SCANNED

COLUMBIA CEMENT COMPANY, INC. 159 HANSE AVENUE FREEPORT, NEW YORK 11520

SITE # 1-30-052

SAMPLING AND ANALYSIS PLAN

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1.0 INTRODUCTION

This site-specific Sampling and Analysis Plan (SAP) has been prepared by Delaware Engineering, P.C. (Delaware) on behalf of Burmah Castrol Trading Limited (BC) for the Columbia Cement Company, Inc. (CCC) site (Site # 1-30-052) located at 159 Hanse Avenue in Freeport, New York. This SAP contains both a Field Sampling Plan (FSP) and a Quality Assurance Project Plan (QAPjP). It outlines the data quality objectives and details the specific sampling procedures and the relevant sampling and analytical protocols to ensure that the data collected during the remedial investigation (RI) are of sufficient quality to support remedial decisions.

1.1 SITE LOCATION, DESCRIPTION AND BACKGROUND

1.1.1 Site Location

The approximately two-acre Site is located in an extensively developed industrial and commercial area in the Village of Freeport in Nassau County, New York. The Site is situated approximately 4,000 feet south of the Sunrise Highway and 2,000 feet west of the Meadowbrook State Parkway. Locally, the Site is east and north of Hanse Avenue, south of Rider Place and west of Buffalo Avenue Extension (Figure 1).

1.1.2 Site Description

The Site slopes very gently from north to south; all elevations are greater than five feet and less than ten feet above mean sea level. A survey benchmark was established by Rust Environment & Infrastructure (Rust) on utility pole F34, located along the middle of the eastern property line, and an assumed datum of 100.00 was applied. The range of elevations encountered on the Site is from 97.63 to 99.07 feet.

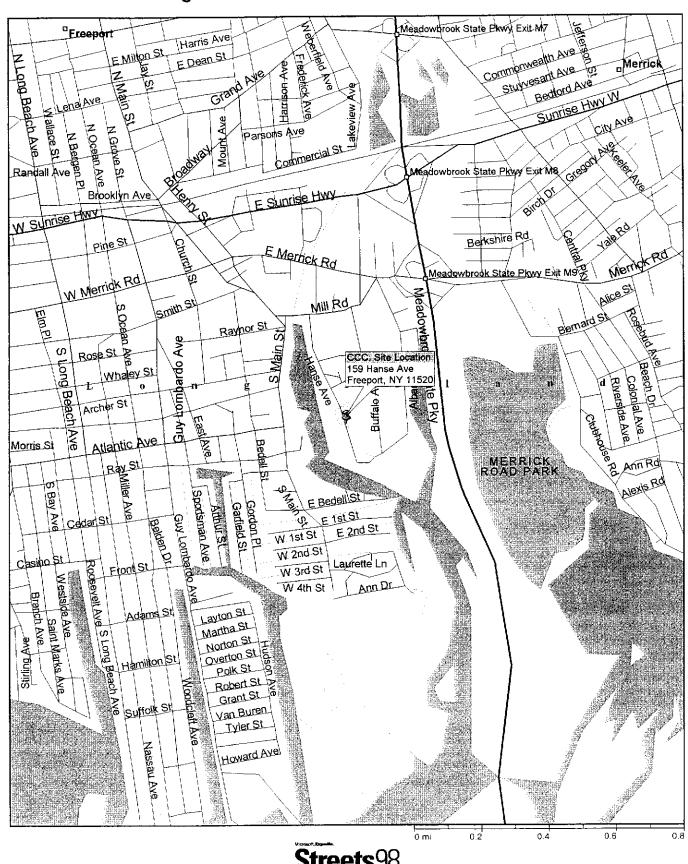
Industrial and commercial facilities bordering the Site include a Columbia Cement warehouse to the north, Lea Ronal Specialty Chemicals Worldwide (224-272 Buffalo Avenue Ext.) to the east, the Knickerbocker building to the south, and Farber Plastics (162 Hanse Avenue) to the west.

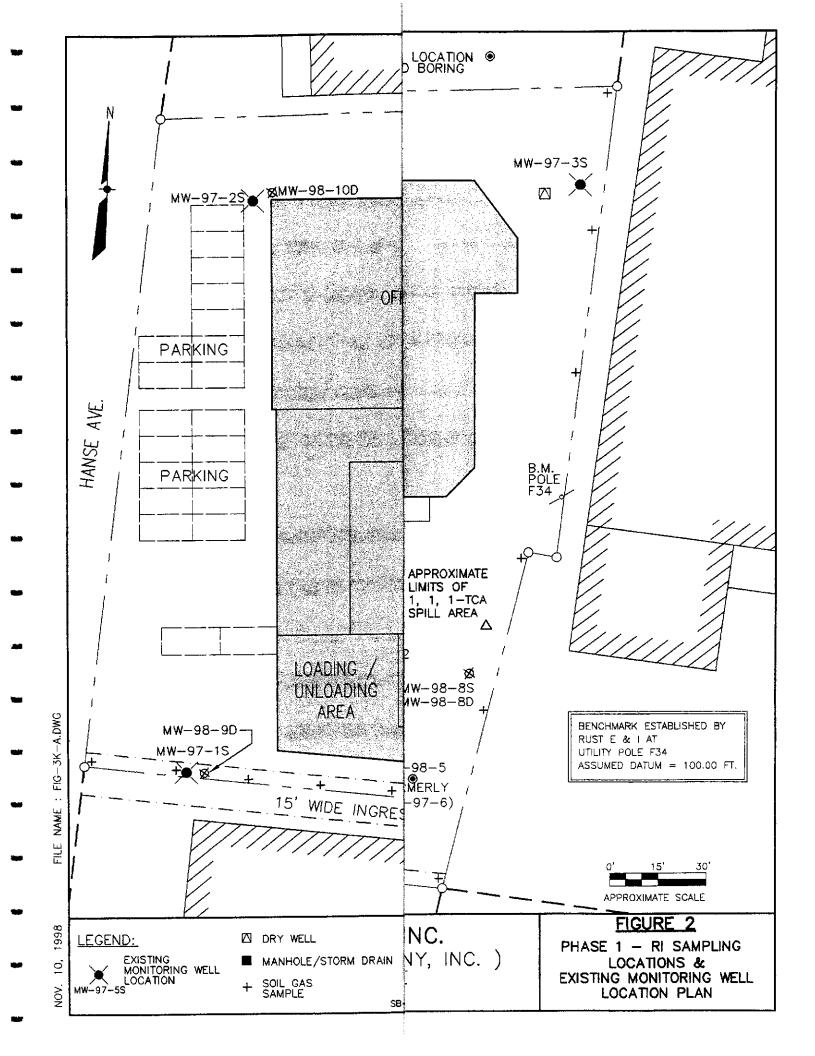
The Site is located approximately 500 feet east of the Freeport Creek, approximately 1,000 feet to the west of the Stadium Park Canal (also referred to as the Merrick River), and 4,000 feet to the northwest of Merrick Bay on the southeast shore of Long Island. The local storm water drainage system serving CCC discharges into Freeport Creek.

1.1.3 Site Background

CCC had manufactured various grades of contact cement and other industrial/commercial adhesives at this location since 1969. The main building is improved with office space, a mill room, two mixing rooms, two filling rooms, two storage rooms, a hazardous waste containment area, a small warehouse/reuse station, a temporary storage area and an unloading/loading area. Fifteen-foot wide ingress and egress easements are located along the northern and southern property boundaries. A parking lot for CCC employees is located west of the building. The southeastern portion of the Site

Figure 1 Columbia Cement Site Location





is paved and serves as an unloading and storage area for process chemicals. Ten 8,000-gallon underground storage tanks (USTs) are present in this area.

The Village of Freeport operated a municipal landfill in this prior to its development for commercial/industrial use. Representatives of the Village indicate that the land filling ceased in the 1960's and that development of this portion of Freeport began soon thereafter. The CCC operations are the first and only commercial/industrial activity at the Site since the land filling ceased.

Spill Incident

On April 28, 1988, Quadrel Brothers of Rahway, New Jersey, delivered approximately 3,500 gallons of 1,1,1-trichloroethane (1,1,1-TCA) to the Site. During delivery, the truck became over pressurized and the tanker buckled. As a result, TCA spilled onto the adjacent pavement. The New York State Department of Environmental Conservation (NYSDEC) was promptly notified, and emergency response to the spill incident was provided by the NYSDEC Region 1 Spill Response Unit.

Approximately 1,740 gallons of TCA remaining in the tank trailer was drained into 55 gallon drums while the approximately 1,760 gallons of spilled material flowed towards an on-Site dry well. As a result, some of the material entered a storm sewer outlet. The storm water sewerage system ultimately drains into Freeport Creek, roughly 1,000 feet to the northwest of the TCA Spill Area. Immediate clean-up activities consisted of: 1) Removing liquid and approximately ten yards of soil from the noted dry well and 2) Removing liquid material from the Village of Freeport storm drain system. The drainage system was purged until sampling results showed concentrations of TCA below 50 parts per billion (ppb). At this time, three exploratory soil borings were advanced and one shallow overburden monitoring well was installed. Split spoon samples taken from two of the borings revealed TCA concentrations in soil ranging from 67 to 42,649 parts per million (ppm).

Following the TCA Spill, a new (northern) underground tank farm, comprised of five 8000-gallon tanks, was installed to the north of the aforementioned dry well/storm water basin. After completion of the northern underground tank farm installation, a concrete pad was installed over both the northern and southern tank farms. In February of 1989, the concrete pad was removed and replaced with a thicker concrete pad because of cracking noted in the original pad.

1.2 SCOPE OF WORK

As discussed in Section 2.0 of the Phase I Remedial Investigation/Feasibility Study Work Plan, the proposed scope of work for the remedial investigation includes the following activities:

• Drill three borings near the spill area (SB-98-1, SB-98-3, SB-98-4), one in the storm drain located in the spill area (SB-98-2) and one (SB-98-5) in the dry well located south of the spill area, and collect soil samples for VOC field screening and laboratory analysis;

- Drill and install one shallow and one deep monitoring well to the east of the spill area, including the collection of continuous split spoon soil samples, field photoionization (PID) screening of all samples and laboratory analysis of selected soil samples;
- Drill and install two deep monitoring wells, one at the west-southwest corner of the site adjacent to existing shallow monitoring well MW-97-1S and one a the westnorthwest corner adjacent to MW-97-2S. Collect continuous split spoon soil samples with field PID screening and laboratory analysis of selected soil samples;
- Measurement of groundwater levels in all on-site monitoring wells (at least two separate events) during different seasons of the year than the existing data;
- Development of the four new monitoring wells;
- Perform in-situ hydraulic conductivity testing in the four new monitoring wells;
- Collect groundwater samples from all thirteen on-site monitoring wells for laboratory analysis;
- Field PID screening of sediments in all on-site dry wells and collection and laboratory analysis of sediment samples. Also, confirm dry well discharge (i.e., are the dry wells interconnected) and storm water discharge points;
- Drill a shallow soil boring (SB-98-6) in the vicinity of former boring TB-97-6 for field PID screening and laboratory analysis;
- Drill a soil boring (BSB-98-7) in a background location to be selected by Delaware Engineering and NYSDEC personnel. Collection and laboratory analysis of background soil boring samples; and,
- Collection of vadose zone soil gas samples with field PID screening and laboratory analysis of twenty percent of the samples. Collection and analysis of a background ambient air sample.

2.0 FIELD SAMPLING PLAN

This section outlines the procedures that will be used in the collection of soil gas, soil/sediment, and groundwater samples and the parameters for which each sample will be analyzed. A summary of the samples to be collected and the required analyses and necessary quality assurance/quality control (QA/QC) samples is provided in Table 1. The specific field tasks are listed below:

- Soil gas sample collection and screening with a photoionization detector (PID);
- Dry well surface/shallow subsurface soil sampling;
- Split-spoon soil sample PID screening and sample collection;
- Groundwater monitoring well installation;
- Groundwater monitoring well development;
- Water level monitoring;
- · Groundwater purging and sample collection; and
- Groundwater monitoring well hydraulic conductivity testing.

2.1 SOIL GAS SURVEY

Subsurface soil gas samples will be collected along the eastern and southern property boundaries by constructing a narrow bore hole which will allow for the active withdrawal of soil gas from an interval of up to four feet below the ground surface. Sample bore holes will be prepared by using a slam bar to drive a %" steel rod to a maximum of depth of four feet, removing it and inserting a ½" diameter hollow aluminum tube into the probe hole to maintain the opening in the shallow vadose zone. Care will be taken to ensure that the tube is not plugged or inserted into shallow groundwater. Following placement of the aluminum tube, surface soil or a bentonite plug will be packed into the annular space around the top of the probe to minimize the potential infiltration of surface air during sampling.

Soil gas samples will be collected in Tedlar® bags using dedicated polyethylene tubing, a vacuum desiccator and a high volume laboratory-style vacuum pump. Both Tedlar and polyethylene are considered chemically-inert materials which will not react with or alter the composition of the samples collected. The vacuum pump will withdraw soil gas up through the subsurface probe at a rate of approximately three liters per minute (3 L/min).

The pump will be operated until approximately two liters (*i.e.*, six sampling train volumes) have been evacuated from the sampling train, at which point two soil gas samples from each location will be collected by filling Tedlar bags. One of the bags will then be screened for total volatiles using a PID equipped with an 11.7 electron volt (eV) lamp (*e.g.*, the Photovac 2020 PID or equivalent). The second bag will be labeled with the sample location and carefully stored in a cool dark place. A total of four samples will be selected for laboratory gas chromatography/mass spectroscopy (GC/MS) analysis of the NYSDEC Analytical Services Protocol (ASP) Target Compound List (TCL) volatile organic compounds (VOC).

There will be a total of five soil gas sampling locations along the eastern property boundary; one

Table 1

Phase I Remedial Investigation - Sample and Analysis Summary Columbia Cement Company, Inc. Facility Freeport, New York

	Number of		umber of Q	Number of QA/QC Samples	S	Total Number	,
Media	Samples	MS/MSD1	Field Dup ²	Field Dup ² Equip Blank ³ Trip Blank ⁴	Trip Blank	of Samples	Analysis
Soil Gas							
Laboratory confirmation samples	5					5	VOC's, 95-1 (modified)
Soil							
* *Spill Area Soil Borings							
SB-98-01, SB-98-02, SB-98-03, SB-98-04	12			,		13	VOC's, 95-1 and TOC
Note: Two samples for full TCL/TAL, highest PID reading from SB-98-02	2	7	_	_		9	Full TCL/TAL and TOC
and highest reading from SB-98-01 or SB-98-03.							
Additional Soil Boring (in the vicinity of boring TB-97-6)							
SB-98-5							VOC's, 95-1
Monitoring Well Installation Soil Borings							
Three deep monitoring wells (three samples each boring)	6					6	VOC's, 95-1 and 1OC
Note: highest PID reading for MWSB-98-8D Full TCL/TAL	-					_	Full TCL/TAL and TOC
Dry Well Hand Auger Soil Borings							
dry well immediately to the west of MW-97-6S	2	2			<u> </u>	55	VOC's, 95-1
Background Soil Boring							č
BSB-98-7	Е					3	Full TCL/TAL, TOC"
Groundwater							
** Groundwater Monitoring Wells	-						
all on Site monitoring wells with the exception of those listed below	6				က	12	VOC's, 95-1
MW-1S, MW-1D-97, MW-98-8S and MW-98-8D	ব	2	,4			۲-	Full TCL/FAL

- 1) QA/QC samples will include a matrix spike (MS) and mutrix spike duplicate (MSD) sample at a frequency of not less than 5% (one MS/MSD pair per every 20 samples collected) for each matrix (aqueous and soil).
 - 2) A blind field duplicate sample will be collected at a frequency of one per every 20 samples for each matrix (aqueous and soil).
- 3) Equipment blanks are not required when dedicated sampling equipment is used. If non-dedicated sampling equipment blanks will be analyzed at a frequency of not less than 5% (one equipment blank per every 20 samples collected).
 - 4) One trip blank will submitted for analysis for each day aqueous matrix volatile organic samples are collected. A trip blank will be included in each cooler that contains aqueous matrix volatile organic samples; therefore all volatile organic samples and containers will be shipped to and from the laboratory in the smallest number of coolers possible in order to minimize the number of trip blanks required.
- 5) All samples will be analyzed using NYSDEC ASP (10/95) analytical procedures for Superfund-CLP Volatile Organics (NYSDEC Method 95-1), Semivolatile Organics (NYSDEC Method 95-2), Pesticides/Aroclors (NYSDEC Method 95-3) and/or the appropriate Analytical Methods for CLP Inorganics, as necessary.
 - spectroscopy (GC/MS) method at the laboratory. The target compound list will be the same as that listed in NYSDEC ASP Method 95-1.

 7) The limited list of volatile organic compounds to be analyzed for using NYSDEC ASP Method 95-1 are as follows: 1,1,1-Trichloroethane; Chloromethane; 1,1-Dichloroethane; 6) A total of five soil gas samples in Tedlar bags for volatile organic analysis by an acceptable gas chromatography/mass.
 - 1,1-Dichloroethene; and Vinyl Chloride.
- 8) The analytical laboratory contracted to perform the sample analyses will be a New York State Department of Health (NYSDOH), Environmental Laboratory Approval Program (ELAP) certified laboratory holding the Analytical Services Protocol (ASP) certification.
 - 9) This sample will not be submitted for analysis if the PID reading is not elevated with respect to background PID readings.
- 10) These samples will be submitted for Full TCL/IAL, and total organic carbon (TOC) analysis.
- 11) * Three samples from each of the four Spill Area soil borings analyzed for VOC's (95-1), except two samples for Full TCL/FAL, one highest PID reading from SB-98-02 and one highest PID reading from either SB-98-01 or SB-98-03 for full TAL/ICL analysis.
 ** All monitoring well samples analyzed for VOCs (95-1) except for MW-1S, MW-1B-97, MW-98-8S and MW-98-8D for full TAL/ICL analysis.

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approximately every 50 linear feet. A total of 12 soil gas sampling points will be located along the southern property boundary: One in the southeast corner of the property, two spaced approximately 50 feet apart heading west along the property boundary, and then nine more spaced approximately 25 feet apart until the southwest corner of the property is reached. (The reduced spacing along the southwest portion of the property is in response use of the adjacent building as office space.) The two samples from those spaced approximately 50' apart exhibiting the highest PID results will be submitted for volatile analysis as described above; likewise, the two samples from those spaced approximately 25' apart exhibiting the highest PID results will also be submitted for analysis.

Background, on-Site air samples will be collected and screened for total volatiles using a PID (as described above) at a minimum frequency of 5% (*i.e.*, one per every twenty samples) throughout the soil gas sampling survey. Background samples consist of ambient air collected through the sampling equipment in the same manner as soil gas samples and are essentially the equivalent of field blanks. At a minimum, background samples will be collected and analyzed prior to the initiation of the soil gas sampling program and when the distance between sampling points is reduced from approximately every 50 linear feet to 25 feet. One background sample will be submitted for laboratory GC/MS analysis of the NYSDEC TCL VOCs.

Blind field duplicate samples will be collected at a minimum frequency of 5% (i.e., one per every twenty samples) by collecting soil gas from the same borehole in two separate Tedlar bags, the second immediately following the collection of the first.

The %" steel rod will be decontaminated between sampling locations as described in Section 3.1.4. Each aluminum tube will be cleaned prior to mobilization and will be dedicated to only one sampling location. The polyethylene tubing will be discarded and replaced for each new sampling location.

2.2 DRY WELL SURFACE/SHALLOW SUBSURFACE SOIL SAMPLING

With the exception of the dry well immediately south of the spill area, Delaware personnel will collect surface/shallow subsurface soil samples from all on-site dry wells using a hand auger, as described in Section 2.2.1 below. Samples will be collected from the sediment surface to two and one-half feet or refusal, whichever occurs first. The material collected from every six inches will be screened in the field for total volatiles using a PID equipped with an 11.7 eV lamp. The sample from zero to six inches and the sample exhibiting the highest PID reading will both be submitted to the laboratory for the analysis of 1,1,1-TCA and its potential breakdown products (*i.e.*, chloromethane, 1,1-dichloroethane, 1,1-dichloroethene and vinyl chloride). Both of the samples collected from the dry well immediately to the west of monitoring well MW-97-6S will be submitted to the laboratory for NYSDEC TCL volatile organics analysis using NYSDEC ASP Method 95-1. The dry well immediately south of the spill area will be sampled as described below in Section 2.3.

2.2.1 Dry Well Soil Sampling with a Hand Auger

Collecting surface soil samples with a stainless steel trowel/spatula and/or shovel is most appropriate for shallow samples collected to a depth of 0-6 inches. Stainless steel or other chemically inert materials (e.g., nylon/PVC) are required. Shallow subsurface soil samples (less than five feet) are

typically collected using hand augers (bucket type) constructed of stainless steel. The depth to which samples can be collected is dependent on soil conditions. Because these samples will be collected from the base of the dry wells, the samples will be collected using a hand auger. The sampling procedures to be used are detailed below.

- Advance the stainless steel pre-cleaned hand auger into the soil until the bucket is full (approximately 6 inches).
- Remove the sample from the bucket auger with a pre-cleaned trowel, spatula, lab spoon or equivalent and place onto clean plastic sheeting.
- Screen each sample with a PID and collect the VOA sample at a discrete interval.
- Repeat steps 1 4 until the desired depth is reached.
- Collect samples for VOC analysis as discrete grab samples without mixing in the field.
- Secure a Teflon lined cap onto the jar(s) and carefully/clearly label the sample containers.
- Place the sample container(s) on ice in a cooler for transport to the laboratory. Complete all chain-of-custody documents and record in the field log book.
- Decontaminate all non-dedicated sampling equipment after use and/or between sample locations as defined in the QAPjP.

2.3 SPILL AREA DRY WELL SOIL BORING

Soil boring SB-98-5 will be drilled in the dry well located south of the spill area, with the boring being advanced to the top of the gray silt and clay unit. Continuous two-foot split spoon samples will be collected from the base of the dry well to this unit following American Society for Testing Materials (ASTM) Method D-1586 as outlined below in Section 2.3.1. The samples will be characterized and logged, and headspace screening of each soil sample will be performed with a PID equipped with an 11.7 eV lamp in order to identify potentially contaminated zones within the soil column.

The soil sample in the upper fill that exhibits the highest field PID reading will be submitted for laboratory analysis for NYSDEC TCL VOCs in accordance with NYSDEC ASP Method 95-1. If no elevated PID readings are detected, then the sample from the base of the dry well will be submitted for analysis. In addition, a sample from both the top of the peat layer and from the top of the gray silt and clay layer will also be submitted for laboratory analysis.

Upon completion, the soil boring will be properly sealed with a cement/bentonite grout to prevent it from serving as a conduit for the downward migration of contaminants in the future. The boring will be sealed by pumping a cement-bentonite grout mixture directly to the bottom of the hollow stem augers using a tremie pipe until grout returns to the surface. The augers will then be extracted incrementally and the grout will be topped off. The grout material will consist of Type I Portland cement mixed with powdered bentonite, prepared using 6 to 7 gallons of water and 3.5 to 4.0 pounds of powdered bentonite for each 94-pound bag of cement.

