non-scope analytes must be clearly denoted as not accredited by that authority. The same is true for any use of laboratory letterhead with the "Authority-accredited" wording or logo. The logo may not be affixed to any material, item, product, part, or packaging, thereby implying accreditation status to that piece. In literature, any use of the logo must be positioned adjacent to the accredited laboratory's name and clearly state that the presence of the logo does not imply certification/approval of the products tested. At no time may the logo appear to suggest that a person is accredited. Misrepresentation of accreditation status is never allowed and must be reported if it occurs. If in doubt, the idea of the logo's use may be presented to the authority for approval.

If accreditation is terminated or suspended, the laboratory will immediately cease to use the "Authority-accredited" wording, the logo, or the certificate number reference in any way and inform clients impacted by the change.

Appendix 7. Data Qualifiers

Qualifiers Organic	Footnote
A	Tentatively identified compound is a suspected aldol condensation.
U or ND	Compound analyzed but not detected.
E	Compound was over the calibration range.
D	Compound analyzed at a secondary dilution factor.
J	Compound detected but below the reporting limit (the value given is an estimate).
N	Identification of tentatively identified compound is based on a mass spectral library search.
B	Compound was detected in the method blank.
C	Mass spectral confirmation of compound.
P	The % difference between the results from both columns is >25% (CLP).
NC	The recovery and or RPD was not calculated.
*	Value outside QC limits.
1	Indicates co-elution
Qualifiers Inorganic	**Footnote**
B or J	The reported values is less than the Reporting Limit but greater than the Instrument Detection Limit (IDL) or Method Detection Limit (MDL).
E	The reported value is estimated because of the presence of interference
NC	The recovery was not calculated due to the concentration of analyte in the sample being >4 times the concentration of spike added.
N	Spiked sample recovery is not within control limits.
S	The reported value was determined by the Method of Standard Additions (MSA).
U or ND	The analyte was analyzed but not detected.
H	Indicates analytical holding time exceedance. Value should be considered estimated
*	Spike or Duplicate analysis and or RPD is not within control limits.
+	Correlation coefficient for the MSA is less than 0.995.