2.3.1 Split Spoon Sampling Procedures and PID Headspace Screening

ASTM Method D-1586, Standard Method for Penetration Test and Split Barrel Sampling of Soils, will be used to obtain representative samples for characterization, laboratory testing, and as a measure of the resistance of soil to sampler penetration. A summary of both the sampling procedure and the PID screening method are presented below:

- Ensure that all down-hole-drilling tools (*i.e.*, split-spoons, drilling rods, and augers) have been properly decontaminated following the procedures detailed in the QAPjP prior to advancement of the borehole.
- Measure the sampling equipment lengths and openings to ensure that they conform to specifications. Confirm the weight of the hammer (140 lbs.) verbally or by visual means.
- Lower the sampler to the desired depth at the bottom of the auger column and check the depth against length of the rods and the sampler.
- Attach the drive head sub and hammer to the drill rods without the weight resting on the rods.
- Lower the weight and allow the sampler to settle up to 6 inches. If it settles more, consider using another sampler.
- Mark four 6-inch intervals on the drill rods relative to a drive reference point on the rig. With the sampler resting on the bottom of the hole, drive the sampler with the 140 lb. hammer falling freely over its 30 inch fall until 24 inches have been penetrated or 100 blows applied for 6 inches.
- Record the number of blows per 6 inches. Determine the "N" value by adding the blows for the middle 12 inches.
- Upon retrieval, open the split spoon and screen with a PID. Quickly log the splits contents, and
 then immediately transfer a subsample into the proper container to be analyzed for volatile
 organic compounds. Place a second subsample into a clean glass jar and seal with aluminum foil
 for subsequent PID screening. Allow this subsample to equilibrate for fifteen to thirty minutes,
 then puncture the aluminum foil cap with the tip of the PID probe and record the maximum
 instrument reading.
- Homogenize the remaining material from the desired depth interval using a limited cone and quarter procedure, which involves continual mixing of the sample into the shape of a cone. Remove large non-analyzable materials (e.g., rocks, twigs, etc.) that fall to the base of the cone by gravity. Upon completion of coning, divide the sample into four quarters of equal size, and alternately remove and place equal amounts from each quarter in appropriate sample containers until they are filled.
- Secure a Teflon lined cap onto the jar(s) and carefully/clearly label the sample containers.
- Place the sample container(s) on ice in a cooler for transport to the laboratory. Complete all chain-of-custody documents and record in the field log book.
- Document all sample properties, depths and locations on the boring log form.
- Label and store recovered samples (excluding those reserved for chemical analysis) until the project has been completed, at which time the samples will be properly disposed of.
- Decontaminate all non-dedicated sampling equipment after use and/or between sample locations as defined in the QAPjP.

2.4 ADDITIONAL SPILL AREA SOIL BORINGS

A total of four additional soil borings (SB-98-01, SB-98-02, SB-98-03 and SB-98-04) will be drilled near the spill area, including one in the storm drain located in the spill area (SB-98-02). A boring (BSB-98-7) will also be drilled at a background location that will be selected by Delaware Engineering and NYSDEC personnel. Each boring will be advanced to the top of the gray silt and clay unit and continuous two-foot split spoon samples will be collected from the ground surface to this unit. During drilling in the spill area, soils will be continuously sampled with split spoons following ASTM Method D-1586 as outlined previously in Section 2.3.1. The samples will be characterized and logged, and headspace screening of each soil sample will be performed with a PID equipped with an 11.7 eV lamp in order to identify potentially contaminated zones within the soil column.

From each boring, except background boring BSB-98-7, the soil sample in the upper fill that exhibits the highest field PID reading will be submitted for laboratory analysis for NYSDEC TCL volatile organics in accordance with NYSDEC ASP Method 95-1. If no elevated PID readings are detected, then a sample at the groundwater interface or the center of the fill area (if groundwater is not encountered within the fill) will be submitted for analysis. In addition, a sample from both the top of the peat layer and from the top of the gray silt and clay layer from each boring will also be submitted for laboratory analysis.

The single sample exhibiting the highest PID reading from borings SB-98-01 and SB-98-03 will be analyzed for the full NYSDEC ASP TCL for organics and the Target Analyte List (TAL) by NYSDEC, ASP, CLP methods. In background boring BSB-98-7, a sample from the center of the fill layer, the top of the peat layer and the top of the gray silt and clay layer will be collected and submitted for laboratory VOC (method 95-1), TAL metals and total organic carbon analysis.

Upon completion, each soil boring will be properly sealed with a cement/bentonite grout as discussed previously to prevent them from serving as a conduit for the downward migration of contaminants in the future.

2.5 ADDITIONAL SOIL BORING (IN THE VICINITY OF BORING TB-97-6)

An additional soil boring (SB-98-06) will be drilled in the vicinity of former test boring TB-97-6, with this boring being advanced to a total depth of fourteen feet (14'). Continuous two-foot split spoon samples will be collected from the ground surface to this depth. As before, soils will be continuously sampled with split spoons during drilling, following ASTM Method D-1586 (outlined previously in Section 2.3.1). The samples will be characterized and logged, and headspace screening of each soil sample will be performed with a PID equipped with an 11.7 eV lamp in order to identify potentially contaminated zones within the soil column.

The soil sample which exhibits the highest field PID reading will be submitted for NYSDEC ASP Method 95-1 laboratory analysis for TCL volatile organics. If no elevated PID readings are detected, then the sample from eight to ten feet will be submitted for analysis.

Upon completion, the soil boring will be properly sealed with a cement/bentonite grout as discussed previously to prevent it from serving as a conduit for the downward migration of contaminants in the future.

2.6 GROUNDWATER MONITORING WELL INSTALLATION, DEVELOPMENT AND HYDRAULIC CONDUCTIVITY TESTING

One shallow/deep monitoring well pair will be installed east of the spill area. One deep monitoring well will be installed at the west/southwest corner of the Site while a second will be installed at the west/northwest corner of the Site.

The shallow well will be designed so that the well screen will intercept the water table. The length of the screen will be sufficient to span the expected range of seasonal water table fluctuation. The screen for each of the deep wells will be installed at the top of the clay unit within the unconsolidated gravelly sand unit. Well screens will be ten feet in length unless field conditions require modification of the screen length.

During installation of the deep wells, soils will be continuously sampled with split spoons following ASTM Method D-1586 for soils (as outlined in Section 2.3.1). Headspace screening of each soil sample will be performed with a PID equipped with an 11.7 electron volt (eV) lamp in order to identify potentially contaminated zones within the soil column. Three sub-surface soil samples will be collected and submitted to a laboratory for VOC analysis (NYSDEC method 95-1). The sample with the highest field PID reading will be submitted as will a sample from the top of the peat layer and a sample from the top of the gray silt and clay layer. The soil sample from boring MW-98-8D that exhibits the highest PID reading will be analyzed for the NYSDEC TCL/TAL parameters (NYSDEC CLP methods). If no PID readings above background are detected then only the samples from the top of the peat layer and the sample from the top of the gray silt and clay layer will be submitted for laboratory analysis.

2.6.1 Monitoring Well Boring/ Well Installation Procedures

The following is a summary of the required protocol for the construction of all monitoring wells:

- The well screen will be 2-inch diameter, machine slot (0.010") PVC for all monitoring wells. Riser material will be 2-inch flush threaded PVC. All threads will be secured using Teflon tape.
- All monitoring wells will be constructed by first advancing a minimum eight-inch outside diameter (O.D.) borehole to one foot below the top of the tidal marsh (e.g., peat) layer. The hollow stem augers will be left in place and a cement-bentonite mixture tremied into the augers. The augers will be subsequently withdrawn, and a length of four inch I.D. Schedule 40 PVC pipe sufficient to leave approximately 2 feet of stick-up will be inserted to the bottom of the borehole. The grout mixture will be removed from within the Schedule 40 PVC pipe by tremie grouting a bentonite slurry into the pipe, thus displacing the cement-

bentonite mixture. After allowing the grout to set overnight, the remainder of the monitoring well borehole will be advanced utilizing a nominal 4-inch diameter casing advanced with drive and wash methods. If bridging of sands is encountered an alternative drilling method, such as conventional mud rotary employing a tricone roller will be utilized;

- Prior to flush mount monitoring well construction, the four-inch inside diameter schedule 40 PVC casing will be cut to approximately 0.75 feet below grade. Sand will be introduced gradually inside the casing, and will fill the annular space between the well screen and adjacent casing. The sand pack will extend from the bottom of the boring to approximately two feet above the top of the screen. During placement of the sand pack, casing will be withdrawn in increments so that the formation materials do not collapse against the well casing and/or screen. The sand pack will consist of clean, graded, silica sand with grain size distribution matched to the slot-size of the screen; i.e., a Unimin™ Grade 0 or equivalent sand. A six-inch layer of clean Unimin™ Grade 00 sand will then be placed above the sand pack to preclude migration of sealing material into the sand pack. A bentonite pellet seal will be placed above the sand pack to form a seal at least two-feet thick. Cement-bentonite grout will be placed from the top of the bentonite pellet seal to approximately three feet below grade;
- The top of the Schedule 40 PVC riser with end cap will be approximately 3" below grade, and the wells will all be flush mounted and even with the existing grade. A vent hole will be drilled near the top of the PVC riser pipe.

Following monitoring well installation, each well will be developed as described in Section 2.3.3 to remove silt and clay particles from the sandpack and well screen, and to improve communication of the well with the surrounding aquifer.

Following the completion of well development activities, slug testing of each well will be performed as described in Section 2.3.4 in order to measure the hydraulic conductivity of the formation in which the well is screened.

2.6.2 Well Development Procedures

Monitoring wells are generally developed following installation in order to improve the hydraulic properties of the sand pack and to remove any sediment within the well and adjacent to the screen/sand pack. Well development should continue until the water is relatively sediment free and the turbidity is less than 50 NTU (if possible). If the 50 NTU goal cannot be achieved, well development will continue until 10 well volumes have been removed.

The procedure may be summarized as follows:

• Select an appropriate well development method depending on water level depth, well productivity, and sediment content of the water. Well development options include:

bailing; surging; manual pumping; inertial pumping; powered suction-life pumping; and air-lift development.

- Decontaminate all non-dedicated equipment prior to use and between wells following the procedures detailed in the QAPjP.
- Assemble and install the equipment in the monitoring well. Take appropriate precautions to
 prevent the introduction of contaminants on the equipment during installation. Assemble and
 operate pumps in accordance with manufacturer's instructions.
- Repeatedly remove water from the well until the discharged water is relatively sediment free.
- Monitor the effectiveness of development at regular intervals (after each well volume is removed) by measuring and recording the pH, conductivity, turbidity and temperature values during development. Record all measurements versus well volumes removed.
- Discontinue well development when the turbidity of the discharged water reaches 50 NTU or
 when the pH, conductivity, turbidity and temperature values stabilize, indicating that formation
 water is being removed and that additional purging will most likely not reduce the turbidity level
 in the water column. A minimum of 10 well volumes will be removed.
- Handle development waters by containerizing for later off-site disposal or disposal elsewhere onsite depending on groundwater analytical data.

2.6.3 Hydraulic Conductivity Testing Procedures

Hydraulic conductivity (K) tests, also called slug or bail tests, are performed on monitoring wells to determine the in-situ hydraulic conductivity of the hydrostratigraphic unit screened. Slug and bail tests involve observing the recovery of water levels toward an equilibrium level after an initial perturbation. Procedures and equipment requirements are expected to vary depending on the rapidness of the water level response.

2.6.3.1 FOR USE WITH IN-SITU, INC. HERMIT MODEL SE 1000C DATA LOGGER AND PRESSURE TRANSDUCER

- Record appropriate initial data in field notebook, including date of test, well identification, well construction details (*i.e.*, screen length, screen diameter, riser diameter and screen depth), type of test and field personnel.
- Clean the slug, line and pressure transducer and cable following the decontamination procedures detailed in the QAPjP prior to initiating the tests at each well.
- Measure and record the static water level in the monitoring well as outlined in Section 2.3.5; only
 monitoring wells which have fully recovered to static level conditions should be tested. The
 depth to water should be recorded as the depth below the measuring point (bmp).
- If using a Hermit data logger, connect the pressure transducer to the data logger and lower the transducer into the well four to ten feet below the water surface. If using a solid slug or a bailer,

then the transducer should be set ten feet below the water table. If using a distilled water slug, the transducer should be set four to five feet below the water table. It is important that the pressure transducer not be subjected to pressures greater than the pressure transducer rating. Secure the position of the transducer by clamping the transducer cable to the well casing using a rubber covered clamp. If the edges of the well casing are sharp, cover them with cloth or duct tape to protect the transducer cable.

- During the slug test, either a 5-foot aluminum rod or a known volume of deionized water will be quickly introduced into the well casing to cause the water level to rise in the well. During the bail tests, the same 5-foot aluminum rod or a dedicated pre-cleaned PVC bailer will be rapidly removed from the well to lower the water.
- Set up the data logger. Check the battery and enter the coefficients appropriate for the transducer selected (scale, offset, linearity, coefficient, delay, and number of transducer).
- Enter the test data, select the test number, check the clock, and set the reference level (stable water level).
- To initiate test, depress START key and presses ENTER. To view data from the ongoing test, depress the DATA key.
- Watch the data logger to determine if well recovery is reasonable. If so, allow the test to run until well recovery returns to baseline and then stop the test.
- If another test is to be performed, replace the bailer or solid object and let the well re-equilibrate or allow the well to re-equilibrate prior to introducing the next slug. Change the settings as necessary and repeat the test.

2.6.3.2 MANUAL COLLECTION OF TEST DATA

Hydraulic conductivity test data may also be collected manually with the use of a water level measuring device. This method is less desirable than using a data logger because initial test data is often missed and it is difficult to collect closely spaced measurements in a formation which recovers rapidly. However, manual data collection may be the preferred method for very slowly recovering formations, where hour measurement intervals are appropriate. The following methods shall be used when performing manual collection of K-test data:

- Establish a schedule according to which water levels will be measured. Measurement intervals should be close during the initial potion of the test and increase over time. Create a table in the field notebook to record times and water level measurements.
- Prepare the electronic water level meter so that a measurement can be immediately taken after the slug or bail is initiated.
- Perform the slug or bail as described above.
- Collect water level measurements following the established schedule. Continue taking measurements until the well has fully recovered or measurements do not appear to be changing significantly over time.

2.6.4 Water Level Measurement Procedures

The following procedure is used to measure water levels using an electronic water level detector:

- Decontaminate the water level probe and the portion of cable that enters the water column with a non-phosphate detergent wash followed by a distilled water rinse. Dry using clean paper towels.
- Test the water level meter to ensure that the batteries are charged and that it is operating properly.
- If necessary, place plastic sheeting on the ground around the well to ensure that the measuring equipment doesn't contact the ground.
- Unlock the protective casing locking cap (if present), remove the monitoring well cap and place it on the plastic.
- Lower the probe into the well slowly until the alarm indicates that water has been reached.
- Read the depth to water from the graduated cable using the surveyed and marked measuring point
 on the monitoring well casing. This measurement should be made to the nearest 100th of a foot.
- Confirm the measurement by repeating the reading.
- Record the depth to water on the well sampling log or in the field notebook.
- Slowly remove the probe from the monitoring well and decontaminate the probe and portion of the cable that enters the water column as described above.
- Replace the monitoring well cap and protective-casing locking cap and lock the well.

2.7 GROUNDWATER MONITORING WELL SAMPLE COLLECTION

Delaware will collect a round of groundwater samples from each of the thirteen monitoring wells on-Site as part of the Phase I Remedial Investigation. All of the groundwater samples collected will be analyzed for the volatile organic compounds in accordance with NYSDEC ASP Method 95-1. Groundwater from monitoring wells MW-1S, MW-1D-97, MW-98-8S and MW-98-8D will be analyzed for the NYSDEC TCL/TAL parameters (NYSDEC CLP methods). Groundwater samples will be collected using either single-use, disposable bailers. The specific sampling procedures for the groundwater monitoring wells are detailed below:

2.7.1 Groundwater Monitoring Well Sample Collection

- Spread plastic sheeting on the ground around the well.
- Unlock and carefully remove the well cover to avoid having any foreign material enter the well.
- Monitor the interior of the riser pipe with a PID for the presence of volatile organic compounds.
- Sound the well to determine the depth. Using an electronic water level detector, measure the
 water level from below the measuring point on the top of the casing. Determine the volume of
 water in the well based on the water column and diameter of the well. Decontaminate the end
 of the probe between wells by washing with liquinox and water, and rinsing with deionized
 water.
- Purge the well using either a Waterra pump with dedicated polyethylene tubing and a bottom check valve or a single-use, disposable bottom-filling bailer with new dedicated 'k' nylon line.
- Purge approximately 3 to 5 well volumes from each well, measured in a 5 gallon bucket, starting
 with removal of the water from the top of the well if using a bailer. (Note: A well volume is
 defined as the volume of water standing inside the casing as measured prior to purging.) If the

well purges to dryness and recharges rapidly (within 15 minutes), continue to remove water as it recharges until the required volumes are attained. Terminate purging if the well purges to dryness and is slow to recharge.

- Perform well sampling within three hours of purging. In the case of wells that recharge slowly, sample as soon as adequate recharge has occurred. It a well does not contain or yield sufficient volume for all required laboratory analytical testing, including quality control, prioritize sample analyses.
- After well purging is completed and the well has sufficiently recharged, collect the samples using a single-use, disposable. Fill the sample containers with a minimum of sample agitation.
- Collect the samples in the general order of the sensitivity of the parameters to volatilization. The preferred collection order for common groundwater parameters is as follows:
 - 1) Volatile organic compounds
 - 2) Semivolatile organic compounds
 - 3) Pesticides/Aroclors
 - 4) Field parameters (pH, specific conductance, turbidity, temperature)
 - 5) Metals

In cases where total metals are a contaminant of particular concern and the monitoring well produces samples with a high sediment load, it may be appropriate to collect the total metals sample first. This will help to avoid the entrainment of sediments in the sample, which could influence the analytical data.

- Clearly label all samples with a waterproof marker (pen for volatiles), and place on ice.
- Collect a separate sample of approximately 200-mL to perform field measurements of pH, conductivity, turbidity and temperature.

3.0 QUALITY ASSURANCE PROJECT PLAN

This Quality Assurance Project Plan (QAPjP) has been prepared by Delaware to supplement the site-specific Work Plan for the Phase I remedial investigation for the Site. The overall objective is to identify procedures for sampling, chain-of-custody, laboratory analysis, instrument calibration, data reduction and reporting, internal quality control, audits, preventive maintenance, and corrective action. It presents the field and laboratory quality assurance/quality control (QA/QC) policies and procedures that will be followed during the implementation of the project.

3.1 SAMPLE LABELING, HANDLING, AND SHIPPING

3.1.1 Sample Identification/Labeling

All samples will be assigned a unique identification code consisting of three or four parts. These parts generally consist of the project, sample type, boring number or location, and additional identification codes (as needed). Examples of the codes used for each sample type are identified below.

Environmental Samples

Soil Gas Samples

Example	CCC-SG-	04	
-	CCC-	SG-	04
	project	sample	sample
		type	number

Spill Area Soil Boring Samples

Example	CCC-SASB-03 (4-4.5')				
-	CCC-	SASB-	03	(4-4.5')	
	project	sample	boring	sample	
		type	number	depth	

Dry Well Soil Boring Samples

Example	CCC-DWSB-12 (6-12")				
-	CCC-	DWSB-	01	(6-12")	
	project	sample	boring	sample	
		type	number	depth	

Monitoring Well Installation Soil Boring Samples

Example	CCC-MV	VSB-6D (8.5-9')		
-	CCC-	MWSB-	6D	(8.5-9')
	project	sample	monitoring well	sample
		type	identification	depth

Dry Well Hand Auger Soil Samples

Example

CCC-DWSS-03 (0-6")

CCC-

DWSS-

03

(0-6")

project

sample type boring number sample depth

Groundwater Monitoring Well Samples

Example

CCC-MW-97-4S

CCC-

MW-97-4S

project

monitoring well identification

Quality Assurance/Quality Control Samples

Matrix Spike/Matrix Spike Duplicate Samples: QA/QC samples will include a matrix spike (MS) and matrix spike duplicate (MSD) sample at a frequency of not less than 5% (one MS/MSD pair per every 20 samples collected) for each matrix (aqueous and soil). They will receive the following code:

Example

CCC-MW-97-1D MS and CCC-MW-97-1D MSD CCC-DWSB-12 (6-12") MS and CCC-DWSB-12 (6-12") MSD

<u>Blind Field Duplicate Samples:</u> Field duplicate samples are sent blind to the laboratory. They will receive the following code:

Examples

X-1

X-2

X-3

X-n

The sample location where a blind field duplicate is collected will be marked both in the field notebook and on the copy of the chain-of-custody record retained by the sampling. A blind field duplicate sample will be collected at a frequency of one per every 20 samples for each matrix (aqueous and soil).

Equipment Blanks: Equipment blanks are not required when dedicated sampling equipment is used. If non-dedicated sampling equipment is used in the soil sampling program, equipment blanks will be analyzed at a frequency of not less than 5% (one equipment blank per every 20 samples collected). In either case, they receive the following code:

Example

EB-mm/dd-# (where mm/dd represents the date the field blank was collected and # represents the order collected, if more than one equipment blank is collected on any given day)

<u>Trip Blanks</u>: Trip blanks are used to monitor potential aqueous sample volatile organic contamination during shipment to and from the laboratory. It also provides information on laboratory water quality since the laboratory provides the trip blank water. One trip blank will submitted for analysis for each day aqueous matrix volatile organic samples are collected. A trip blank will be included in each cooler that contains aqueous matrix volatile organic samples, therefore all volatile organic samples and containers will be shipped to and from the laboratory in the smallest number of coolers possible in order to minimize the number of trip blanks required.

Example TB -mm/dd-# (where mm/dd represents the date the trip blank was collected and # represents the order collected, if more than one equipment blank is collected on any given day)

All sample containers will be labeled prior to sample collection. A non-removable label on which the following information is recorded with a permanent water-proof marker (pen for volatile samples) will be affixed to each sample container for shipment to the laboratory:

- project name/location (CCC);
- sample identification code;
- date and time the sample was collected (except for blind field duplicates, where the time will be omitted);
- sample type (soil or aqueous); and
- analysis requested.

3.1.2 Containers, Preservation, and Holding Times

All sample containers used will be of traceable quality purchased and supplied by the laboratory. The selection of sample containers used to collect the samples is based on the following considerations:

- sample matrix;
- analytical methods;
- potential contaminants of concern;
- reactivity of container material with sample; and
- QA/QC requirements.

The required containers, preservatives and holding times will conform to the **NYSDEC Analytical Services Protocol** (10/95) and are presented in Appendix A. No chemical preservative is required for soil samples, although the samples will be kept on ice in a cooler at a temperature of 4° C ($\pm 2^{\circ}$ C).

3.1.3 Chain-of-Custody Protocol and Shipping Requirements

A chain-of-custody record will be initiated by Delaware personnel upon sample collection and by the laboratory providing the sample containers. The laboratory record traces the path of the initial sample bottles and preservation at the laboratory to the field for sample collection. The Delaware chain of custody is initiated at the point of sample collection and documents their return to the laboratory for analysis.

The Delaware Project Manager or designated representative will notify the laboratory of the anticipated schedule of upcoming field sampling activities. This notification will include information concerning the number and type of samples, as well as the anticipated date(s) of shipment of samples to the laboratory. The laboratory will be responsible for supplying insulated containers (typically coolers) for storing and shipping the samples. Field samplers receiving the sample containers check each cooler and inspect the contents for breakage upon receipt. All sample bottles within each shipping container are individually labeled with an adhesive identification tag provided by the laboratory.

Once the sample containers are filled, they are immediately placed in the cooler with sealed bags of ice ("wet ice") or synthetic ice packs ("blue ice") to maintain the samples at 4°C (±2°C). To the extent possible, the chain of custody is filled out prior going in the field. Following sample collection, the field sampler properly completes the chain of custody for each sample. The chain-of-custody forms are then signed and placed in a sealed plastic Ziploc bag in the cooler. The shipping containers are then closed and properly sealed and the cooler is shipped to the laboratory via an overnight courier or hand delivered under appropriate chain-of-custody procedures. Whenever possible, the samples will be shipped within 24 hours of collection. Samples will not be shipped later than 48 hours following collection. Upon receipt of the coolers at the laboratory, the cooler's contents are inspected and the chain of custody signed, thus accepting custody of the samples.

3.1.4 Cleaning of Field Sampling Equipment

All non-dedicated equipment and tools used to collect samples for chemical analyses (including trowels, spatulas, spoons, scoops, hand augers, and split-spoons) will be decontaminated using the following procedures:

- Non-phosphate detergent wash;
- Tap water rinse;
- Laboratory-grade methanol or isopropanol rinse; and
- Distilled/deionized water rinse.

If equipment is to be stored for future use, allow it to air dry, and then wrap it in aluminum foil (shiny-side out) or seal in plastic bags. Decontamination fluid will be discharged directly to the ground away from any surface water or containerized on-site if necessary.

3.1.5 Cleaning of Pumps and Pumping Equipment

In general, all suction-lift pumps and pumping equipment that have come in contact with the water column during well development and/or purging will use dedicated and pre-cleaned tubing. If submersible pumps are used, the following cleaning procedure will be employed:

- Wash the exteriors of the pump, wiring, and cables with non-phosphate detergent;
- Rinse with potable water;
- Pump a minimum of 25 gallons of potable water through the pump housing and through the pump tubing if a dedicated pre-cleaned discharge hose is not used for each well:
- Perform a final rinse by pumping 5 gallons of distilled/deionized water through the pump and pump tubing.

3.2 ANALYTICAL LABORATORY/ANALYTICAL METHODS

The analytical laboratory contracted to perform the sample analyses will be a New York State Department of Health (NYSDOH), Environmental Laboratory Approval Program (ELAP) certified laboratory holding the Analytical Services Protocol (ASP) certification. The Quality Assurance Plan (QAP) for the laboratory selected will be submitted under separate cover as Appendix C to this document.

All samples will be analyzed using NYSDEC ASP (10/95) analytical procedures for Superfund-CLP Volatile Organics (NYSDEC Method 95-1), Semivolatile Organics (NYSDEC Method 95-2), Pesticides/Aroclors (NYSDEC Method 95-3) and/or the appropriate Analytical Methods for CLP Inorganics deliverables, as necessary. The sample and analysis summary for the Phase I remedial investigation is presented in Table 1.

3.3 DATA QUALITY REQUIREMENTS

3.3.1 Data Quality Objectives

Data quality objectives (DQO) for data measurement are generally defined in terms of six parameters: precision, accuracy, representativeness, comparability and completeness (PARCC). The following DQO have been established to ensure that the data collected as part of this program are sufficient and of adequate quality for their intended uses. Data collected and analyzed in conformance with the DQO process described in this QAPjP are used to assess the uncertainty associated with decisions related to the Site.

3.3.2 Precision

Precision measures the reproducibility of measurements under a given set of conditions. To maximize precision, established sampling and analytical procedures are consistently followed. Analytical precision is monitored through analysis of matrix spike duplicates and field duplicates.

Matrix spike duplicates for organic compounds are analyzed at a frequency of once for every 20 samples as specified by the ASP. Precision is expressed as the relative percent difference (%RPD):

$$%RPD = 100 \times 2[(X_1 - X_2)/(X_1 + X_2)]$$

where X_1 and X_2 are reported concentrations for each duplicate sample and subtracted differences represent absolute values. The equation is taken from "Data Quality Objectives for Remedial Response Activities" (EPA/540/G-87/003, March 1987).

3.3.3 Accuracy

Accuracy measures the bias in a measurement system. Laboratory accuracy is assessed through use of laboratory internal QC samples, matrix spikes, and surrogate recovery. The laboratory objective for accuracy is to equal or exceed the accuracy demonstrated for the applied analytical methods on similar samples. A matrix spike and matrix spike blank are analyzed once for every twenty samples, as specified in the ASP.

Accuracy values can be presented in a variety of ways. Average error is one way of presenting this information; however, more commonly, accuracy is presented as percent bias or percent recovery. Percent bias is a standardized average error (the average error divided by the actual or spiked concentration and converted to a percentage). Percent bias is unit-less and allows accuracy of analytical procedures to be compared easily. Percent recovery provides the same information as percent bias. Routine organic analytical protocols require a surrogate spike in each sample. Percent recovery is defined as:

% Recovery = (R/S) x 100

Where

S = spike surrogate concentration
R = reported surrogate concentration
and % Bias = % Recovery - 100

This equation is taken from "Data Quality Objectives for Remedial Response Activities" (EPA/540/G-87/003, March 1987). Percent recovery criteria published by the NYSDEC as part of the NYSDEC ASP (10/95) and those determined from laboratory performance data are used to evaluate accuracy in matrix spike and blank spike quality control samples.

3.3.4 Representativeness

Representativeness is a qualitative parameter that expresses the degree to which sample data accurately and precisely represent actual conditions. In the field, the representativeness of the data depends on selection of appropriate sampling locations, collection of an adequate number of samples, and use of consistent sampling procedures. The sampling procedures, as described in the FSP, are designed with the goal of obtaining representative samples for each of the different matrices.

In the analytical laboratory, the representativeness of the analytical data is a function of the procedures used in processing the samples. The objective for representativeness is to provide data of the same high quality as other analyses of similar samples using the same methods during the same time period within the laboratory. Representativeness is determined by comparing the quality control data for these samples against other data for similar samples analyzed at the same time.

3.3.5 Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. Analytical results are comparable to results of other laboratories with the use of the following procedures/programs: Instrument standards traceable to National Institute of Standards and Testing (NIST), Environmental Protection Agency (EPA) or NYSDEC sources; the use of standard methodology; reporting results from similar matrices in consistent units; applying appropriate levels of quality control within the context of the laboratory quality assurance program; and participation in inter-laboratory studies to document laboratory performance. By using traceable standards and standard methods, the analytical results can be compared to other laboratories operating similarly. The QA program documents internal performance, and the inter-laboratory studies document performance compared to other laboratories. Periodic laboratory proficiency studies are instituted as a means of monitoring intra-laboratory performance.

3.3.6 Completeness

Completeness is the percentage of measurements made that are judged to be valid measurements. The completeness goal is to generate the maximum amount possible of useable data (i.e., 100% usable data). Data is considered usable unless qualified during validation as "R," rejected.

3.3.7 Reporting Limits

The estimated reporting limits or practical quantification limits that are desired for each analysis are the Contract Required Detection Limits specified in the NYSDEC ASP (10/95). All such limits are dependent upon matrix interferences and reporting limits may vary as a result of dilution.

3.4 FIELD QUALITY ASSURANCE SAMPLES

3.4.1 Blind Field Duplicate Samples

Field duplicate samples are used to assess the variability of a matrix at a specific sampling point and to assess the reproducibility of the sampling method. Field duplicate samples are defined as a second sample collected from the same location, at the same time, in the exact same manner as the first and placed into a separate container with no prior mixing. Field duplicate samples are collected at a frequency of one per every twenty (20) samples per matrix. Each duplicate sample is analyzed for the same parameters as the samples collected that day. Thus, both field and laboratory variability are evaluated. Acceptance and control limits for the laboratory follow NYSDEC ASP guidelines for organic analyses. However, any deviations in the data with respect to the limits will be discussed in the report. Although there are no established QC limits for field duplicate RPD data, Delaware considers RPD values of 40% or less an indication of acceptable sampling and analytical precision.

3.4.2 Split Samples

Split samples are usually used for performance audits or inter-laboratory comparability of data. The collection of split samples is not anticipated during the course of this project. However, if the NYSDEC or other appropriate agency requests split samples to be collected, then the following applies: A split sample is defined as two separate samples taken from a single aliquot that has been thoroughly mixed or homogenized prior to the formation of the two separate samples.

3.4.3 Equipment Blanks

Equipment blanks are not required when dedicated sampling equipment is used. If non-dedicated sampling equipment is used for the soil sampling program, equipment blanks will be analyzed at a frequency of not less than 5% (i.e., one equipment blank per every 20 samples collected).

3.4.4 Trip Blanks

Trip blanks are used to monitor potential sample volatile organic contamination during shipment to and from the laboratory. It also provides information on laboratory water quality since the laboratory provides the trip blank water. One trip blank will be submitted for analysis for each day that aqueous volatile organic samples are collected. A trip blank will be included in each cooler that contains aqueous volatile organic samples, therefore all aqueous volatile organic samples and containers will be shipped to and from the laboratory in the smallest possible number of coolers in order to minimize the number of trip blanks required.

3.5 LABORATORY QUALITY ASSURANCE SAMPLES

3.5.1 Method Blanks

Method blanks are used to assess the background variability of the method and to assess the introduction of contamination to the samples by the method, technique, or instrument as the sample is prepared and analyzed in the laboratory. A method blank is defined as an aliquot of laboratory deionized water on which every step of the method is performed and analyzed along with the samples. Method blanks are analyzed at a frequency of one (1) for every 20 samples analyzed, or every analytical batch, whichever is more frequent.

3.5.2 Spiked Samples

Two types of spiked samples are analyzed as part of the analytical QA/QC program, and include matrix spikes (MS) and matrix spike duplicates (MSD). Matrix spike samples are analyzed to evaluate instrument and method performance on samples of similar matrix. Matrix spike duplicates are analyzed to determine the precision of the method and instrument. These samples are analyzed and the percent recovery is determined to assess matrix interferences affects on the methods. One MS/MSD sample pair will be analyzed for every 20 samples.

3.6 EQUIPMENT CALIBRATION AND MAINTENANCE

3.6.1 Field Equipment

3.6.1.1 CALIBRATION

Field equipment that may be used during collection of environmental samples at the Site includes the Horiba U-10 Water Quality Checker or equivalent, a Photovac 2020 photoionization detector (PID) equipped with an 11.7 eV lamp or equivalent, an MSA Passport® combustible gas indicator or equivalent, and an MIE *personal* DataRAMTM (pDR) or equivalent to measure real-time concentrations of airborne particulates.

Field QC check control limits (pH, conductivity and turbidity) for the Horiba U-10 Water Quality Checker are outlined below. In addition, field determinations of pH, conductivity, turbidity, temperature, salinity and redox potential will be obtained in duplicate once for every 20 aqueous samples collected.

- <u>pH</u>: If the pH QC sample (pH 7.0 or pH 10.0 buffer after initial automatic calibration with pH 4.0 buffer) exceeds ± 0.5 pH units from the true value, the source of the error is determined and the instrument re-calibrated. If a continuing calibration check with pH 7.0 buffer is off by ± 0.5 pH unit, the instrument is re-calibrated.
- Conductivity: QC samples must be within $\pm 10\%$ of the true values. (The true value for conductivity in the automatic calibration solution is 4,490 umhos/cm.)
- <u>Turbidity</u>: QC samples must be within ± 10% of the true values. Turbidity QC samples are commercially prepared polymer standards such as those available from Advanced Polymer System, Inc. or equivalent. The initial automatic calibration solution has a turbidity value of 0 NTU.

The PID, Passport and pDR are each calibrated according to the manufacturer's instructions at the beginning of the day, whenever the instrument is turned off for more than two hours and at the discretion of the Site Safety Officer (SSO).

3.6.1.2 MAINTENANCE

Prior to field sampling events, each piece of field equipment is inspected to ensure it is operational. If necessary, the equipment is serviced. Meters that require charged batteries are fully charged or have fresh batteries. Due to Delaware's relationship with a number of firms which rent instrumentation, safety and sampling equipment, significant downtime should not occur. In addition to this, field personnel carry key spare parts and equipment into the field to prevent downtime.

3.6.2 Laboratory Equipment

All laboratory equipment is calibrated according to the requirements of the respective NYSDEC ASP (10/95) method for each analysis and/or in accordance with the manufacturer's specifications. In general, preventative maintenance of laboratory equipment follows the guidelines recommended by the manufacturer. Generally speaking, a malfunctioning instrument which cannot be repaired directly by laboratory personnel is repaired following a service call to the manufacturer.

3.7 DATA DOCUMENTATION

3.7.1 Field Notebook

Field notebooks will be initiated at the start of on-site work. One notebook will be maintained by the geologist overseeing intrusive activities while another notebook will be maintained by the SSO. All original forms and notebooks used during field activities become part of the permanent project file. Each field notebook will include the following daily information, where applicable:

- date;
- meteorological conditions;
- crew members:
- brief description of proposed field activities for that day;
- locations where work is performed;
- problems and corrective actions taken;
- records of all field measurements;
- a description of all modifications to the work plan;
- a record of all field data sampling point locations;
- pertinent sample collection information;
- chain-of-custody information; and
- documentation of the calibration of field instrumentation used.

3.8 CORRECTIVE ACTIONS

Corrective actions are required when a problem arises that impedes the progress of the investigation as detailed in the project plans, or when field or analytical data are not within the objectives specified in the Work Plan or QAPjP. Corrective actions include those actions implemented to promptly identify, document, and evaluate the problem and its source, as well as those actions taken to correct the problem. These corrective actions are documented in the project file. Prior to implementing any deviations from the approved procedures contained in the QAPjP, the Project Manager must be notified.

3.8.1 Field Procedures

Project personnel continuously monitor ongoing work performance as part of their daily responsibilities. If a condition is noted that would have an adverse impact on data quality, corrective actions are taken. Situations that require corrective action include the following:

- standard operating procedures and or protocols identified in the project-specific work plan or QAPjP have not been followed;
- equipment is not calibrated properly or in proper working order;
- QC requirements have not been met; and
- performance or system audits identify issues of concern.

The problem, its cause, and the corrective action implemented are documented. The PM is responsible for initiating and approving corrective actions.

3.8.2 Laboratory Procedures

During all investigations/studies, instrument and method performance and data validity are monitored by the analytical laboratory performing the analyses. The laboratory calibrates its instruments and documents the calibration data. Laboratory personnel continuously monitor the performance of its instruments to ensure that performance data fall within acceptable limits. If instrument performance or data fall outside acceptable limits, or when any condition is noted that has an adverse effect on data quality, then the laboratory implements appropriate corrective actions. Situations that require corrective action include the following:

- protocols defined by the project-specific QAPjP have not been followed;
- identified data acceptance standards are not obtained;
- equipment is not calibrated properly or in proper working order;
- sample and test results are not completely traceable;
- QC requirements have not been met; and
- performance or system audits identify issues of concern.

The laboratory QA Officer is responsible for initiating and approving corrective actions. The corrective actions may include one or more of the following:

- re-calibration or standardization of instruments;
- acquiring new standards;
- repairing equipment; and
- reanalyzing samples or repeating portions of work.

System audits and calibration procedures with data review are conducted by the laboratory at a frequency so that errors and problems are detected early, thus avoiding the prospect of redoing large segments of work. Delaware provides independent data validation and/or data review and summary, and the laboratory is notified as soon as possible of any situation which requires corrective action so that the corrective action may be implemented in a timely manner.

3.9 DATA REDUCTION, REVIEW AND REPORTING

3.9.1 Laboratory Data

The laboratory is required to meet all applicable documentation, data reduction, and reporting protocols as specified in the NYSDEC ASP (10/95) CLP deliverable format. Calculations of sample concentrations are performed using the appropriate regression analysis program, response factors, and dilution factors, where applicable. The laboratory, through its assigned QAO, conducts its own internal review of the analytical data generated for a specific project prior to sending the data to Delaware. Deficiencies discovered during the laboratory internal data validation, as well as the corrective actions used to correct the deficiency, are documented in the laboratory Case Narrative submitted with each data package.

The laboratory reports the data in tabular form by method and sample. The laboratory is required to submit analytical results that are supported by a complete NYSDEC ASP CLP data package to enable the quality of the data to be determined. This standard backup data includes supporting documentation (chromatograms, raw data, etc.), sample preparation information, and sample handling information (i.e., chain-of-custody documentation).

3.9.2 Data Review

In addition to the laboratory's in-house review of the data, Delaware chemists will review the laboratory standard quality control summary forms prior to its incorporation into a final report and complete a Data Usability Summary Report (DUSR). This data review will follow the NYSDEC Guidance for Development of Data Usability Reports (Appendix B); complete validation of the data in accordance with the National Functional Guidelines will not be performed. Upon receipt of the laboratory data analytical package, the data reviewer:

- 1. Reviews the data package to determine completeness. It must contain all sample chain-of-custody forms, case narratives including sample/analysis summary forms, QA/QC summaries with supporting documentation, relevant calibration data, instrument and method performance data, documentation of the laboratories ability to attain the method detection limits for target analytes in required matrices, data report forms with examples of calculations, and raw data. The laboratory is promptly notified of any deficiencies, and must produce the documentation necessary to correct the deficiencies within 10 calendar days.
- Reviews the data package to determine compliance with the applicable portions of the work plan. The data reviewer confirms that the data is produced and reported consistent with the QAPJP and laboratory quality control program, protocol-required QA/QC criteria are met, instrument performance and calibration requirements were met, protocol required calibration data are present and documented, data reporting forms are complete, and problems encountered during the analytical process and actions taken to correct the problems are reported. Field duplicate data are evaluated to determine field variability.

3. <u>Prepares a tabular summary of the reported data</u>. The data reviewer summarizes the data in a tabular format to provide the data in more accessible format.

3.9.3 Field/Engineering Data

Field data (i.e., information collected in the field through observation, manual measurement, and/or field instrumentation) is recorded in a dedicated project field notebook, on the appropriate field data sheets, and/or on the appropriate field data forms. This data is reviewed by the field manager and the project manager for adherence to the work plan and QAPjP requirements. The final reporting of the data is reviewed by the project field personnel, who also participate in data reduction and evaluation.

Field documentation, data calculations, transfers, and interpretations are conducted by field personnel, and reviewed for accuracy by the project manager and/or his designee for:

- general completeness;
- readability;
- usage of appropriate procedures;
- appropriate instrument calibration and maintenance;
- reasonableness in comparison to present and past data collected;
- correct sample locations; and
- correct calculation and interpretations.

Approximately 5% of all calculations are checked through recalculation. If appropriate, field data forms and/or calculations are included in project report appendices. All of the original field notebooks, logs, forms and documents are kept in the project file.

3.10 QUALITY ASSURANCE CONTROLS

The Project Manager and the QAO are responsible for ensuring that quality QA/QC records such as chain-of-custody forms, field notebooks, and data summaries are being properly prepared. The Project Manager is responsible for ensuring that all records are properly filed. Information received from outside sources, such as laboratory analytical reports, is retained at Rust. Access to working project files is restricted to project personnel.

3.10.1 Field Audits

The Project Manager is responsible for ensuring that all field investigations are performed in accordance with the requirements and specifications outlined in this QAPjP. As part of Delaware's field QA/QC program, a field audit is performed by Delaware's Quality Assurance Officer (QAO) or a designated representative on projects where sampling activities extend for more than two weeks. The primary purpose of the field audit is to monitor project sampling practices. The QA/QC field audit is performed during sampling to evaluate the performance of work during the collection of samples for laboratory analysis.

For projects of relatively short duration (*i.e.*, continuous field work of less than two week), a formal audit of field activities is not performed. The field team leader or appropriate task manager monitor field performance and document all work performed in field notes, a narrative, and/or a checklist of tasks, as appropriate. The Project Manager and/or QAO review this documentation to ensure the necessary information has been recorded and conduct discussions with field team members to verify that field activities were performed according to the project Work Plan, QAPjP and HASP. The QAO communicates any concerns to the field team as appropriate. A formal field audit will not be performed in conjunction with this project.

3.10.2 Meetings

Periodic meetings between the Project Manager and QAO will be held to review quality assurance procedures, field work, laboratory performance and data documentation and review. Any potential problems identified during the review are documented and addressed. If necessary, they are reported to management for review and appropriate corrective action.

APPENDIX A

SAMPLE PRESERVATION, CONTAINERS AND HOLDING TIME REQUIREMENTS

PART II SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS

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The Laboratory shall adhere to the preservation procedures and holding times listed in Table I below unless specifically directed otherwise by the Bureau of Technical Services and Research or the Project Officer. All holding times are from Verified Time of Sample Receipt (VTSR) at the Laboratory.

The Laboratory shall provide all necessary preservatives to properly stabilize the samples. The Laboratory must adhere to all analytical holding times. Failure to do so will result in the imposition of any contract specified penalties.

Table I - Required Containers, Preservatives, and Holding Times

Parameter Name	Container ¹	Preservative ^{2,3}	Maximum Holding Time⁴
Aqueous Samples			
Bacteriological Tests:			
Total Coliform	Sterilized P,G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	6 hours
Fecal Coliform	Sterilized P,G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	6 hours
Fecal Streptococci	Sterilized P,G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	6 hours
Inorganic and Convention	als Tests:		
Acidity	P,G	Cool, 4°C	12 days
Alkalinity	P,G	Cool, 4°C	12 days
Ammonia	P,G	Cool, 4°C, H ₂ SO ₄ to pH < 2	26 days
BOD ₅	P,G	Cool, 4°C	24 hours
BOD ₂₀	P,G	Cool, 4°C	24 hours
Bromide	P,G	Cool, 4°C	26 days

Table I - Required Containers, Preservatives, and Holding Times (Continued)

Parameter Name	Container ¹	Preservative ^{2.3}	Maximum Holding Time⁴
Aqueous Samples (Cont	inued)		
CBOD ₅	P,G	Cool, 4°C	24 hours
COD	P,G	Cool, 4°C, H ₂ SO ₄ to pH < 2	26 days
Chloride	P,G	Cool, 4°C	26 days
Color	P,G	Cool, 4°C	24 hours
Cyanide, Total	P,G	Cool, 4°C, NaOH to pH > 12	12 days
Cyanide, Amenable to Chlorination	P,G	Cool, 4°C, NaOH to pH > 12, 0.6 g ascorbic acid ⁵	12 days ⁶
Fluoride	P only	Cool, 4°C	26 days
Hardness	P,G	HNO ₃ to pH < 2	6 months
Kjeldahl Nitrogen	P,G	Cool, 4°C, H ₂ SO ₄ to pH < 2	26 days
Organic Nitrogen	P,G	Cool, 4° C, H_2 SO ₄ to pH < 2	26 days
Metals ⁷ , except Chromium ⁺⁶ and Mercury	P,G	HNO ₃ to pH < 2	6 months
Chromium ⁺⁶	P,G	Cool, 4°C	24 hours
Mercury	P,G	HNO ₃ to pH < 2	26 days

Table I - Required Containers, Preservatives, and Holding Times (Continued)

	Parameter Name	Container¹	Preservative ^{2,3}	Maximum Holding Time⁴
	Aqueous Samples (Cont	tinued)		
-	Nitrate + Nitrite	P,G	Cool, 4°C, H ₂ SO ₄ to pH < 2	26 days
	Nitrate	P,G	Cool, 4°C	24 hours
	Nitrite	P,G	Cool, 4°C	24 hours
	Oil and Grease	G only	Cool, 4°C, H ₂ SO ₄ to pH < 2	26 days
100	Total Organic Carbon	P,G	Cool, 4°C, H ₂ SO ₄ to pH < 2	26 days
	Orthophosphate	P,G	Cool, 4°C	24 hours
-	Total Phenois	G only	Cool, 4°C, H ₂ SO ₄ to pH < 2	26 days
-	Phosphorous, Total	P,G	Cool, 4°C, H ₂ SO ₄ to pH < 2	26 days
	Residue, Total	P,G	Cool, 4°C	5 days
	Residue, Filterable	P,G	Cool, 4°C	24 hours
	Residue, Non-Filterable	P,G	Cool, 4°C	5 days
	Residue, Settleable	P,G	Cool, 4°C	24 hours
	Residue, Volatile	P,G	Cool, 4°C	5 days

Table I - Required Containers, Preservatives, and Holding Times (Continued)

Parameter Name	Container ¹	Preservative ^{2,3}	Maximum Holding Time
Aqueous Samples (Cont	inued)		
Silica	P only	Cool, 4°C	26 da
Specific Conductance	P,G	Cool, 4°C	26 da
Sulfate	P,G	Cool, 4°C	26 da
Sulfide	P,G	Cool, 4°C,add zinc acetate plus NaOH to pH > 9	5 da
Surfactants (MBAS)	P,G	Cool, 4°C	24 ho
Turbidity	P,G	Cool, 4°C	24 ho
Organic Tests ⁸ :			
Purgeable Halocarbons	G, Teflon [®] lined septa	Cool, 4°C 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH <2 (Optional)	7 d Unpreser 10 d Preser
Purgeable Aromatics	G, Teflon [®] lined septa	Cool, 4°C 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH <2 (Optional)	7 d Unpreser 10 d Preser
Acrolein and Acrylonitrile	G, Teflon [®] lined septa	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ , Adjust to pH 4 - 5 ⁹	7 d
Phenolics ¹⁰	G, Teflon [®] lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	5 days a VTSR t extract 40 days analys

Table I - Required Containers, Preservatives, and Holding Times (Continued)

Parameter Name	Container ¹	Preservative ^{2,3}	Maximum Holding Time⁴
Aqueous Samples (Cor	ntinued)		
Benzidines ^{10,11}	G, Teflon® lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	5 days after VTSR until extraction ¹²
Phthalate esters ¹⁰	G, Teflon® lined cap	Cool, 4°C,	5 days after VTSR until extraction; 40 days for analysis ¹²
Nitrosamines ^{10,14}	G, Teflon® lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ , Store in dark	5 days after VTSR until extraction; 40 days for analysis ¹²
PCBs ¹⁰	G, Teflon® lined cap	Cool, 4°C	5 days after VTSR until extraction; 40 days for analysis ¹²
Nitroaromatics and Isophorone ¹⁰	G, Teflon [®] lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ , Store in dark	5 days after VTSR until extraction; 40 days for analysis ¹²
Polynuclear Aromatic Hydrocarbons ¹⁰	G, Teflon® lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ , Store in dark	5 days after VTSR until extraction; 40 days for analysis ¹²

Table I - Required Containers, Preservatives, and Holding Times (Continued)

Parameter Name	Container ¹	Preservative ^{2,3}	Maximum Holding Time⁴
Aqueous Samples (Con	tinued)		
Haloethers ¹⁰	G, Teflon [®] lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	5 days after VTSR until extraction; 40 days for analysis ¹²
Chlorinated Hydrocarbons ¹⁰	G, Teflon® lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ ,	5 days after VTSR until extraction; 40 days for analysis ¹²
Chlorinated Dioxins and Furans ¹⁰	G, Teflon [®] lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ ,	5 days after VTSR until extraction; 40 days for analysis ¹²
Pesticides ¹⁰	G, Teflon [®] lined cap	Cool, 4°C, Adjust pH to 5 - 9 ¹⁴	5 days after VTSR until extraction; 40 days for analysis ¹²
Radiological Tests:			
Alpha, beta and Radium	P,G	HNO ₃ to pH < 2	6 months

Footnotes for Table I

- 1. Polyethylene (P) or Glass (G).
- 2. Sample preservation should be performed immediately upon 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 such compliance. For preservation requirements of Table I, 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 (HCI) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric Acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric Acid (H₂SO₄) 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. Samples may be held for longer periods only if the monitoring laboratory has data on file to show that specific types of samples under study are stable for the longer time, and has received written permission prior to analysis from the Regional Administrator under 40 CFR Part 136.3(e) AND from the Bureau of Technical Services and Research. Some samples may not be stable for the maximum time period given in the table. A monitoring laboratory is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability.
 - 5. Should only be used in the presence of residual chlorine.
- 6. Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
- 7. Samples should be filtered immediately on-site before adding preservative for dissolved metals.
- 8. Guidance applies to samples to be analyzed by GC, LC or GC/MS for specific compounds.

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- **9.** The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
- 10. 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 chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6 9; samples preserved in this manner may be held for five days before extraction and for forty 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 12, 13 (re the analysis of benzidine).
- 11. If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 \pm 0.2 to prevent rearrangement of benzidine.
- 12. This does not supersede the contract requirement of a 30 day reporting time.
- **13.** Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
- 14. For the analysis of diphenylnitrosamine, add 0.008% sodium thiosulfate and adjust the pH to 7 10 with NaOH within 24 hours of sampling.
- 15. The pH adjustment may be performed upon receipt in the Laboratory and may be omitted if the samples are extracted with 72 hours of collection. For the analysis of aldrin, add 0.008% sodium thiosulfate.

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Soil/Sediment/Solid Samples

The same containers and holding times as listed for aqueous samples are to be used for soil/sediment/solid samples, except for chlorinated dioxins and furans which are allowed 30 days until extraction. Preservation for all analyses is limited to cooling to 4°C.

Toxicity Characteristic Leaching Procedure Samples

	From :	VTSR	From :	TCLP Extraction	From :	Preparative extraction	Total Elapsed Time
	То:	TCLP extraction	To:	Preparative extraction	То:	Determinative analysis	
Volatiles		7		NA		7	14
Semivolatiles		5		7		40	52
Mercury		5		NA		28	33
Metals, except Mercury		180		NA		180	360
NA = Not ap	oplicable)					

APPENDIX B

NYSDEC GUIDANCE FOR THE DEVELOPMENT OF DATA USABILITY SUMMARY REPORTS

New York State Department of Environmental Conservation Division of Environmental Remediation

Guidance for the Development of Data Usability Summary Reports

Background:

The Data Usability Summary Report (DUSR) provides a thorough evaluation of analytical data without the costly and time consuming process of third party data validation. The primary objective of a DUSR is to determine whether or not the data, as presented, meets the site/project specific criteria for data quality and data use.

Though the substitution of a DUSR for a full third party data validation may seem to be a relaxation of the Division's quality assurance requirements, this is definitely not the case. The development of the DUSR must be carried out by an experienced environmental scientist, such as the project Quality Assurance Officer, who is fully capable of conducting a full data validation. Furthermore, the DUSR is developed from a full New York State Department of Environmental Conservation Analytical Services Protocol (NYSDEC ASP) Category B or a United States Environmental Protection Agency Contract Laboratory Protocol (USEPA CLP) deliverables package.

The DUSR and the data deliverables package will be reviewed by the Division's Quality Assurance Unit. In most cases, we expect that this review will result in agreement or with only minor differences that can be easily reconciled. If data validation is found to be necessary (e.g. pending litigation) this can be carried out at a later date on the same data package used for the development of the DUSR. Personnel Requirements:

The Environmental Scientist preparing the DUSR must hold a Bachelors Degree in a relevant natural or physical science or field of engineering and must submit a resume to the Division's Quality Assurance Unit documenting experience in environmental sampling analysis and data review.

Preparation of a DUSR:

The DUSR is developed by reviewing and evaluating the analytical data package. During the course of this review the following questions must be asked and answered:

- 1. Is the data package complete as defined under the requirements for the NYSDEC ASP Category B or USEPA CLP deliverables?
- 2. Have all holding times been met?
- 3. Do all the QC data: blanks, instrument tunings, calibration standards, calibration verifications, surrogate recoveries, spike recoveries, replicate analyses, laboratory controls and sample data fall within the protocol required limits and specifications?
- 4. Have all of the data been generated using established and agreed upon analytical protocols?
- 5. Does an evaluation of the raw data confirm the results provided in the data summary sheets and qualify control verification forms?
- .6. Have the correct data qualifiers been used?

Once the data package has been reviewed and the above questions asked and answered the DUSR proceeds to describe the samples and the analytical parameters. Data deficiencies, analytical protocol deviations and quality control problems are identified and their effect on the data is discussed. The DUSR shall also include recommendations on resampling/reanalysis. All data qualifications must be documented following the NYSDEC ASP '95 Rev. guidelines.

Contact the Division of Environmental Remediation Quality Assurance Group at (518) 457- 9280, with any questions on the preparation of a DUSR.

Revised 09/97

APPENDIX C

LABORATORY QUALITY ASSURANCE PLAN

QUALITY ASSURANCE PLAN

New York State

Department of Environmental Conservation

Analytical Services Protocol

Prepared by

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5-1	Manual Data Entry Form Pesticide, /PCB	2 of 12 0	5/01/98
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1. Organization and Personnel

1.1 QA Policy and Objectives

Controlling and ensuring the quality of the data produced by the laboratory is the responsibility of all personnel. It requires the ongoing dedication of the management and the support, understanding and commitment of the staff. Routinely, all Quality Assurance Functions are monitored independently by analytical personnel and audited by the Quality Assurance Unit.

The objective of any laboratory quality assurance program is the production of laboratory data of known quality. This requires a comprehensive and effective quality control program to measure and verify laboratory performance, and the use of approved or proven methods to produce data that is accurate, precise, and complete. In addition, the system must identify factors which adversely affect quality and provide for corrective action where required. The system must also provide for the maintenance of records relating to sample submittal and the production of laboratory data.

Three essential elements exit in an effective quality assurance program:

- 1. Prevention
- 2. Assessment
- 3. Correction

Prevention requires that an orderly program of planning and positive actions be executed before or during analyses to ensure that the analytical systems are functioning properly. This includes training, calibration of instruments, instrument maintenance, and frequent standardization of standard solutions, use of recommended or validated methodologies, and quality control planning.

Assessment requires that periodic checks of performance are conducted to determine precision and accuracy. This includes analyses of spike and duplicate samples and a peer review of instrument responses and calculations.

Corrective action requires qualitative and quantitative identification of defects in quality and the restoration to an acceptable functioning analytical system. This may involve trouble-shooting to correct equipment malfunction, examination of new standards, re-evaluation of methodology, and re-

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training.

The objectives of the QA Management Program at Toxikon are specifically defined as follows:

- 1. To assess the accuracy and precision of the data generated within the laboratory
- 2. To provide a measure of the accuracy and precision of analytical methods
- 3. To identify weak methodology
- 4. To detect training need within the analytical group
- 5. To provide a permanent record of instrument performance as a basis for projecting repair or replacement
- To upgrade overall quality of laboratory performance
- 7. To ensure that the analytical work will withstand legal scrutiny in regulatory actions.

QUALITY ASSURANCE PLAN

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1.2 QA Management

1.2.1 Organization

ANALYTICAL CHEMISTRY DIVISION

Laxman Desai, D.Sc. Scientific Director ANALYTICAL CHEMISTRY

N. Digiulio, M.S.
Director
OUALITY ASSURANCE/REGULATORY AFFAIRS

P. Letberg, B.S. Laboratory Manager Support Services Support Services R.Faiella, BA Proj. Admin.

Computer Svcs.

D. Sheeley, M.S. Technical Director Analytical Chemistry

S. Corbin, B.S. QA Officer Quality Assurance

ORGANICS K.Klausner, 3S X.Liu, HS Proj. Admin. INORGANICS D. Blackwell, BS Supervisor C. Leedom, MS Supervisor B.Ouelletta Chemistry Supervisor GC/HPLC Supervisor GC/HS Group Leader Extractions Wet Chemistry K. Baxter, BA Metals N.Calrns, 85 P.Colonero Guatomer Syca. 3.Rizzuto,8S Analyst Y.King,as Analyst A.Sweeney,as Aasc. Superv. Analyst F.D'Agostino Analyst 3.Parekh,88 C.Dilorenzo,3S Supervisor A.Lycna, 8s Analyse Sample Log-In C.Ly, 9S Analyst J.Regan, as Analyst Analyst Bottle Room Analyst J.Musanda, BS D.Angelo,3s T. Tessier, BS H.Surdick Analyse V.Rotenberg, 38 Analyst Analyst Bottle Room Analyst W.McNamara,BS J. Condon Might Superv. Courier B.Ouellette,BS T.Moulton Night Superv. Courier Y. Kando, Bs J. Maxwell, BS Analyst Hanager

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1.2.2 Assignment of QC and QA Responsibilities

Scientific Director

The Scientific Director designates project managers as required by the specific project. The Scientific Director ensures that personnel (including an independent QAU), resources, facilities, equipment, materials and methodologies, are available in the laboratory for all scheduled work. It is the overall responsibility of the Director to ensure that personnel clearly understand the work and are technically qualified. The Director must communicate any quality assurance finding which is reported to him by the QAU to project managers and ensure that corrective action has been taken and documented. The Scientific Director is responsible for the proper functioning of the Quality Assurance Program within the laboratory.

Technical Director

The Technical Director is responsible for the management of the organic and inorganic technical staff and for the maintenance of lab certifications, the schedule of performance, the flow of information, and the reporting of data.

Manager

The Laboratory Manager is responsible for the conduct of all work performed under his direction and the issuance of laboratory reports, sample storage, sample disposal, overseeing sample log-in and documentation and computer services.

<u>Supervisors</u>

Laboratory supervisors are responsible for the technical conduct of the work performed within their group and for the interpretation, analysis, documentation and reporting of raw data. They are responsible for training and continuing compliance of analysts, with methods, standard operating procedures and quality assurance requirements. They oversee the preventive maintenance and calibration for laboratory instruments. Supervisors serve as lead analyst within their service group and review data generated by their staff.

Quality Assurance Unit

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QAU monitors each project to insure that the facilities, equipment, personnel, methods, practices, records, and controls are in conformance with regulatory requirements.

Analytical Personnel

Analysts conduct laboratory analyses according to recommended or fully validated methodology, Standard Operating Procedures, and protocols. Responsibilities include accurate record keeping, documentation of data and events that could affect the quality or integrity of the data and reporting out-of-control situations and nonconformances to their supervisor. Analytical personnel take direction from their supervisor.

Sample Receipt Officer

The Sample Receipt Officer is responsible for sample custody procedures including logging-in, sample and extract security, and sample distribution.

Data Reduction / Administration

Data reduction personnel are responsible for entering test results and QC data into the Laboratory Information Management System network and reducing the data packages to a concise final report. The data reduction personnel are also responsible for maintaining organization of data within the laboratory network system and downloading data onto floppy discs for archiving as needed. Administrative responsibilities include final logout of the project from the tracking system, filing the final data package, and billing.

1.2.3 Reporting Relationships

Analytical personnel report to the supervisor of their group. Supervisors report directly to the Technical Director while support personnel report to the Laboratory Manager. The Technical Director and the Laboratory Manager report directly to the Scientific Director. The Quality Assurance Manager reports jointly to the Vice President of Regulatory Affairs and the Scientific Director.

1.2.4 QA Document Control Procedures

The quality control system of documentation for revisions of the Quality Assurance Manual and the Procedure Manuals for the chemistry division uses a standardized indexing format and provides for convenient replacement of pages that may be

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changed within the technical procedure descriptions. The indexing format at the top of the page includes the following:

Section Number Revision: Date: Page:

A digital numbering system identifies sections within the text. The "Section No" at the top of each page identifies major three-digit or two digit sections, where applicable. Almost all of the references in the text are to the section number, which can be found easily by scanning the top of the pages. References to subsections are used within the section. The date represents the date of the latest revision. Page number is the specific page in the section. The total number of pages in the section is shown in the "Table of Contents." An example of the page label for the first page of a section may be the following:

Section Number 3.0 Revision: 001 Date: 05/09/87 Page: 1 of 5

Each new two digit level, i.e. (3.0, 4.0), begins on a new page. This format groups the pages together to allow convenient revision by major sections. For subdivisions within a section (i.e. 3.1 or 3.1.1), it is also desirable to start on a new page. However, this applies only to discreet and separate procedures. Some manual sections may not have to be separated by a new page for every new subdivision (i.e. QAP) since the subdivisions do not always denote separate idea or need for stand alone pages. In these cases, the entire page could easily be revised and subdivisions may be condensed onto similar pages.

The Table of Contents follows the same structure as the text. It contains a space for total number of pages within each section. This allows the manual user to know how many pages are supposed to be in each section. When a revision to the text is made, the Table of Contents page must be updated. For example, the Table of Contents page detailing Section 3.0 may appear as follows:

QUALITY ASSURANCE PLAN

Section: 1.0
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Page: 7 of 32

	SECTION	PAGES	REVISION	DATE
3.1 3.2 3.3	Statement of Policy Introduction QA Plans QA Management QA Objectives	5 2 of 5 2 of 5 3 of 5 4 of 5	001 001 001 001 001	05/08/97 05/08/97 05/08/97 05/08/97 05/08/97

This format applies to sections not always on discreet and separate pages as described earlier. These sections state the page on which these sections begin with the total number of pages stated at the top "5" for that section. Alternately, if each sections as placed on a separate page, it may appear as follows:

	SECTION	PAGES	REVISION	DATE
3.1 3.2 3.3	Statement of Policy Introduction QA Plans QA Management QA Objectives	1 1 1 1	002 001 002 001 001	05/08/97 05/08/97 05/08/97 05/08/97 05/08/97

A revision to a section would change the Table of Contents to appear as follows:

	SECTION	PAGES	REVISION	DATE
3.1		1	002 001	05/08/97 05/08/97
3.2	QA Plans	1	001	05/08/97
3.3	QA Management	1	.00.2	05/08/97
3.4	QA Objectives	1	001	05/08/97

A distribution record is established and maintained up to date so that future versions of manual sections and the additions of new sections may be distributed to manual users. Changes may be made by the issuance of an entirely new document or replacement of complete sections. The laboratory personnel are given updates by the QAU who also keeps a signed record of the distribution list. The lab personnel are required to remove outdated sections of the manuals or SOP's, destroy, and replace with the updated version once the distribution list is signed for receipt of a section or SOP. Lab management personnel are responsible for monitoring that current material only is in use in the laboratory.

Procedures for the Preparation, Approval, Review, Revision,

Section: 1.0 Revision: 000 Date: 050198

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and Distribution of SOP's is covered in section 3.5 of this manual.

1.2.5 QA Program Assessment Procedures

Quality Assurance Reports are submitted after each system audit on a quarterly basis to the Scientific Director. Copies are circulated to the Laboratory Manager and Department Supervisors. Performance audit reports are submitted on a weekly basis to the Scientific Director and Laboratory Manager. If during a performance audit there is the need for immediate action, a corrective action is initiated. The Quality Assurance Unit is responsible for submitting all QA reports. Reports of training audits, health and safety audits, and operational audits are submitted to the Scientific Director.

1.3 Personnel

1.3.1 Resumes

04/06/98 04/06/98

REVISION:

000

STEWART MARKS SAMPLE CONTROL TECHNICIAN, ENVIRONMENTAL CHEMISTRY

B.S., Northeastern University, Boston, Massachusetts (1982)

1998 - Present

Toxikon Corporation, Bedford, Massachusetts

Since joining Toxikon, Mr. Marks has been the Sample Custodian for the Environmental Chemistry Division. Mr. Marks has been responsible for the receipt and log-in of all chemistry samples. He ensures regulatory compliance with the samples. He performs quality control functions to verify all sample processing activities are accomplished in accordance with proper procedures. He is responsible for documenting pertaining information with samples per EPA, FDA and client's requirements. Mr. Marks verifies chain of custody and conditions of samples. He assists with hazardous waste disposal.

1984 - 1997

Cybermation, Inc., Woburn, Massachusetts

(1994 - 1997)

Materials Manager

(1984 - 1987)

Engineering ECO/Documentation Control Supervisor

The Badger Company., Inc., Cambridge, Massachusetts Senior Engineering Aide/Documentation

US Air Force

DATE HIRED: 04/07/98
RESUME DATE: 04/07/98
REVISION: 000

ANDY KWOK METALS PREP TECHNICIAN

B.S. Chemistry, University of Massachusetts*Boston, Boston, MA (1997)

1998 - Present Toxikon Corporation, Bedford, Massachusetts

As a Metals Prep Technician, Mr. Kwok preps water, waste water and soil samples for instrument analysis. He cleans glassware and is responsible for sample documentation.

DATE HIRED: 03/16/98
RESUME DATE: 03/16/98
REVISION: 000

MICHAEL EBISTON ORGANICS EXTRACTION TECHNICIAN

B.S. Environmental Biology, Westfield State College, Westfield, MA (1998)

1998 - Present Toxikon Corporation, Bedford, Massachusetts

Since joining Toxikon, Mr. Ebitson has been working in the organics department, primarily running all organic extraction procedures, specifically methods 608, 625, 8270, 8080 and Herbicides. In addition, he is responsible for running various inorganic test methods under the direction of his supervisor. His responsibilities include record keeping, equipment calibration and preventive maintenance, documentation of data and special events that may affect data integrity.

02/02/98 02/02/98

RESUME DA

000

ROBERTA LALLY WET CHEMISTRY ANALYST

B.S. Agriculture (Environmental Soil Science), University of Delaware (1997)

1998 - Present Toxikon Corporation, Bedford, Massachusetts

As a Wet Chemistry Analyst, Ms. Lally is involved in extraction and preparation of samples for the Wet Chemistry Department. She also handles a wide range of inorganic analyses, e.g. UV/Vis and FTIR Spectroscopy.

Ms. Lally's experience includes preparing and analyzing samples for various tests following EPA guidelines on drinking water wastewater, solids and sludge. Her experience includes analysis, TCLP extractions, TPH by IR, oil and grease, bacteria, minerals, oxygen demand, phenols and solids.

04/07/98 04/07/98 000

REVISION:

JAMES LOVETT MIS MANAGER

MCP - Microsoft Certified Professional

MCPS - Microsoft Certified Product Specialist (NT 4.0 Server)

MCSE - Microsoft Certified System Engineer+Internet Specialist (April 1998)

1998- Present

Toxikon Corporation, Bedford, Massachusetts

As MIS Manager, Mr. Lovett is responsible for administering the computer network system. He assists with local area networks, including installation and troubleshooting of all hardware and software. He interfaces with the departments to provide programming support services for He oversees the LIMS system and maintains ISO-9001 certification and computer validation issues and related documentation.

1995 - 1998

IKON Office Solutions Northern New England, Bedford, New Hampshire

Mr. Lovett's primary responsibilities included configuring and maintaing a Novell server. He provided support to MIS division for hardware and software conflicts and configuration.

1994 - 1996

Computer Telephone Corporation, Bedford, New Hampshire

04/14/98 04/14/98

REVISION:

000

STEPHEN N. PRIDE GC ANALYST

B.A. Biology, Merrimack College, Andover, MA (1994)

Graduate courses taken towards M.S. Environmental Studies

Emergency Medical Technician

American Red Cross First Aid, CPR

1998- Present

Toxikon Corporation, Bedford, Massachusetts

As a GC Analyst, Mr. Pride is involved in the preparation and the analyses of volatile and semi-volatile samples for organic analyses. He maintains gas chromatography instrumentation. He processes data from HP computer data systems. He is responsible for analyses by EPA 8000, 600 and 500 series methods and special projects.

1996 1998

Accutest, Marlborough, Massachusetts

Mr. Pride worked in the lab as a GC Volatile Analyst. His responsibilities involved running the Massachusetts Volatile Petroleum Hydrocarbon, V602/8020, and V8015 methods. He has knowledge of Tekmar ALS2016, LSC2000, and HP5890 instruments. He also worked as an extraction chemist, preparing spikes and surrogates according to EPA procedures for various analytes.

1995 - 1996

Alpha Analytical Labs

Mr. Pride worked as an Organic Prep Chemist.

1994 - 1995

Americorps National Civil Community Corps.

12/12/94 10/15/96

REVISION:

002

DOUGLAS V. SHEELEY LAB DIRECTOR, ENVIRONMENTAL SCIENCES

M.S. Master of Science, State University of New York at Brockport

B.S. Bachelor of Science, State University of New York at Brockport

1994-Present

Toxikon Corporation, Bedford, Massachusetts

Since joining Toxikon, Mr. Sheeley has managed the organic and inorganic technical staff at Toxikon. He is responsible for maintaining lab certifications (CLP, EEP, HRS & GLP) and compliance with regulations for, but not limited to, CERCLA, SARA, UST, RCRA, SDWA, TCLP and NPDES. He is responsible for new methods development and the installation of new instruments. Mr. Sheeley oversees the LIMS system and ensures the schedule of performance, flow of information and reporting of data.

1987-1993

Nytest Environmental, Port Washington, NY

While at Nytest, Mr. Sheeley was the Laboratory Director who directed a staff of 75 and was instrumental in bringing the lab into the US EPA contract lab program. He directed the lab's expansion and relocation and managed NYSDEC data validation contracts. During this time, he has presented professional seminars at NJ Expo in 1987, 1988, the NJ ECRA in 1987, 1988, 1989 and Hazmat in 1987, 1988, and 1989.

1980-1987

Elson T. Killam Associates, Millburn, NJ

As Laboratory Director, Mr. Sheeley purchased, installed and maintained the instruments, including GC/MS, AA, Auto Analyzer, GC and IR units. He developed a computerized data handling and report generation system and obtained and maintained certifications. Mr. Sheeley supervised and conducted industrial wastewater and drinking water pilot plant treatment projects and managed the laboratory phase of a project which generated over 90,000 samples in one year.

1974-1980

Monroe County Pure Waters Agency, Rochester, NY

As Assistant Laboratory Director, Mr. Sheeley assisted in the laboratory start-up. He operated TOC, GC, AA, IR and other systems while being responsible for the analysis of process control parameters for three sewage treatment plants.

 DATE HIRED:
 03/17/87

 RESUME DATE:
 06/22/95

 REVISION:
 003

PAUL M. LEZBERG LABORATORY MANAGER

B.S. 1983 Biology, Colby College, Waterville, Maine

1986-Present

Toxikon Corporation, Bedford, Massachusetts

Mr. Lezberg is presently functioning in the role of the Laboratory Manager at Toxikon. He is responsible for overseeing the conduct of all work performed in the laboratories of the analytical division as well as providing status reports on the operation to the Scientific Director. Mr. Lezberg conducts data package and final report reviews and is responsible for implementing all procedures and quality control measures in the laboratory. He is also responsible for conducting and providing direction in the conduct of laboratory analyses and problem solving. As the lab manager, he must communicate daily with the clients on all technical matters relative to specific projects.

1985-1986

American Biogenics, Woburn, Massachusetts

Prior to joining Toxikon in 1986, Mr. Lezberg held the position of Manager of Regulatory Compliance at Bioassay Systems/American Biogenics in Woburn, Massachusetts. In this position, Mr. Lezberg was responsible for managing the QA/QC and Safety program as it pertained to the conduct of toxicological testing performed by the firm. He was responsible for implementing and managing an effective Health and Safety program which guarded both workers and the environment from exposure to potential hazards. He maintained the program by instituting or modifying engineering controls, work practices, and personnel protective equipment. Further responsibilities included monitoring the conduct of the toxicological studies for compliance with the FDA and EPA GLP's. In this capacity, he was responsible for laboratory management pertaining to protocol review, laboratory audits, writing lab standard operating procedures, review of raw data and technical reports, and interaction on a daily basis with technical personnel.

1983-1985

Bioassay Systems, Woburn, Massachusetts

Mr. Lezberg worked in a laboratory supervisor position for two years performing multiple cytogenetic assays designed to identify potential mutagens and carcinogens for government contract work as well as for the private sector. He supervised all aspects of the assays, including cell culture, preparation of test samples, and data analysis and reduction.

 DATE HIRED:
 1/26/98

 RESUME DATE:
 1/26/98

 REVISION:
 000

R. ALAN DOUGHTY, Ph.D. QUALITY ASSURANCE MANAGER, ENVIRONMENTAL CHEMISTRY

M.B.A. Roosevelt University, Chicago, Illinois (1978)

Ph.D. Organic Chemistry, University of Miami, Miami, Florida (1967)

M.S. Organic Chemistry, University of Minnesota, Minnesota (1963)

B.S. Physics, Purdue University, Lafayette, Indiana (1961)

1998 - Present

Toxikon Corporation, Bedford, Massachusetts

Quality Assurance Manager

As Quality Assurance Manager, Environmental Chemistry, Dr. Doughty has the responsibility of overseeing activities related to chemistry regulatory programs, including keeping the laboratory in compliance with required regulations and certifications for selected tests and documentation requirements. He has regulatory responsibilities, handles all audits and reviews Toxikon's Contingency Plan.

1994 -1996

NET, Bedford, Massachusetts

Laboratory Manager

As Quality Assurance Manager during illness of incumbent, Dr. Doughty revised the laboratory's Quality Assurance Plan to meet Massachusetts and Federal agency requirements (NEESA, USCOE, HAZWRAP). He shortened turnaround time for all laboratory work and wrote the chemical hygiene plan.

1993 - 1994

Orlando Labs Orlando, Florida

Laboratory Director

Dr. Doughty was responsible for operations, project management, quality assurance and health and safety.

1988 - 1992 RMT, Inc., Madison, Wisconsin

Laboratory Director

Dr. Doughty wrote the company's first Quality Assurance Plan to meet Wisconsin and EPA requirements. He also supervised a GLP pesticide study group.

1984 - 1988 International Technology Corp., Martinez, California

Director, Field Analytical and Sampling

1982 - 1984
The Bionetics Corp., Chicago, Illinois
Project Manager

1977 - 1982
The Quaker Oats Company, Chicago, Illinois
Director, Scientific Resources

 DATE HIRED:
 09/14/92

 RESUME DATE:
 2/2/98

 REVISION:
 003

BRIAN K. OUELLETTE METALS SUPERVISOR

Towards B.A. Chemistry, Suffolk University, Boston, MA

Environmental Data Reporting System (1996)

1992 - Present

Toxikon Corporation, Bedford, Massachusetts

Since joining Toxikon, Mr. Ouellette has been working in the inorganics department, primarily assisting in developing the CLP level documentation capacity of the laboratory including special documentation protocols, i.e. NYSDEC ASP. He runs ICP, Graphite Furnace, and Flame AA instruments.

1989 - 1991

Stevens Analytical labs, Stoneham, MA

Mr. Ouellette was an inorganics prep technician. He prepared various tests following EPA guidelines on drinking water wastewater, solids and sludge. His experience includes metals prep and analysis, mercury prep and analysis, TCLP extractions, TPH by IR, oil and grease, bacteria, minerals, oxygen demand, phenols and solids. He aided in implementation of a comprehensive QA/QC plan.

08/08/96 08/08/96

REVISION:

002

VALENTINA ROTENBERG METALS ANALYST

M.S. Chemistry, Polytechnical Institute, Khabarovsk, Russia

1996 - Present

Toxikon Corporation, Bedford, Massachusetts

Since joining Toxikon, Ms. Rotenberg has been working in the inorganics department, primarily assisting in developing the CLP level documentation capacity of the laboratory including special documentation protocols, i.e. NYSDEC ASP. She runs ICP, Graphite Furnace, and Flame AA instruments. She has also worked in the extraction lab for inorganic analyses, including UV/VIS and FTIR Spectroscopy.

1989 - 1991

Energy & Environmental Engineering, Inc., Somerville, Massachusetts

Ms. Rotenberg was the Inorganic and Wet Chemistry Laboratory Supervisor. She did data processing and report preparation for commercial and CLP packages. She was responsible for QC, training and management of metals and wet chemistry analyses. She supervised the operation, trouble shooting, and maintenance of Leeman Labs, Inc., ICP PS1000AT, PE5100 Furnace AA, Cold Vapor Analyzer Model 400 and Wet Chemistry Equipment.

Senior Analytical Chemistry (1995) ICP Metals Analyst (1992 - 1996) AA Operator (1991 - 1996) Chemist (1989 - 1991)

1975 - 1988

State R&D Institute of Polymer Plastic, St. Petersburg, Russia Department of Environmental Protection & Toxicology Sr. Research Scientist

DATE HIRED:

06/27/94 [08/15/88 - 08/21/90)]

RESUME DATE:

10/15/96

REVISION:

002

CAROL LEEDOM WET CHEMISTRY SUPERVISOR

M.S. 1988 Water Resource Sciences, University of Michigan, Ann Arbor, MI

B.S. 1980 Environmental Science, Purdue University, West Lafayette, IN

1994 - Present/ 1988 - 1990 Toxikon Corporation, Bedford, Massachusetts

As the Wet Chemistry Supervisor, Ms. Leedom is involved in extraction and preparation of samples for inorganic analyses. She handles a wide range of inorganic analyses, e.g. UV/Vis and FTIR Spectroscopy for GLP protocols under EPA methodologies. Ms. Leedom is responsible for scheduling the tests and oversees the work of the inorganic extractions department staff.

1983 - 1985

University of Michigan, Ann Arbor, Michigan

As a Research Assisting in the Department of Environmental Engineering, Ms. Leedom assisted with gel chromatography, collected, extracted and prepared natural surface water and landfill drainage samples for GC/MS analyses and assisted with rate studies of priority pollutants onto sediments and DOC.

DATE HIRED: RESUME DATE: REVISION: 10/07/96 10/07/96 000

> YA-LING KING WET CHEMIST

B.A., Taiwan Normal University, Taiwan

1996 - Present Toxikon Corporation, Bedford, Massachusetts

As a Wet Chemist, Ms. King is involved in extraction and preparation of samples for inorganic analyses. She handles a wide range of inorganic analyses, e.g. UV/Vis and FTIR Spectroscopy for GLP protocols under EPA methodologies.

1994 - 1996 Thermo Analytical, Waltham, Massachusetts

Ms. King was the Lead Analytical Chemist responsible for wet chemistry parameters using EPA, CLP, SW-846 and RCRA methodologies. She prepared and analyzed samples for parameters including cyanide, phenol, total organic carbon (TOC), total organic halogens (TOX), chloride, fluoride, alkalinity, sulfate, hexavalent chromium, residual chlorine, total solids (TS), total dissolved solids (TDS), total suspended solids (TSS), total volatile solids (TVS), total phosphorous ortho-phosphate, total kjeldahl nitrogen, ammonia, nitrate/nitrite, chemical oxygen demand (COD), total petroleum hydrocarbons (TPH), turbidity and pH.

1986 - 1993 Enseco - Erco Laboratory, Cambridge, Massachusetts

As an Analytical Chemist for the Wet Lab, Ms. King performed samples in various matrices (e.g. water, soil and sludge) as well as metals digestion for furnace, ICP and mercury analysis. She was responsible for review of inorganic data.

10/15/91 06/27/95 002

REVISION:

ARTHUR SWEENEY METALS ANALYST

B.S. Science, 1990, Salem State College, Salem, Massachusetts

1991 - Present

Toxikon Corporation, Woburn, Massachusetts

Since joining Toxikon, Mr. Sweeney has been working in the inorganics department, primarily assisting in developing the CLP level documentation capacity of the laboratory including special documentation protocols, i.e. NYSDEC ASP. He runs ICP, Graphite Furnace, and Flame AA instruments. He has also worked in the extraction lab for inorganic analyses, including UV/VIS and FTIR Spectroscopy.

1989 - 1991

ENSR/AnalytiKEM, Wilmington, MA

Mr. Sweeney worked in the inorganics department using Perkin-Elmer, Graphite Furnace, and flame AA. He used various Standard and EPA methods.

10/23/95 09/29/97

REVISION:

001

JEFF REGAN METALS PREP TECHNICIAN

B.A. Environmental Science, Salem State College, Salem MA (1990)

1997- Present

Toxikon Corporation, Bedford, Massachusetts

As a Metals Prep Technician, Mr. Regan preps water, waste water and soil samples for instrument analysis. He cleans glassware and is responsible for sample documentation.

1992 - 1995

Thermo Analytical, Waltham, Massachusetts

As a Chemist in the Metals Preparation department, Mr. Regan performed mercury prep and automated analysis, TCLP extractions, microwave digestion for biota samples and was responsible for maintenance and upkeep of DI water system.

1020

Deveraux Beach, Marblehead, MaSsachusetts / Dane Street Beach, Beverly, Massachusetts

Mr. Regan did a geological study to compare depositional and accretionary beaches, the greenhouse effect and its impact on water supply.

09/09/91 10/15/96

REVISION:

002

XIAOCHUN LIU GC/MS ANALYST

B.S. Chemistry, Beijing Polytechnic University, Beijing, China (1992)

M.S. Chemistry, Northeastern University, Boston, MA (1988)

1991 - Present

Toxikon Corporation, Bedford, Massachusetts

In this position, Ms. Liu is responsible for the operation of the duplicate Hewlett Packard instruments. She assists with training, technical administration and overseeing for all organic analyses and trace contaminates. Responsibilities include the coordination with other disciplines in the laboratory including extraction, report preparation and Quality Assurance.

1988 - 1991

Northeastern University, Boston, Massachusetts

As a Research Assistant for the Department of Chemistry and Barnett Institute of Analysis and Materials Science, Ms. Liu used ICP to do metal analysis, assisted with wet chemistry procedures and used HPLC to separate dithiophosphate compounds. She was also Teaching Assistant for physical and general chemistry laboratory courses.

1982 - 1986

Beijing Polytechnic University, Beijing, China

As a Research Assistant for the Department of Chemistry and Environmental Science, Ms. Liu performed environmental wastes analysis using GC. She also instructed and supervised analytical chemistry lab courses as a Lecturer.

DATE HIRED: RESUME DATE: REVISION: 02/27/95 06/27/95 001

CHRISTINA DILORENZO GC/MS ANALYST

B.S. Chemistry, UMass*Dartmouth, North Dartmouth, MA

1995 - Present

Toxikon Corporation, Woburn, Massachusetts

In this position, Ms. DiLorenzo is responsible for performing organic analyses in the GC/MS department under SOP, GLP and EPA guidelines for all soil and water samples. The department handles both volatiles and semi-volatiles analysis on HP instruments.

1994 - 1995

Analytical Laboratory, Inc., Somerville, Massachusetts

Ms. DiLorenzo analyzed groundwater samples using a modified EPA method, assisting in development of soil analysis.

05/01/95 056/26/965

REVISION:

001

JEFFREY MAXWELL MIS MANAGER

B.S.B.A. Business Management and Operations, Fitchburg State College, Fitchburg,

1995 - Present

Toxikon Corporation, Bedford, Massachusetts

As MIS Manager, Mr. Maxwell is responsible for administering the Novell network. He assists with local area networks, including installation and troubleshooting of all hardware and software. He interfaces with the departments to provide programming support services for users. He oversees the LIMS system and is assisting with ISO-9000 certification.

1994 - 1995

Thom McAN, Worcester, Massachusetts

As Senior Technical Services Analysts, Mr. Maxwell supported the implementation of Unix based Point of Sale package. He was the administrator for three local area networks.

1988 - 1994

BASF Information Systems, Bedford, MA

As Technical Programmer, Mr. Maxwell formulated Lan/PC based solutions for manufacturing, marketing and quality assurance areas to increase production efficiency. He also was a Senior Analyst who created various support programs for customer service.

1987 - 1990

Multiple System Environments, Leominster, Massachusetts

Mr. Maxwell authored and sold PC based Laboratory Information Management Systems (LIMS).

1987 - 1988

GT Environmental Labs, Milford, New Hampshire

Mr. Maxwell was the MIS Manager who managed successful mainframe laboratory information management system optimization and upgrade. He developed enhancement programs for the Perkin-Elmer LIMS package and created purchasing policies and procedures.

1984 - 1987 Frequency Sources, Chelmsford, Massachusetts Mr. Maxwell was a Programmer/Analyst.

DATE HIRED:

12/04/95

RESUME DATE:

03/01/96

REVISION:

002

FRANK D'AGOSTINO SAMPLE CUSTODY SUPERVISOR, ENVIRONMENTAL CHEMISTRY & TOXICOLOGY

The Use, Limitations and Maintenance of North Respirator, North (1992)

Certificate Environmental Technology, Aquinas College, Newton, Massachusetts (1995)

40 Hour OSHA Training

29 CFR 1910-120

8-Hour Hazwoper Annual Refresher Certification, Aquinas College (1995)

Regulations Every Small Quantity Generator Should Know, Triumvirate Environmental (1996)

DEP Inspection Preparation, Triumvirate Environmental (1997)

1995 - Present

Toxikon Corporation, Bedford, Massachusetts

Mr. D'Agostino is the Sample Custody Supervisor for the Environmental Chemistry & Toxicology Divisions. For Toxicology, Mr. D'Agostino is responsible for the receipt and log-in of all *In Vivo* and *In Vitro* samples. He maintains protocol and confidentiality files, generates work order due tables and is responsible for test sample reconciliation at study completion as well as test sample returns for sponsor request. Mr. D'Agostino assists in customer service functions such as assisting clients with the correct way to complete test requisition forms, and advising clients for appropriate sample requirements.

- In the Environmental Chemistry Division, Mr. D'Agostino oversees log-in activities, bottle orders, courier services and sample disposal duties.
- - 1995

Hygiea Environmental Company, Newton, Massachusetts

Mr. D'Agostino was an intern at Hygiea where he worked in the Log-in department, as well as participating in field sampling, PLM analysis, indoor air quality, AA, PCM and asbestos

DATE HIRED: 06/11/97
RESUME DATE: 07/30/97
REVISION: 001

NICOLE CAIRNS GC ANALYST

B.S. Environmental Science, UMass*Amherst, Amherst, Massachusetts (1997)

1997 - Present

Toxikon Corporation, Bedford, Massachusetts

As a GC Analyst (VOA), Ms. Cairns is involved in the preparation and the analyses of volatile and semi-volatile samples for organic analyses. She maintains gas chromatography instrumentation. She processes data from integrators and HP computer data systems. She is responsible for analyses by the 8000, 6000 and 500 series.

1996-1997

-25

Maxymillian Technologies, Pittsfield, Massachusetts

Ms. Cairns prepared and analyzed samples of various matrices for an assortment of organic and inorganic environmental samples.

DATE HIRED:

08/21/94

RESUME DATE:

08/21/95

REVISION:

009

JEAN-PIERRE LUKENGU MUSANDA GC/MS ANALYST

B.A. Biochemistry, Suffolk University, Boston, Massachusetts (1988)

1996 - Present

Toxikon Corporation, Bedford, Massachusetts

In this position, Mr. Musanda is responsible for performing organic analyses in the GC/MS department under SOP, GLP and EPA guidelines for all soil and water analyses. The department handles both volatiles and semi-volatiles analysis on HP instruments. Mr. Musanda assists with extraction, report preparation and training chemists in operating the various instruments.

1987 - 1996

National Environmental Testing, Inc., Bedford, Massachusetts

(1992 - 1996)

As the G/MS Group Leader Data Reviewer, for the Volatile Organics Department, Mr. Musanda was responsible for processing, reviewing, validating and data entry of all results generated by the GC/MS Department. Mr. Musanda performed validation of all volatile sub-contracted works and acted as the technical liaison between the lab, the project coordinator and the customer.

(1989 - 1992)

As GC/MS Senior Analyst, Mr. Musanda was responsible for analyzing organic samples utilizing sophisticated GC/MS equipments and purge and trap techniques, instrument maintenance, troubleshooting and data process.

handling.

1992 - 1993

Morrison Knudsen, Watertown, Massachusetts

Mr. D'Agostino worked as a Foreman.

1991

Webster Services, Inc., Dorchester, Massachusetts

Mr. D'Agostino did site excavation work.

1991

Jeremiah Sullivan & Sons, Everett, Massachusetts

As a foreman, he supervised and assisted crews in excavation of water and sewer lines.

DATE HIRED: 11/16/87
RESUME DATE: 2/2/98
REVISION: 003

DAVID BLACKWELL ORGANICS EXTRACTION DEPARTMENT SUPERVISOR

B.S. Chemistry, University of Lowell, Lowell, Massachusetts (1986)

1987 - Present Toxikon Corporation, Bedford, Massachusetts

Since joining Toxikon, Mr. Blackwell has been working in the organics department, primarily running all organic extraction prep procedures, specifically methods 608, 625, 8270, 8080 and Herbicides. In addition, he is responsible for running various inorganic test methods under the direction of his supervisor. His responsibilities include record keeping, equipment calibration and preventive maintenance, documentation of data and special events that may affect data integrity. Mr. Blackwell is responsible for all shifts in the organics extraction department.

1987 Cambridge Analytical Associates, Bedford, Massachusetts

Mr. Blackwell spent approximately six months performing organic extracts, data entry and routine QA/QC.

11/17/97

REVISION:

000

CHRISTOPHER KNIGHT GC ANALYST

B.A. Zoology, University of New Hampshire, NH (1984)

Mass Spectral Interpretation conducted by Professional Analytical and Consulting Svcs. (1994)

Hewlett Packard Analytical Education for Operation of Hp-HPLC3D-DOS Chemstation. (1995)

1997 - Present

Toxikon Corporation, Bedford, Massachusetts

As a GC Analyst, Mr. Knight is involved in the preparation and the analyses of volatile and semi-volatile samples for organic analyses. He maintains gas chromatography instrumentation. He processes data from HP computer data systems. He is responsible for analyses by EPA 8000, 600 and 500 series methods and special projects.

1990 - 1997

Groundwater Technology Environmental Laboratories, Milford, New Hampshire

As a senior analytical chemist, Mr. Knight analyzed environmental samples by gas chromatography, mass spectrometry and high pressure liquid chromatography. He maintained instrumentation, calibration, quality assurance criteria, made standards and reviewed and validated reports according to certification regulations.

1988 - 1998

E.N.S.R. Consulting and Engineering, Wilmington, Massachusetts

Mr. Knight was an Environmental Analyst who analyzed environmental samples for organic contaminants by gas chromatography and mass spectroscopy. He supervised sample preparation for both organic and inorganic analysis. He analyzed extracts in compliance with EPA methodology and was involved with the development of new methods.

1987 - 1988

New England Baptist Hospital, South Boston, Massachusetts

Certified Laboratory Technician

 DATE HIRED:
 02/17/98

 RESUME DATE:
 02/17/98

 REVISION:
 000

PAULETTE POLK ANALYST, PRODUCT CHEMISTRY

B.S. Toxicology, Northeastern University, Boston, Massachusetts (1997)

1998 - Present

Toxikon Corporation, Bedford, Massachusetts

As Product Chemistry Analyst, Ms. Polk assists with the special chemistry laboratory. These activities include trace residue studies under FIFRA methodology and GLPs, method development and validation, and product chemistry. Product chemistry responsibilities include stability testing and physical chemical characterization of new chemicals and pesticides for EPA registration. She also assists in EPA priority pollutant analysis of organic compounds and herbicide analysis.

1997 - 1998

T.R. Wilbury Laboratories, Marblehead, Massachusetts

As Quality Assurance Officer, Ms. Polk performed in-life audits as well as audits of study protocols, biological and analytical chemistry raw data, draft reports and steps of corrective action for regulatory compliance as per EPA and FDA Good Laboratory Practices.

1996 - 1997

Krueger Food Laboratory, Cambridge, Massachusetts

As Senior Laboratory Technician, Ms. Polk conducted chemical analyses, including GC, HPLC and spectrophotometric enzymatic test procedures.

1996

Northeastern University, Boston, Massachusetts

Research Assistant, Toxicology Lab

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supervisory experience.

GC/MS/DS Operator

Christina DiLorenzo has a B.S. degree in chemistry and four years' experience in operating and maintaining GC/MS.

Systems Manager

Jeff Maxwell is responsible for the management of all computing systems. He has a B.S. degree in Business and thirteen years experience in systems management and programming.

Programmer Analyst

Jeff Maxwell is responsible for the installation, operation and maintenance of software. He has a B.S. degree and ten years! experience as a programmer.

Mass Spectral Interpretation Specialist

Xiaochun Liu has a M.S. degree in chemistry and six years' experience in GC/MS.

GC Laboratory Supervisor

Doug Sheeley is responsible for all technical efforts of the GC laboratory. He has a B.S. degree in chemistry, thirteen years' experience in operating a GC/EC and twenty-three years of supervisory experience.

Pesticide Residue Analysis Expert for Organochlorine Pesticides and

PCB's

Christopher Knight has a B.A. degree and nine years' experience in operating and maintaining GC and interpreting GC chromatograms.

Purge and Trap Volatile Organics Analysis Expert

Nicole Cairns has a B.S. degree in Environmental Sciences and two years' experience in GC volatile analysis.

Sample Preparation Laboratory Supervisor

David Blackwell has a B.S. degree in chemistry and ten years' experience in sampe preparation for organic analyses.

Sample Extraction / Concentration Specialist

David Blackwell has a B.S. degree in chemistry and ten years' experience in extraction / concentration.

Sample Custodian Frank D'Agostino is a high school graduate with two years' experience as a sample custodian.

Data Reporting and Delivery Officers

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1.3.2 Education and Experience Pertinent to This Protocol

Laboratory Supervisor

Douglas Sheeley has a M.S. degree and twenty-four years' experience in laboratory management.

Project Manager

Paul Lezberg has a B.S. degree and fourteen years' experience in managing projects.

Quality Assurance / Quality Control Officer

Alan Doughty has a PhD. in chemistry with fourteen years of environmental laboratory experience. He has written the Quality Assurance Programs for three different environmental laboratories.

ICP Spectroscopist

Brian Keith Ouellette is working towards his B.A. in chemistry. He has five years' experience as an ICP spectroscopist.

ICP Operator

Valentina Rotenberg has an M.S. degree in chemistry and 8 years experience as an ICP operator.

Flameless Atomic Absorption Spectroscopist

Arthur Sweeney has a B.S. degree in science and eight years' experience in AA Spectroscopy.

Flame and Cold Vapor Absorption Spectroscopist

Arthur Sweeney has a B.S. degree in science and eight years' experience in flame and Cold Vapor spectroscopy.

Inorganic Sample Preparation Specialist

Jeff Regan has a B.A. degree in environmental science and five years' experience in inorganic sample preparation.

Classical Wet Chemical Techniques Analyst

Carol Leedom has a M.S. degree in environmental sciences and five years' laboratory experience and three years' supervisory experience.

Inorganic Chemist (backup)

Ya-Ling King has a B.A. degree and eleven years' experience in wet chemistry and metals digestion.

GC/MS Laboratory Supervisor

Xiaochun Liu has a M.S. degree in chemistry and six years' laboratory experience in operating a GC/MS and four years'

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JP Musanda has a B.A. degree in biochemistry, four years' experience in data reporting and delivery and eight years' experience in GC/MS.

Organic Chemist (backup)
Christopher Knight has a B.A. degree and nine years' experience in GC and eight years' experience in GC/MS.

1.3.3 Training Progress

Toxikon's Laboratory training programs are designed to educate the employee in understanding and performing all tasks efficiently and competently as delineated in a particular departmental job description. Each employee must demonstrate proficiency and understanding of a task prior to unsupervised task execution. This will safeguard both quality performance and safety of the individual.

Toxikon provides ongoing training programs to train employees, in the technical component of their job requirement. In addition, higher level technical training may be required depending on the scope of the individual's position and level of understanding required.

External technical training programs including equipment training, software training, and other specifically required programs are made available through Toxikon Corporation. These training programs are offered at least annually or more frequently as required.

Ongoing training, On the Job Training (OJT), SOP revision retraining, and annual retraining for both external and internal training programs are provided at Toxikon.

Technical training is broken down into 2 components. This includes the following:

Core Curriculum Syllabus Job Specific Syllabus

The core curriculum consists of both active and descriptive SOP's that are generally required knowledge of an employee at Toxikon. Examples include Quality System SOP's, generic equipment calibration procedures,

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facility description and layout information, security issues, and general training procedures. This curriculum may be expanded or reduced at the discretion of Senior Management.

The job specific syllabus consists of Active Departmental SOP's that are required to perform the specific job or task as described in the individual job description. This syllabus generally includes test specific methodology and equipment SOP's, in addition to environmental monitoring and other position specific tasks.

Laboratory training is a discrete function internally provided by the direct Manager/Supervisor and/or his or her designee. All aspects of technical training are documented and acknowledged by the trainer and the trainee. All training documentation is filed in each employee's training file.

Forms used to document training include:

Laboratory Training Documentation: Organic Extraction Curriculum (Figure 1-3)

Laboratory Training Documentation: GC Curriculum (Figure 1-4)

Laboratory Training Documentation: GC/MS Curriculum (Figure 1-5)

Laboratory Training Documentation: Metals Curriculum (Figure 1-6)

Laboratory Training Documentation: Log-In Curriculum (Figure 1-7)

Laboratory Training Documentation: Quality Assurance Curriculum (Figure 1-8)

Laboratory Training Documentation: Glassware Prep Curriculum (Figure 1-9)

Laboratory Ongoing Training Documentation / Additional Documentation (Figure 1-10)

Laboratory SOP Retraining Documentation/OJT/Revision (Figure 1-11)

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Figure 1-2

Technical Training Documentation: Wet Chemistry Curriculum

Employee:	
osition:	
Date of Hire:	

SOP number	Training Date	Instructor Signature	Employee Signature	SOP Title
7.3				Document Control Procedures
7.6				Significant Figures
7.7				Data Validation
7.8				Quality Control Review and Corrective Action
7.10				Review of Technical Data
7.12				Reagent Inventory, Traceability, Labeling, and Acceptability
7.16	î	*		Data Recording and Storage Maintenance
7.17				Standard Preparation and Traceability of Primary Standards Against Working Standards
7.19				Glassware Preparation
7.20				Control Charting
7.21				Prevention of Sample Contamination
7.23				Batch QC Reporting and Numbering System
7.25				Documentation for Single Analyst Precision and Accuracy
7.26				Environmental Chemistry Technical and Contract Policies
3.5				Balances
3.2.5		*		Waterbath / Incubator Maintenance
2.8				Personnel Attire

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SOP number	Training Date	Instructor Signature	Employee Signature	SOP Title
3.2.2				Lab Ovens
3.4.1				Lab Ovens
7.2.3				Batch QC Reporting & Number
8.5				Soil Dry Weight Determination
11.4				Method Detection Limits
7.12				Reagent Inventory & Labeling
7.10				Review of Technical Data
3.5.6				Mettler AE 163 Balance
3.2.9				Precision Scientific Flashpoint Tester
3.2.11				BOD Incubator
3.3.1				Refrigerators
3.7.11				YSI DO Meter
3.8.18		1		PHM62 Selective Ion Meter
3.8.22				Corning 320 pH Meter
3.8.42				Alpkem RFA 300
3.8.43				PHE Model 1310 IR
3.8.44				HACH 2100 A Turbidimeter
3.8.63				Ol Model 700 TOC Analyzer
1.6.38		1.	T-47	In-house Hazardous Waste Storage
7.19				Glassware Washing
3.8.36	_			Pipet Washing
3.8.79				COD Reactor
3.8.65				Tecator TKN Digestion Block
1.6.13				Care & Use of Face Masks
11.5.3				Acidity
11.5.5				Alkalinity
11.32				Ammonia

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SOP number	Training Date	Instructor Signature	Employee Signature	SOP Title
11.3.2				Ammonia distillation
11.45				Ash
11.7				BOD
11.51				Chloride
11.24				Total Residual & Free Chlorine
11.23				Chlorophyll
11.14				Chromium - hexavalent
11.52				COD
11.27				Color
11.21				Conductance
11.25				Corrosivity - Water
11.13				Amenable Cyanide
11.13	· -			Cyanide
11.12				Cyanide - Automated
11.30	· · · · · · · · · · · · · · · · · · ·			Flashpoint
11.18				Fluoride via Electrode
11.50				Hardness
11.6				IR Scan
11.22				lodide
3.8.46				Karl Fisher % Moisture
8.5				% Moisture
11.16				Nitrate
11.28				ODOR
11.19-20				Oil & Grease Gravimetric
11.19-20				Oil & Grease Hexane
11.19-20				Oil & Grease by IR
11.10/11 .32				Organic Nitrogen

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SOP number	Training Date	Instructor Signature	Employee Signature	SOP Title
11.17				pH - soil
11.31				Phenol Manual
11.59				Paint Filter
11.17				Ph - Water
11.33				Ortho-Phosphorous - automated
11.33			•	Total Phosphorous - digestion
11.26				Reactive Cyanide
11.26				Reactive Sulfide
11.58				Settable Solids (SS)
8.5				% Solids
11.49				Silica
11.56				Sulfate
11.4.3				Sulfide Titrimetric
11.57		,		Sulfite
11.15				Surfactants
11.46				Total Dissolved Solids (TDS)
11.10			<u>,</u>	Total Kjeldahl Nitrogen (TKN)
11.9				Total Organic Carbon (TOC) water
11.9				Total Organic Carbon (TOC) soils
11.10				Total Nitrogen
11.19-20				Total Petroleum Hydrocarbons (TPH) soils
11.19-20				Total Petroleum Hydrocarbons (TPH) water
11.42				TSP/PM10 Air Filter Analyses
11.47				Total Solids (TS)
11.60				Total Suspended Solids (TSS)

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SOP number	Training Date	Instructor Signature	Employee Signature	SOP Title
11.29				Turbidity
11.45				Total Volatile Solids (TVS)
11.48				Volatile Suspended Solids (VSS)
11.41				Winkler Titration
<u> </u>				
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Figure 1-3

Technical Training Documentation: Organics Extraction Curriculum

Employee:	
Position:	
Date of Hire:	

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
7.3				Document Control Procedures
7.6				Significant Figures
7.7				Data Validation
7.8				Quality Control Review and Corrective Action
7.10				Review of Technical Data
7.12			•	Reagent Inventory, Traceability, Labeling, and Acceptability
7.16				Data Recording and Storage Maintenance
7.17				Standard Preparation and Traceability of Primary Standards Against Working Standards
7.19				Glassware Preparation
7.20			_w	Control Charting
7.21	-		_,	Prevention of Sample Contamination
7.23				Batch QC Reporting and Numbering System
7.25				Documentation for Single Analyst Precision and Accuracy
3.5				Balances
3.2.5				Waterbath / Incubator Maintenance
2.8				Personnel Attire
3.2.2				Lab Ovens
3.4.1				Lab Ovens

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Technical Training Documentation: OE Curriculum Con't.

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
1.5.4.1				Error Correction
3.2.2				Laboratory Oven Use
3.2.5				Incubator / Waterbath Use
3.3.1				Refrigerator/ Freezer Use
3.8.8				Operation of the MODULAB Analytical Research Grade UF/ Polishing System
3.8.22				Calibration, Operation and maintenance of the Corning 320 Ph Meter
3.8.22.1				Calibration, Operation and Maintenance of the Corning M90 Ph Meter 3.8.22.1
3.8.72				Operation of the USE Filter Ro/DI Water System
8.5			*	Soil Dry Weight Determination
11.1		7		TCLP Extract Preparation
11.4				Definition and Procedure for the Determination of Method Detection Limits
11.62				Continuous Liquid-Liquid Extraction for 600 Series Methods
11.63				Liquid-Liquid Extraction (EPA method 3510)
11.64				Continuous Liquid to Liquid Extraction of Non- and Semi- Volatile Organic Compounds (EPA Method 3520)
11.65				Ultrasonic Extraction of Non- and Semi- Volatile Organic Compounds (EPA Method 3520)
11.69				Method 3540B Soxhlet Extraction
11.70				Method 3660A Sulfur Cleanup
3.1.1				Equipment Suitability and Maintenance
3.1.1.1				Preventive Maintenance Logs
3.1.2				Equipment Calibration General Procedure

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Technical Training Documentation: OE Curriculum Con't.

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
3.2.7				Precision Thelco Drying Oven
3.5.2				Operation and Maintenance of Ohaus Electronic Balance, Model GT4111, SN#7409
3.5.6				Operation and Maintenance of Mettler AE 163 Balance, SN B54935
3.7.33				HP 5890 Series li Gas Chromatograph
3.8.48				Damon / IEC Division Centrifuge, Model CU 5000
3.8.54				Rotary Extractor
3.8.56				Branson Ultrasonic Clearer
3.8.57				Waring Blender
9.12				pН
11.5		,		Pesticides PCBs Analysis by Gas Chromatography
1.5.1				Total Petroleum Hydrocarbon Analyses by Gas Chromatography
11.5.3				Polynuclear Aromatic Hydrocarbons by HPLC Method 9310
11.5.9				Petroleum Hydrocarbons by Modified Method 8015 (DRO)
11.5.11				Extractable Petroleum Hydrocarbons (EPH)
11.34				Organochlorine Pesticides and PCBs (Methods 608 and 8080)
11.36				Chlorinated Herbicides
11.67				Analysis of Aqueous, Soil, Sludge or Solid Samples for Semi-volatile Organics by GC/MS (8270)
7.26				Environmental Chemistry Technical and Contract Policies

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Figure 1-4

Technical Training Documentation: Gas Chromatography Curriculum

Employee:	
Position:	
Date of Hire:	

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
7.3				Document Control Procedures
7.6				Significant Figures
7.7				Data Validation
7.8				Quality Control Review and Corrective Action
7.10				Review of Technical Data
7.12		7	•	Reagent Inventory, Traceability, Labeling, and Acceptability
7.16		7		Data Recording and Storage Maintenance
7.17				Standard Preparation and Traceability of Primary Standards Against Working Standards
7.19				Glassware Preparation
7.20				Control Charting
7.21				Prevention of Sample Contamination
7.23				Batch QC Reporting and Numbering System
7.25				Documentation for Single Analyst Precision and Accuracy
3.5				Balances
3.2.5				Waterbath / Incubator Maintenance
2.8				Personnel Attire
3.2.2				Lab Ovens
3.4.1				Lab Ovens

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Technical Training Documentation: GC Curriculum Con't.

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
1.5.4.1				Error Correction
3.2.2				Laboratory Oven Use
3.3.1				Refrigerator/ Freezer Use
3.7.4				Installation, Operation and Maintenance of the Tracor 540 Gas Chromatograph
3.7.32				Operation and Maintenance of the Perkin Elmer AutoSystem Gas Chromatograph
3.7.33				Operation and Maintenance of the Hewlett Packard 5890 Series II Gas Chromatograph
3.7.35				Installation, Operation and Maintenance of the Tremetrics 9000 Gas Chromatograph
3.8.3		r	4.	Operation of the MODULAB Analytical Research Grade UF/Polishing System
3.8.49				TEKMAR LSC 2000 & ALS 2016
3.8.72				Operation of the US Filter RO/DI Water System
7.9				Compilation of Completed Data Package
7.26				Environmental Chemistry Technical and Contract Policies
11.4				Definition and Procedure for the Determination of Method Detection Limits
11.5				Pesticide/PCB Analysis by Gas Chromatography
11.5.1				Total Petroleum Hydrocarbon Analysis by Gas Chromatography
11.5.2				Analysis for Halogenated Volatile Organics by Gs Chromatography EPA Method 8010
11.5.2.1				Analysis for Halogenated Volatile Organics by Gas Chromatography EPA Method 8020
11.5.4				Analysis of Halogenated/Aromatic Organics by Gas Chromatography - EPA Method 601

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Technical Training Documentation: GC Curriculum Con't.

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
11.5.4				Analysis of Halogenated/Aromatic Organics by Gas Chromatography - EPA Method 601
11.5.4.1				Analysis of Volatile Aromatic Organics by GAS Chromatography - EPA Method 602
11.5.5				Analysis for Halogenated/Aromatic Volatile Organics by Gas Chromatography using EPA Methods 502.1/502.2/503.1
11.5.6				Analysis for Halogenated/Aromatic Volatile Organics by Gas Chromatography using Method 8021
11.5.8				Total Petroleum Hydrocarbon Analysis by Gas Chromatography - EPA Modified Method 8015 (Gasoline Range Organics)
11.5.9		F		Total Petroleum Hydrocarbon Analysis by Gas Chromatography - EPA Modified Method 8015 (Diesel Range Organics)
11.5.10				Analysis of Volatile Organic Compounds by Gas Chromatography EPA Method TO-3 (Modified)
11.5.12				Volatile Petroleum Hydrocarbons by GC
11.5.11				Extractable Petroleum Hydrocarbons (EPH)
11.36				Chlorinated Herbicides
7.26				Environmental Chemistry Technical and Contract Policies

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Figure 1-5

Technical Training Documentation: GC/MS Curriculum

Employee:	
Position:	
Date of Hire:	

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
7.3				Document Control Procedures
7.6				Significant Figures
7.7				Data Validation
7.8	·			Quality Control Review and Corrective Action
7.10				Review of Technical Data
7.12			4.	Reagent Inventory, Traceability, Labeling, and Acceptability
7.16		,		Data Recording and Storage Maintenance
7.17				Standard Preparation and Traceability of Primary Standards Against Working Standards
7.19				Glassware Preparation
7.20				Control Charting
7.21				Prevention of Sample Contamination
7.23				Batch QC Reporting and Numbering System
7.25				Documentation for Single Analyst Precision and Accuracy
3.5				Balances
3.2.5				Waterbath / Incubator Maintenance
2.8				Personnel Attire
3.2.2				Lab Ovens
3.4.1				Lab Ovens

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Technical Training Documentation: GC/MS Curriculum Con't.

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
1.5.4.1				Error Correction
3.2.5				Incubator/Waterbath Use
3.3.1				Refrigerator/ Freezer Use
3.5.13				Calibration Weights for Laboratory Balances
3.5.19				Operation and Maintenance of Denver Instrument Company XE Series, Model 400 Balance, SN 43076
3.7.30				HP GC/MS System for Analysis of Volatiles
3.7.31				HP GC/MS System for Analysis of Volatiles
3.8.8				Operation of the MODULAB Analytical Research Grade UF/Polishing System
3.8.52				Brinkman Dispensate
3.8.72				Operation of the US Filter RO/Di Water System
7.4				Preventative Maintenance Log, Column Log and Equipment Log Procedures and Description
7.9				Compilation of Completed Data Package
7.26				Environmental Chemistry Technical and Contract Policies
11.4				Definition and Procedure for the Determination of Method Detection Limits
11.3.5				Analysis of Purgeable Organics by GC/MS EPA Method 624
11.66				Analysis of Aqueous, Soil, Sludge or Solid Samples for Volatile Organics by GC/MS (8240A)
11.67				Analysis of Aqueous, Soil, Sludge or Solid Samples for Semi-Volatile Organics by GC/MS (8270A)
11.68				Analysis of Volatile Organics by GC/MS EPA Method 8260A

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Figure 1-6

Technical Training Documentation: Metals Curriculum

Employee:	
Position:	
Date of Hire:	

SOP	Training Date	Instructor Signature	Employee Signature	SOP Title
7.3				Document Control Procedures
7.6		:		Significant Figures
7.7				Data Validation
7.8				Quality Control Review and Corrective Action
7.10				Review of Technical Data
7.12		-	* 14	Reagent Inventory, Traceability, Labeling, and Acceptability
7.16		7		Data Recording and Storage Maintenance
7.17				Standard Preparation and Traceability of Primary Standards Against Working Standards
7.19				Glassware Preparation
7.20				Control Charting
7.21	_			Prevention of Sample Contamination
7.23				Batch QC Reporting and Numbering System
7.25	• · ·			Documentation for Single Analyst Precision and Accuracy
3.5				Balances
3.2.5				Waterbath / Incubator Maintenance
2.8				Personnel Attire
3.2.2				Lab Ovens
3.4.1				Lab Ovens

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Technical Training Documentation: Metals Curriculum Con't.

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
1.5.4				Laboratory Record Keeping
1.5.4.1				Error Correction
3.1.1				Equipment Suitability and Maintenance
3.1.1.1			•	Preventive Maintenance Logs
3.1.2				Equipment Calibration General Procedure
3.3.1				Refrigerator/ Freezer Use
3.5.13				Calibration Weights for Laboratory Balances
3.5.17				Use and Maintenance of the Ohaus GT410 Balance
3.7.13			•	Operation and Maintenance of the Leeman Plasma-Spec ICP
3.7.29		,		Operation and Maintenance of the Perkin Elmer 5100 PC with the Zeeman 5100/HGA 600 Graphite Furnace Accessory
3.7.34				Perkin Elmer 4100 FIAS Automated Cold Vapor Atomic
3.8.5				Pipet Calibration
3.8.8				Operation of the MODULAB Analytical Research Grade UF/Polishing System
			_	

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Figure 1-7

Technical Training Documentation: Sample Log-in, Environmental Chemistry Curriculum

Employee:	
Position:	
Date of Hire:	

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
7.1				Sample Receipt and Security for Chemical Analysis
7.2				Specific Preservation and Storage of Samples
7.21				Prevention of Sample Contamination
3.3.1				Refrigerator/Freezer Use
7.26	*	Environmental Chemistry Technical and Contract Policies		
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Figure 1-8

Technical Training Documentation: Quality Assurance Environmental Chemistry Curriculum

Employee:	
Position:	
Date of Hire:	

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
1.5.2				Development of Standard Operating Procedures
1.5.2.1				Content of Standard Operating Procedures
1.5.2.10				Procedures for Writing, Reviewing, and Distribution of SOP's
7.8				Quality Control Review and Corrective Action
7.9		. 17	*	Compilation of Completed Data Package
7.10		I		Review of Technical Data
7.20				Control Charting
7.21				Prevention of Sample Contamination
7.23			· · · · · · · · · · · · · · · · · · ·	Batch QC Reporting and Numbering System
7.26				Environmental Chemistry Technical and Contract Policies
 		1		

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Figure 1-9

Technical Training Documentation: Glassware Preparation Curriculum

Employee:	
Position:	
Date of Hire:	

SOP Number	Training Date	Instructor Signature	Employee Signature	SOP Title
7.19				Glassware Preparation
3.2.7				Precision Thelco Drying Oven
3.8.8				Operation of the MODULAB Analytical Research Grade UF/Polishing System
3.8.72			**	Operation of the US Filter Ro/DI Water System
1.5.4.1		,		Error Corrections
-				
-				

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Figure 1-10				
Laboratory Ongoing	Training	Documentation/	Additional	Documentation

Employee: _	
Position:	

SOP#	Title of SOP	Instructor Signature	Employee Signature	Date
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Figure 1-11

Laboratory SOP Retraining Documentation/OJT/Revision*

Employee:	
Position:	

SOP#	Title of SOP	Instructor Signature	Employee Signature	Date
		Í		
	1	<u> </u>		
		7		

^{*} Attach all OJT and copies of Retraining Compliance Forms

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2. Facilities and Equipment

2.1 Instrumentation and Backup Alternatives

The instrumentation currently in the Toxikon environmental laboratory is listed in Table 2-1.

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TABLE 2-1
INSTRUMENTATION: INORGANIC LABORATORY

Instrument & Model	Manufacturer	Number
Balances		_
AE 163	Mettler	1
L420P	Sartorius	1
Centrifuges	Beckman	1
	IEC	1
Fume Hoods	Hemco	2
	Gray	1
	Kewaunee	2
Furnace, 6000 Muffle	Thermolyne	1
Heating/Stir Plates		
Type 3000	Thermolyne	1
Mantles	Glascol	30
	Thermowell	1
	Corning	5
Meter,		
Turbidity, 2100A;	Hach	1
Dissolved Oxygen, 52A	YSI	1
pH, 140	Corning	1
PHM 62		1
Specific Conductance	Fisher 152	1
Oven	VWR Model 1305U	2
Refrigerator/Freezer		_
$4(+/-2)^{0}C$ and $-20(+/-2)^{0}C$	Labline	1
Spectrophotometer		_
Visum, automated	Alpkem	1
Spectrophotometer	Gilford	1
- FTIR	Perkin Elmer	1
UV/VIS	Perkin Elmer	1
Pipet Aid		1
TKN Digestor	Tecator	1
TOC Analyzer	OI Analytical Instruments	1
TOX Analyzer	Mitsubishi Kasei Corp.	1
BOD Incubator	Fisher Scientific	1
Flash Point	Precision Scientific	1

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TABLE 2-1 INSTRUMENTATION: ORGANIC LABORATORY

Instrument & Model	Manufacturer	Number
Cleaner, Orchabonio	Branson	1
Detector Gas Chromatograph		
5890 (Dual ECD)	Hewlett Packard	2
5890 (Dual FID)	Hewlett Packard	3
Data Handling System	P.E., HP 3365	6
540 (FID/PID PET)	Tracor	1
5890 (FID/PID PET)	Hewlett Packard	2
Purge and trap, LSC-2	Tekmar	1
LSC 2000	Tekmar	1
LSC 3000	Hewlett Packard	1
PET 9000	Tekmar	1
ECD/NPD 5890	Hewlett Packard	1
Gas Chromatograph/Mass Spectromete		
Purge and trap, 4200	Tekmar	1
Purge and trap, LSC-2	Tekmar	1
HP 5890B ?	Hewlett Packard	2
Purge and Trap LSC-2000/ALS 2016/		1
วักรว		
5971 (with Unix Comp. System)	Hewlett Packard	1
5970 Mass Spec	Hewlett Packard	4
7673 Auto Injection Tower	Hewlett Packard	1
-cos auto Injection Mower	Hewlett Packard	1
some numble with Durge and Tran	Hewlett Packard	1
coso punalo with Auto Ini Tower	Hewlett Pathalu	1
cc/wc equipped with in-house libi	ary search factific	(75,000
compounds in library from manufac	cturer)	÷
High Performance Liquid Chromatograp	on	1
1050	Hewlett Packard	1
Refrigerator/Freezer	Frigidaire	1
	Avanti	2
4(T)-2) C UNG 20(') -) -		1
$4(+/-2)^{\circ}C$ and $-20(+/-2)^{\circ}C$	Kelvinator Hewlett Packard	2
Integrator, 3392A	Hewlett Packard	10
3396A	Shimadzu	. 1
	Precision 186	1
Waterbath	Vortex	1
Mixer Mixer	Glascol	1
Heating/Stir Plates	0143001	_

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Balance Tare XC Series 400

Denver Instrument Co. 1

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TABLE 2-1 (con't) INSTRUMENTATION: ORGANIC LABORATORY

Instrument & Model	Manufacturer	Numbe
Recorder	Fischer	1
Series 5000	Goerz	1
Electro	Kipp & Zone	1
SD 40	P.E.	1
024 056-3007	1.4.	1
TCLP Zero Headspace Extractors for \	OAs (ZHE) and filters	8
Telle Belo heddspace Bhol access 100	,	
INSTRUMENTATION:	METAL LABORATORY	
Instrument & Model	Manufacturer	Numbe
Vacuum pumps		1
Dispensette	Brinkmann	2
D.I. Water System	Modulab	1
Heating/Stir Plates	•	
	Thermolyne	3
Pipet Aid		1
Spectrophotometer		
Inductively Coupled Plasma Emi	.ssion	1
ICP 2.5	Leeman	1 1
Enviro I/61E (with Autosampler)	Thermo Jarrell Ash	1
3100	P.E. P.E.	1
5100 PC	P.E.	8
TCLP Extractor (Share with OE)		_
Waterbath	Precision	1
Balance	Ohaus GT410	1
		

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2.2 Maintenance Activities and Schedules

A comprehensive program of maintenance, upgrading and refurbishing is a necessary component in the daily operation of the laboratory. Each piece of equipment is listed on a master log (maintained by the QAU) and this record is used to schedule routine maintenance. When scheduled maintenance is due, the equipment is taken off-line. Maintenance is routinely conducted during non-working hours and on weekends, if possible. Each operator is responsible for basic equipment maintenance and record keeping to achieve performance requirements within the laboratory.

A preventive maintenance equipment log exists for each piece of equipment in the laboratory. Standard operating procedures (SOPs) are used to describe equipment operation and calibration procedures. Preventive maintenance logs are separate from SOPs and list the following:

Equipment Name:
Iodel Number:
Serial Number:
lanufacturer:
ate of Receipt:
ocation:
esponsible Personnel:
lternate Personnel:

These records are maintained (including a record of service provided by contractors) by the operator. When a book is completed, it is archived by the QAU; a new book is then issued.

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Table 2-2

Routine Preventive Maintenance (Metals)

Instrument	Activity	Frequency
ICP	Clean nebulizer and change pump tubing. Rinse water recirculation	About every two weeks or as needed. Every 6 months or as needed.
	Oil peristaltic pump	Monthly or as needed.
AA (CVAA manual)	Clean cell windows Change desiccant	Daily (each use) Daily
	Check alignment	Daily
GFAA	Clean Furnace Windows Check tubes Check tip alignment Check lamp alignment	Daily (each use) Daily (each use) Daily (each use) Daily (each use)

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Table 2-3

Preventive Maintenance (GC/MS)

- Instrument	Activity	Frequency
GC/MS Monthly	Change Septum	Semi-VOA=Daily/VOA =
	Check Carrier Gas	Daily
	Check all other gase	s Daily
	Change in-line filte	cs Quarterly or as needed
	Change carriers and other gases pressur	all As needed(when tank e reaches 50 psi)
	Change first 1/4" of column packing & gla	Bi-monthly or as needed
	Remove first meter o capillary guard colu	
	Check system for gas & tank	leaks At each column maintenance change
± ************************************	leaks	um At each column maintenance & general maintenance lving venting of system
	Clean volatile auto sampler glass tubes	As needed
	Change Injection porglass insert	As needed
	Change Injection porglass wool	Daily

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Table 2-3 (Cont.)

Preventive Maintenance (GC/MS)

Instrument	Activity	Frequency
GC/MS	Check Autosampler syringe	Weekly
	Change Autosampler syringe	As needed
	Check and refill Autosampler rinsing solution vials	Daily
	Change packed column	Quarterly or as needed
	Change capillary guard & column connector	As needed (when $)$
	Change & clean jet separator	As needed
	Change vacuum pump oil	Quarterly/Semi-annually
	Change capillary column	As needed
	Clean volatile LSC-2000 valves & lines	Weekly or as needed
	Clean injection port/ capillary column adaptor	Monthly or as needed
	Back up memory cartridges onto tape cartridges	As needed
	Printer maintenance	Monthly or as needed

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Table 2-4
Preventive Maintenance (GC)

Instrument	Activity	Frequency
GC	Change Septum	Semi-VOA=Daily/VOA =Monthly
	Check Carrier Gas	Daily
	Check all other gases	Daily
	Change in-line filters	Quarterly or as needed
	Change carriers and all other gases	As needed (when tank pressure reaches <500psi)
	Change first 1/4" of column packing & glass wool	Bi-monthly or as needed
	Remové first meter of capillary guard column	As needed
	Check system for gas leaks	At each column maintenance & tank change
-	Clean volatile auto sampler glass tubes	As needed
1 15 15.4	Change packed column	Quarterly or as needed
·	Change capillary guard & column connector	As needed (when $\leq 1m$)
	Change capillary column	As needed
	Clean volatile LSC valves & lines	Weekly or as needed
	Clean injector port/ capillary column adaptor	Monthly or as needed

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Figure 2-2

WASTE STREAMS

Containag	65:140	100	 HE-GL	11-01,	Pcu-LP	V1M.S		11g-1.p	AGTD	PYR-1105	10	-
Bulk Storago Containur	55 gal' - nor1711	25 941 - DOT172	55 yal - DOT17E	55 gal nor17E	101 - 105 gg 1 - 100 134 gg 1 - 105 gg	55 941 BOY 34	104	5 gal - porta	55 yal - nor34	55 gal - nor34	55 gal - DOTITE	25 gal - 60x12r
Sourca of Gungral.Lon	Piald Complan	Fluid Sampton	Spairt Lab Sulvant	Spand. Lab Solvane Hon chlucinalud	Lab Samplan Fluld Samplan	Lab Samptan da)	Broken Thermometer	Fluft Samplun	եռի Ձոտրիսո	Lah Samplun	Spant Lab Solvant	Spant Lab Bolvent
Bonczipilon of Vanta	Mantu Soll - FP>140, Hiso. Contaminants "Spundy-Dry"	011	Huchylena Chlaclda	Hukann, Acatona, Ethur, Ethanol, Hathanol, Ethyl Acutata	PCH'u < 50 mg/kg Sullu and Ollu	Sustanta Charles Chilas Chilorida)	Burency	Aubauton and other Higgellangoun Maclan	Utria, sui furia, Undrachioria	Phosphorts Asid Sodium Hydruxida Solution	Mixed Chlorinatud Solvanta- DGH, Frann, Chloroform	Fraon
Hanks	Oll Nauro	Statu Nugulated Oli Hautu	Vantu Nathylene Chloridu est	Wasta Flaumahku Liguld, H.O.S.	Hazardoun Substancos, N.O.S. Wasto Identification Porm required.	Hazardina Substances, R.O.S.	Mante Poluon II	Hata Solid, R.O.S.	Naken Acid, 11.0.5. Liquid	Nautu Phouphoric Acid Solution	Hanta Chlorinated Solventa	Uantu Pruon
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Figure 2-3

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DEPARTMENT OF ENVIRONMENTAL PROTECTION
DIVISION OF HAZARDOUS MATERIALS
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from Autor	
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2.3 Waste Disposal Facilities

Toxikon employs the technical expertise of Triumvirate Environmental Services Inc, to manage our waste disposal needs. All documentation including waste description, manifests and labelling information, characteristics of waste, chemical composition, volumes, special handling information, and other required information is provided to Triumvirate on forms provided to Toxikon by Triumvirate Figure 2-1 is an example of a waste identification form. The waste streams generated by Toxikon Corporation are provided in Figure 2-2.

Full documentation associated with waste and sample disposal is provided to Toxikon by Laidlaw. Figure 2-3 is an example of a hazardous waste manifest document. In addition, Toxikon has an internal SOP which provides staff members with internal waste stream management. Figure 2-4 is an example of the hazardous waste log form used internally by Toxikon. Toxikon's EPA waste disposal # is MARO00005942.

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Figure 2-4

HACARDOUS WASTE LOG

TIN TY	:pe: pe:		•		
	****	Description	Volume/Weight	Concentration	Tachnic
					[
					
					<u>. </u>
				in this barrel!	

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3.0 Document Control

3.1 Laboratory Notebook Policy

Laboratory notebooks are maintained by analysts in the laboratory. Every page in the notebook must be signed and dated by the analyst when completed. Any unused space must be marked out by a Z line across the page so that no additional information can be added.

Every notebook page must be reviewed, signed and dated by the analysts' supervisor.

Laboratory notebooks are archived at Toxikon in firerated cabinets in a card-coded access room equipped with a halon fire protection system. Laboratory notebooks are maintained for a minimum of three years.

3.2 Samples Tracking / Custody Procedures

Sample custody includes all records and documentation that are required to trace a sample from the point of origin through disposal after analysis. Formal chain of custody starts when the precleaned containers with the custody seals are dispatched to the field from the lab. Information on a custody seal includes:

Person Collecting Sample:	Sample #
Date Collected:	Time Collected:

After bottles are shipped, the clients have legal chain of custody during sampling. The next step in the formal chain of custody at Toxikon is when the samples are shipped / delivered to Toxikon by the client, either by mail or courier.

The Sample Custodian or designee serves as the Sample Receipt Officer (SRO). The SRO is responsible for receiving all test samples and performs the following:

*Compares the test sample(s) received with appropriate packing slip, chain of custody, purchase order, authorization letter, Toxikon quotation or other available documents describing the analyses to be performed. The client is immediately notified of damaged, incomplete, or incorrect sample shipments.

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*Assigns a Toxikon identification number to each project. This number identifies the year, month, and a sequential value assigned to the particular project. For example, if the project is the 77th to be received in March 1997, it will be assigned number 9703077. The next would be assigned 9703078, and so on. Each sample within a project is also assigned a number as a sequential suffix (.1, .2, etc.) to the project number. For example, two samples received for testing under project number 9703077 would be referred to as samples 9703077.1 and 9703077.2. If multiple analyses are requested for one sample, each sequential suffix assigned to a sample has a letter (i.e. A., B, C) attached to it. These letters are for each For example, analysis requested on one sample. 9703077.1.A and 9703077.2.B indicate that sample 1 requires two tests to be run on it (i.e. RCRA metals and TPH).

*Completes a laboratory Chain-of-Custody Record for each project. The Chain-of-Custody Record form is a 3-page carbonless document. One copy is glen to the client upon receipt of samples by the laboratory, one is retained by the laboratory, and the last is sent with the final report to the client.

On sample receipt, the SRO verifies sample integrity (i.e. leaking, broken bottles, etc.), proper and complete sampe documentation and identification, and proper preservation of samples. Samples that need to be stored at 4 degrees C should be delivered in ice. Upon receipt of a sample cooler, the cooler temperature should be measured and recorded on the COC. A sample log-in sheet, form DC-1 (Fig. 3-1) should be completed.

The criteria to reject samples include leaking/broken bottles, incomplete COCs and improper preservation of samples. Clients are informed of the problem immediately either by phone or in writing.

The test sample is labelled, at a minimum, in the following manner:

Company	Name: Requested:			 	_
Sample:		Water:	Soil:	Other	_

Each project received is logged into a Master Project Log Book by the SRO. An example of this log is in figure 3-2. In

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addition to the Master Project log book, the data reduction personnel is responsible for maintaining sample documentation in a computerized log. This project log is generated through he Laboratory Information Management System (LIMS) at Toxikon, called SAM (i.e. Sample Analysis Management).

A Sample Control Record (Fig. 3-4) and Organic Extraction Form (Fig 3-5) is used to track samples and extracts respectively in the laboratory.

All information not present on the sample logs are covered in the chain-of-custody. All documentation / logs are signed / initialed by appropriate personnel. Errors in all documentation are deleted with a single line entry.

Samples are stored in a locked room with a full time sample custodian. Standards are stored separately from all samples and Volatile (VOC) samples segregated from all other samples. Laboratory personnel have to enter the log-in time and log-out time and date on log forms when they remove samples from the sample storage room. The full-time sample custodian is responsible for the security and accessibility of samples, and makes sure samples are properly logged-in and logged-out. Further, to ensure security, locked refrigerators are provided for the storage of samples.

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Figure 3-1

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		···-			
			****	ESPONDING .	
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3DG Number:					REMARKS:
IAS Number		HYSOES	EAMPLE	ASSIGNED	SCHOOLS
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REMARKS;	i				SHIPMENT ETC
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2 25am-ol-Custody	Present/Absent*		·· 	<u> </u>	
Records				 	
Contract Lab	3-yeart/Absent*				
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Figure 3-2

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		= = = = = = = = = = = = = = = = = = = =	DATE RCV ' D
			TOXIKOH PROJECT #
			CLIEFT HUMBER & DIVIUXON CULTERT PROJECT LOG BOOK CONTAINED CONTAI
			HARTER PROJECT LOG HOOK LITERT HUNDER & TYPE OF SAMPLE CONTAINER(S)
			AG. GAALECER
			DISPOSAL DATE

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				WPECIAL WPECIAL WASHING GOILMENTS				- view				CHRUSH BUSINESS DAY TURN AROUND	al information known ar buspackad isa bangdab odiar Man
WORK ONDER #:	ANALYBEB											DRUSH BUSIL	Sample disposal information An there are other known or emplocted continuous in these early at the third t
ODY RECORD	CONTAIRER TYPE	P. PIASINA U. UIASS V. VUA										DATE	DATE.
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Figure 3-4

Sample Control Record

Toxikon Number	Client-
Number of Samples	Sample Custodian————
Number of Containers	Sata/Time
Location	Disposal Data:

Laboratory Sample #	Removed Sy	Cata & Time Removed	Reason	Date & Time Returned	Location Returned
	-	**			
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Figure 3-5

TOXIKON PREP. SHEET

ALYSIS	METHOD		APPROVE				21.1	SOLV. Vol.		Conc.	Final Vol.	Final Cond
SAMPLE ID	THUOMA	1	*DRY W	l .	UD VODED	1	PH		ı		Vol. (ML)	(UO/ML)
	NaSO4	Wt.IVol	<u> </u>	VOL.	by/ on	IN I.	FINAL	ML	by \ on	by \ on	VOL (ML)	(nowr)
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								.	•			
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Sample Rollinguishe	ad to OEPT,					Dato	:		Analyst	:		

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3.3 Logbook Maintenance and Archiving Procedures

There is a preventive maintenance equipment log generated for each piece of equipment in the laboratory along with a standard operating procedure which describes its operation as well as calibration procedures.

These records are made out and kept current by the operator. The operator must make sure that all outside services tasks are entered onto this form as well.

Records of column usage for all chromatographic equipment are also kept in a similar manner to the preventive maintenance equipment log. The column log is kept separate for the raw data entries and contains the following:

Cover	page:	
	Column ID #:	
	Use:	
	Manufacturer:	
	Date of Receipt:	
	Location:	
	Responsible Department:	

The second page contains a statement that the record shall be maintained and contain the information as designated in the third and all subsequent pages.

The third and subsequent pages contain the following:

Date of Packing/ Purchase	Lot # of Pre- coated Column Packing and Liquid Phase I	Conditioning Temp Flow Rate/Time	Length/ Shape	Backgroun d Current	Tech
---------------------------------	---	--	------------------	------------------------	------

Instrumentation logs are maintained by the GC, GC/MS and the metals labs for designated instruments. The logs consist of raw data printouts which include all batch QC and sample analysis data (e.g. ICP printouts for the metals lab and Chromatograms for the GC lab.)

All raw data, chromatograms, column logs, equipment run logs, equipment calibrations and preventive maintenance and QA/QC records are archived at Toxikon in a card-coded access room equipped with a halon fire protection system. Within the room, records are stored in fire-rated file cabinets. These records are archived by year and department.

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Records can only be accessed by the quality assurance unit and / or lab manager who have accessibility tot he room. No other personnel can enter the archive. If materials are to be removed from the archives, they are logged out in the archive logbook and logged in when returned. Laboratory records are maintained for a minimum of three years.

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- 3.4 Case File Organization, Preparation and Review Procedures
 - 3.4.1 A file for Protocol work will contain the following information:
 - 3.4.1.1 Standard Operating Procedures
 - 3.4.1.2 Quality Assurance Plan
 - 3.4.1.3 Weekly Sample Receipt Summaries containing the following items:
 - * Lab name
 - * Contract number
 - * NYSDEC Case #
 - * NYSDEC SDG #
 - * Lab ID#
 - * Name of NYSDEC Sample Submitter
 - * Code numbers for requested analyses from Contract Laboratory Sample Information Sheet
 - * Sample Analysis Price full sample price from contract for each sample # reported
 - * List of NYSDEC sample numbers of all samples in the SDG, identifying the first and last samples received, and their dates of receipt
 - 3.4.1.4 Semi-annual verification of instrument parameters containing the following items:
 - * Semiannually verification of instrument detection limits and linear range
 - * For the ICP instrumentation and methods, annually interelement correction factors (including method of determination), wavelengths used, and integration times.
 - 3.4.2 A separate file will be maintained for each

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Sample Delivery Group SDG). This file will be maintained in the following order.

3.4.2.1 All original shipping documents, including:

NYSDEC Chain of Custody Record

Air bills

NYSDEC Contract Lab Sample Information Sheets
Sample Tags (if present) sealed in plastic bags

- 3.4.2.2 Copy of receiving forms or logbook
- 3.4.2.3 Sample data summary package containing:
 - * NYSDEC Data Package Summary Forms
 - * SDG Narrative
 - * By fraction (VOA, SV, PEST, INORG, CONV) and by sample within each fraction tabulated target compound results and tentatively identified compounds
 - * By fraction (VOA, SV and PEST) surrogate spike analysis results by matrix (water and / or soil) and for soil, by concentration (low or medium)
 - * By fraction (VOA, SV and PEST) matrix spike / matrix spike duplicate / matrix spike blank results - as required by method
 - * By fraction (VOA, SV and PEST) QC Check Sample / Standard Recovery Summary - if required by method
 - * By fraction (INORG and CONV only) duplicate sample results
 - * By fraction (VOA, SV, PEST, INORG, CONV) blank data and tabulated results including tentatively identified compounds
 - * By fraction (VOA and SV only) internal

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standard area data

3.4.2.4 Sample Data Package containing:

3.4.2.4.1 For Superfund

- 3.4.2.4.1.1 SDG Narrative
- 3.4.2.4.1.2 Contract Lab Sample Information Sheets
- 3.4.2.4.1.3 Chain-of-Custody Forms
- 3.4.2.4.1.4 Superfund CLP Volatiles
 Data

QC Summary

System Monitoring Compound Summary

Matrix Spike / Matrix Spike Duplicate / Matrix Spike Blank Summary

Method Blank Summary

GC/MS Instrument Performance Check

Internal Standard Area and RT Summary

Instrument Detection Limits

Sample Data

Target Compound Results

Tentative Identified Compounds

Reconstructed Total Ion Chromatograms (RIC) for each sample or sample extract

For each sample, by each compound identified

-Copies of raw spectra and copies of background-subtracted mass spectra

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of target compounds that are identified in the sample and corresponding background-subtracted TCL standard mass spectra.

-Copies of mass spectra of organic compounds (tentatively identified compounds), with associated bestmatch spectra (three best matches)

Standards Data

Initial Calibration Data

VOA standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for the initial (five point) calibration

All initial calibration data that pertain to samples int he data package

Continuing calibration

Raw QC Data

BFB (for each 12-hour period for each GC/MS system utilized

Blank Data in chronological order

Matrix Spike Blank Data

Matrix Spike Data

Matrix Spike Duplicate Data

Copy of Calculations

Copy of Extraction Logs

3.4.2.4.1.5 Superfund CLP Semiviolatiles
Data

QC Summary

Surrogate Percent Recovery Summary

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Matrix Spike/Matrix Spike Duplicate Summary

Method Blank Summary

GC/MS Instrument Performance Check

Internal Standard Area and RT Summary

Instrument Detection Limits

Sample Data

TCL Results

Tentatively Identified Compounds

Reconstructed total ion chromatograms

For each sample, by each compound identified:

Copies of raw spectra and copies of background-subtracted mass spectra of target compounds that are identified in the sample and corresponding background-subtracted Superfund TCL standard mass spectra.

Copies of mass spectra of nonsurrogate organic compounds (Tentatively Identified Compounds) with associated pest-match spectra (three best matches)

Standards Data

Initial Calibration Data

Continuing calibration

Semivolatile GPC Calibration Data

Raw QC Data

DFTPP (for each 12-hour period, for

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each GC/MS system utilized)

Blank Data - in chronological order by extraction date

Matrix Spike Blank Data

Matrix Spike Data

Matrix Spike Duplicate Data

Copy of Calculations

Copy of Extraction Logs

3.4.2.4.1.6 Superfund-CLP Pesticide/Aroclor Data

QC Summary

Surrogate Percent Recovery Summary

Matrix Spike/Matrix Spike Duplicate / Matrix Spike Blank Summary

Method Blank Summary

Instrument Detection Limits

Sample Data

Superfund TCL Results - Organic Analysis Data Sheet

Copies of pesticide chromatograms from second GC column confirmation

If pesticide/Aroclors are confirmed by GC/MS, copies of reconstructed ion chromatograms, raw spectra and copies of background-subtracted mass spectra of Superfund target compounds (Superfund-TCL) that are identified in the sample and corresponding background-subtracted Superfund-TCL standard mass spectra.

Standards Data

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Initial Calibration of Single Component Analyte

Initial Calibration of Multi-Component Analyte

Analyte Resolution Summary

Calibration Verification Summary

Analytical Sequence

Florisil Cartridge Check

Pesticide GPC Calibration

Pesticide Identification Summary for Single Component Analyte

Pesticide Identification Summary for Multi Component Analyte

Chromatograms and data system printouts are required for:

Resolution Check Mixture

Performance Evolution Mixtures, all

Individual Standard Mixture A, at three concentrations, each initial calibration

Individual Standard Mixture B, at three concentrations, each initial calibration

All multicomponent analyte (Toxaphene and Aroclors), each initial calibration

All mid point concentrations of Individual Standard Mixtures A and B used for calibration verification

Florisil cartridge check solution, all lots

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Pesticide GPC Calibration Check Solution, all calibrations relating to samples in the SDG

All multicomponent analyte standards analyzed for confirmation

A printout of retention times and corresponding peak areas or peak heights

Pesticide GPC Calibration Data

Raw QC Data

Blank Data

Matrix Spike Data

Matrix Spike Duplicate Data

Matrix Spike Blank Data

Copy of Calculations

Copy of Extraction Logs

3.4.2.4.1.7 Inorganic Data

Results - Inorganic Analysis Data Sheet

Quality Control Data

Initial and Continuing Calibration Verification

CRDL Standard for AA and Linear Range Analysis for ICP

Blanks

ICP Interference Check Sample

Spike Sample Recovery

Post Digest Spike Sample Recovery

Duplicates

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Laboratory Control Sample

Standard Addition Results

ICP Serial Dilutions

Holding Times

Verification of Instrument Parameters

Instrument Detection Limits (Quarterly)

ICP Interelement Correction Factors (Annually)

ICP Interelement Correction Factors
(Annually)

ICP Linear Ranges (Quarterly)

Raw Data

Calibration standards, including source and prep date

Initial and continuing calibration blanks and preparation blanks

Initial and continuing calibration and verification blanks

Interference check samples, ICP serial dilution samples, CRDL Standard for ICP and AA, Laboratory Control Sample and Post Digestion Spike

Diluted and undiluted samples and all weights, dilutions and volumes used to obtain the reported values

Duplicates

Spikes

Instrument used, any instrument adjustments, data corrections or

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other apparent anomalies on the measurement record

All information for furnace analysis clearly and sequentially identified on the raw data

Time and date of each analysis

Integration times for AA analyses

Copy of calculations

Digestion Logs

3.4.2.4.2 For ASP CATEGORY A

3.4.2.4.2.1 SDG Narrative

3.4.2.4.2.2 Contract Lab Sample Information Sheets

3.4.2.4.2.3 NYSDEC Data Package Summary Forms

3.4.2.4.2.4 Chain-of-Custody Forms

3.4.2.4.2.5 GC/MS Volatiles Data

Sample Data

TCL Results - Organic Analysis Data Sheet

Tentatively Identified Compounds

3.4.2.4.2.6 GC/MS Semivolatiles Data

Sample Data

TCL Results - Organic Analysis Data Sheet

Tentatively Identified Compounds

3.4.2.4.2.7 Pesticide/PCB Data

Sample Data

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Superfund TCL Results - Organic Analysis Data Sheet

3.4.2.4.2.8 GC Organic Data

Sample Data

TCL Results - Organic Analysis Data Sheet

3.4.2.4.2.9 Inorganic Data

Results - Inorganic Analysis Data Sheet

3.4.2.4.2.10 Toxicity Characteristic Leaching Procedure (TCLP) Data

Results - Toxicity Characteristic Leaching Procedure Analysis Data Sheet

3.4.2.4.3 For ASP CATEGORY B

- 3.4.2.4.3.1 SDG Narrative
- 3.4.2.4.3.2 Contract Lab Sample Information Sheets
- 3.4.2.4.3.3 NYSDEC Data Package Summary Forms
- 3.4.2.4.3.4 Chain-of-Custody Forms
- 3.4.2.4.3.5 GC/MS Volatiles Data

QC Summary

System Monitoring Compound Summary

Matrix Spike / Matrix Spike Duplicate Summary

QC Check Sample / Standard

Method Blank Summary

GC/MS Instrument Performance Check

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Instrument Detection Limits

Sample Data

TCL Results - Organic Analysis Data Sheet

Tentatively Identified Compounds

Reconstructed Total Ion Chromatogram s(RIC) for each sample or sample extract

For each sample, by each compound identified

Copies of raw spectra and copies of spectra of target compounds identified int he sample and corresponding background-subtracted TCL standard mass spectra

Copies of mass spectra of organic compounds (Tentatively Identified Compounds) with associated bestmatch spectra (three best matches)

Standards Data

Initial Calibration Data

Continuing Calibration

Raw QC Data

BFB (for each 12-hour period, for each GC/MS system utilized)

Blank Data - in chronological order

Matrix Spike Blank Data

Matrix Spike Data

Matrix Spike Duplicate Data

QC Check Sample/ Standard

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Copy of Calculations

Copy of Extraction Logs

3.4.2.4.3.6 GC/MS Semivolatiles Data

QC Summary

Surrogate Percent Recovery Summary

Matrix Spike/Matrix Spike Duplicate Summary

QC Check Sample/Standard

Method Blank Summary

GC/MS Instrument Performance and Mass Calibration

Internal Standard Area and RT Summary

Instrument Detection Limits

Standards Data

Initial Calibration

Continuing Calibration

Raw QC Data

BFD (for each 12-hour period, for each GC/MS system utilized)

Blank Data - in chronological order

Matrix Spike Blank Data

Matrix Spike Data

Matrix Spike Duplicate Data

QC Check Sample/ Standard

Copy of Calculations

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Copy of Extraction Logs

3.4.2.4.3.7 GC/MS Semivolatiles Data

QC Summary

Surrogate Percent Recovery Summary

Matrix Spike/Matrix Spike Duplicate Summary

QC Check Sample/Standard

Method Blank Summary

GC/MS Instrument Performance and Mass Calibration

Internal Standard Area and RT Summary

Instrument Detection Limits

Sample Data

TCL Results - Organic Analysis Data Sheet

Tentatively Identified Compounds

Reconstructed Total Ion Chromatograms(RIC) for each sample or sample extract

For each sample, by each compound identified

Copies of raw spectra and copies of spectra of target compounds identified int he sample and corresponding background-subtracted TCL standard mass spectra

Copies of mass spectra of organic compounds (Tentatively Identified Compounds) with associated bestmatch spectra (three best matches)

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GPC chromatograms (if GPC performed)

Standards Data

Initial Calibration

Continuing Calibration

Semivolatile GPC Calibration Data

Raw QC Data

DFTPP (for each 12-hour period, for each GC/MS system utilized)

Blank Data - in chronological order

Matrix Spike Blank Data

Matrix Spike Data

Matrix Spike Duplicate Data

QC Check Sample/ Standard

Copy of Calculations

Copy of Extraction Logs

3.4.2.4.3.8 GC/EC Pesticide/Arochlor Data

QC Summary

Surrogate Percent Recovery Summary

Matrix Spike/Matrix Spike Duplicate Summary

QC Check Sample/Standard

Method Blank Summary

Instrument Detection Limits

Sample Data

TCL Results - Organic Analysis Data Sheet

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Copies of pesticide chromatograms

Copies of pesticide chromatograms from second GC column confirmation

Manual work sheets

UV traces from GPC (if GPC performed)

If pesticide/Arocolors are confirmed by GC/MS, copies of reconstructed ion chromatograms, raw spectra and copies of background-subtracted mass spectra of Superfund target compounds that are identified in the sample and corresponding backgroundsubtracted Superfund-TCL standard mass spectra

Standards Data

Initial Calibration of Single Component Analyte

Initial Calibration of Multi Component Analyte

Analyte Resolution Summary

Calibration Verification Summary

Calibration Verification Summary

Analytical Sequence

Florisil Cartridge Check

Pesticide GPC Calibration

Pesticide Identification Summary for Single Component Analyte

Pesticide Identification Summary for Multi Component Analyte

Chromatograms and data system printouts for all standards

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including the following:

Resolution Check Mixture

Performance Evaluation Mixtures, all

Individual Standard Mixture A, at three concentrations, each initial calibration

Individual Standard Mixture B, at three concentrations, each initial calibration

All multicomponent analyte (Toxaphene and Aroclors), each initial calibration

All mid point concentrations of Individual Standard Mixtures A and B used for calibration verification

Florisil cartridge check solution, all lots

Pesticide GPC Calibration Check Solution, all calibrations relating to samples in the SDG

All multicomponent analyte standards analyzed for confirmation

A printout of retention times and corresponding peak areas of peak heights

Pesticide GPC Calibration Data

Raw QC Data

Blank Data - in chronological order

Matrix Spike Data

Matrix Spike Duplicate Data

QC Check Sample/ Standard

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Copy of Calculations

Copy of Extraction Logs

3.4.2.4.3.9 GC Organic Data

QC Summary

Surrogate Percent Recovery Summary

Matrix Spike/Matrix Spike Duplicate/Matrix Spike Blank Summary

OC Check Sample/Standard

Method Blank Summary

Instrument Detection Limits

Sample Data

TCL Results - Organic Analysis Data Sheet

Copies of chromatograms

Copies of chromatograms from second GC column confirmation

GC integration report or data system printout and calibration plots (area vs. concentration) (where appropriate)

Manual work sheets

GPC chromatograms (if GPC performed)

Standards Data

Initial Calibration of Single Component Analyte

Continuing Calibration Data

QC Check Sample/Standard (as required per method)

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Standard chromatograms and data system printouts

Raw QC Data

Blank Data

Matrix Spike Data

Matrix Spike Duplicate Data

QC Check Sample/ Standard

Copy of Calculations

Copy of Extraction Logs

2.4.2.4.3.10 Inorganic Data

Results - Inorganic Analysis Data Sheet

Quality Control Data

Initial and Continuing Calibration Verification

CRDL Standard for AA and Linear Range Analysis for ICP

Blanks

ICP Interference Check Sample

Spike Sample Recovery

Post Digest Spike Sample Recovery

Duplicates

Quality Control Sample

Standard Addition Results

ICP Serial Dilutions

Holding Times

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Verification of Instrument Parameters

Instrument Detection Limits (Semiannually)

ICP Interelement Correction Factors (Annually) Part 1

ICP Interelment Correction Factors (Annually) Part 2

ICP Linear Ranges (Quarterly)

Raw Data

Calibration standards, including source and prep date

Initial and continuing calibration blanks and preparation blanks

Initial and continuing calibration and verification blanks

Interference check samples, ICP serial dilution samples, CRDL Standard for ICP and AA, Laboratory Control Sample and Post Digestion Spike

Diluted and undiluted samples and all weights, dilutions and volumes used to obtain the reported values

Duplicates

Spikes

Instrument used, any instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation

All information for furnace analysis clearly and sequentially identified

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on the raw data, including NYSDEC sample number, sample and analytical spike data, percent recovery, coefficient of variation, full MSA data, MSA correlation coefficient, full MSA data, MSA correlation coefficient, slope and intercepts of linear fit, final concentration (standard additional concentration), and type background correction used: BS for Smith-Heiftje, BD for deuterium Arc, or BZ for Zeeman

Time and date of each analysis

Integration times for AA analyses

Digestion Logs

3.4.2.4.3.11 Wet Chemical Data

Results - Wet Chemical Analysis Data Sheet

Quality Control Data

Initial and Continuing Calibration Verification

CRDL Standard for Wet Chemical Analysis

Blanks

Spike Sample Recovery

Post Digest Spike Sample Recovery

Duplicates

Laboratory Control Sample

Holding Times

Raw Data

Calibration standards, including

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source and prep date

Initial and continuing calibration blanks and preparation

Initial and continuing calibration and verification blanks

Diluted and undiluted samples &by NYSDEC sample number) and all weights, dilutions and volumes used to obtain the reported values.

Duplicates

Spikes

Instrument used, any instrument adjustments, data corrections or other apparent anomalies on the measurement record

Time and date of each analysis

Digestion and Distillation Logs

3.4.2.4.3.12 Results - Toxicity Characteristic Leaching Procedure Analysis (TCLP) Analysis Data Sheet

TCLP Inorganic Quality Control Data

Initial and Continuing Calibration Verification

CRDL Standard for AA and Linear Range Analysis for ICP

Blanks

ICP Interference Check Sample

Spike Sample Recovery

Post Digest Spike Sample Recovery

Duplicates

a Ay

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Quality Control Sample

Standard Addition Results

ICP Serial Dilutions

Holding Times

Verification of Instrument Parameters

Instrument Detection Limits (Semiannually)

ICP Interelment Correction Factors
(Annually)

ICP Interelment Correction Factors
(Annually)

ICP Linear Ranges (Quarterly)

Raw Data

Calibration standards, including source and prep date

Initial and continuing calibration blanks and preparation blanks

Initial and continuing calibration and verification standards

Interference check samples, ICP serial dilution samples, CRDL Standard for ICP and AA, Laboratory Control Sample and Post Digestion Spike

Diluted and undiluted samples (by DEC sample number) and all weights, dilutions and volumes used to obtain the reported values.

Duplicates

Spikes

Instrument used, any instrument

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adjustments, data corrections or other apparent anomalies on the measurement record

All information for furnace analysis clearly and sequentially identified ont he raw data, including NYSDEC sample number, sample and analytical spike data, percent recovery, coefficient of variation, full MSA data, MSA correlation coefficient, full MSA data, MSA correlation coefficient, slope and intercepts of fit, final sample concentration (standard concentration), and addition type background correction used: BS for Smith-Heiftje, BD for deuterium Arc, or BZ for Zeeman

Time and date of each analysis

Integration times for AA analyses

Digestion and Distillation Logs

3.4.3 All original laboratory records, not already submitted in the Sample Data Package, of sample transfer, preparation and analysis, including, but not limited to, the following documents:

Original preparation and analysis forms or copies of preparation and analysis logbook pages

Internal sample and sample extract transfer chainof-custody records

All instrument output, including strip charts from screening activities

3.4.4 All other original SDG-specific documents in the possession of the Laboratory, including, but not limited to, the following documents:

Telephone contract logs

Copies of personal logbook pages

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All hand written case-specific notes

Any other case-specific documents not covered by the above.

The Quality Assurance Officer will review the file for completeness using the Organics Complete SDG (CSF) Inventory Sheets (Fig 3.6) for Organics (DC-2-ORG) and Inorganics (DC-2-IN). Fig. 3-7.

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Figure 3-6

CRGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY N		- <u></u>	CITY/STATE	<u></u> -
CASE NO	SOG NO	SDG NCS, TO FOLI	LOW	SAS NO
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	Form CC-2) .Do not :	number)		
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<u>Zorathes Data</u>				
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		mary (Form H-CLP-VCA)		
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	лх Зылгталу "Ролт (У-		<u> </u>	- — —
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Des	st library matches			_
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nitial Calibrat	on Data Form VI-CLF	P-VCA)		
RtOs and Qua	int Reports for all Stan	carcs		
Continuing Ca	moration (Form VII-QU	F-VOA:		
RIOs and Qua	int Reports for all Stan	dards		
				
Raw QC Data				
3F3		•		
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Matrix Spike 3	lank Data			
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Matrix Spike D	uplicate Data			
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CC Summary				
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	nary (Form III-CLP-SV)			
	iummary (Form IV-CLE			
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(Farm VIII)	3-CLP-SV and Form VI	IIIC-CLP-SV)		

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Figure 3-6a

ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont.)

Semivolatiles Data (cont.) 3. Sample Data TCL Results Form I-CLP-SV) Tentatively identified Compounds (Form I-CLP-SV-TIC) Reconstructed total ion chromatograms (RIC) for each sample For each sample: Raw spectra and packground-subtracted mass spectra of target compounds identified Quantitation reports Mass spectra of all reported TICs with three pest identified GPC phromatograms of GPC performed) Standards Data All instruments mitial Calibration Data Form JI-CLP-SVI	PAG FRCM	SE NGS. TO		CHECK NYSDEC
Semivolatiles Data (cont.) 3. Sample Data TCL Results Form I-CLP-SV) Tentatively identified Compounds (Form I-CLP-SV-TIC) Reconstructed total ion chromatograms (RIC) for each sample For each sample: Raw spectra and packground-subtracted mass spectra of target compounds identified Quantitation reports Mass spectra of all reported TICs with three pest identified GPC phromatograms of GPC performed) Standards Data All instruments mitial Calibration Data Form JI-CLP-SVI	FRCM	TO	LAB	NYSDEC
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nitial Calibration Çara Form 71-ÖLP-SVN				
RICs and Quant Reports for all Standards				
Continuing Calibration Form VB-CLP-SV)				
RIGs and Quant Reports for all Standards	_			
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Matrix Spike Blank Data				
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Matrix Spike Oublicate Data				
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CO Summary	<u> </u>			
Surrogate Percent Recovery Summary				
(Form HOLP-PEST)				
MS/MSD Summary (Form (II-OLP-PEST)				
Method Blank Summary (Form IV-CLP-PEST)				
Sample Data				
Sample Data TOL Results: Form (-OLP-PEST)				
Chromatograms (Primary Column)				
Chromatograms from second GC column confirmation				
				_
GC Integration report or data system printout and calibration plots				
Manual work sneets				
UV traces from GPC (if available)				
For pesticides: Aradiars confirmed by GC/MS, copies				
of raw spectra and copies of background-suptracted mass spectra of target compounds (samples & standards)				 ,

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Figure 3-6b

ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont.)

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			Form VII-CL2-PEST-2)		
		ensa Form /M-CL			
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Figure 3-6c

CRGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont.)

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SDG ೦ು	ver Sheet			
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Figure 3-7

INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

	LABORATORY NAME			CITY/STATE				•
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1	Inventory Sheet (Form DC-2) (Do not	лиmber)					
. 2.	Cover Page							
_. 3.	Inorganic Analysi	s Data Sheet						
	(FORM ! - IN)							
4.	Initial & Continuin	g Calibration					 -	
	Verification (FORI	M IIA - IN)						
5.	CRDL Standards	For AA and ICP						
	(FORM (IB - IN)							
5.								
7.	ICP Interference C	Check Sample.				-		-
	(FORM IV - IN)		4.					
3.	Spike Sample Rec	overy ≱FORM VA - I	N)					
9.	Post Digest Spike	Sample Recovery					. ———	
	(FORM VB - IN)							
10.	Dublicates :FORM	VI - IN1						
11.	Lappratory Control	Sample (FORM VII	- IN)					
	Standard Addition i		- JN)					
13.	ICP Sena. Dilutions	(FORM IX - IN)						
	Instrument Detection	•	IN)					
15.	ICP Interelement C	orrection Factors						
	(FORM XIA - IN)							
16.	ICP interference Co	prrection Factors				•		
	(FORM XIB - IN)							
	ICP Linear Ranges	•		•				
	Preparation Log (FC	•		_				
	Analysis Run Log (F	FORM XIV - IN)		-				
	ICP Raw Data			_				
	Furnace AA Raw Da	ata		_				
22.	Mercury Raw Data			_				

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Figure 3-7a

INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont.)

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		Information Sheet (C.	SIS)		
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3.6 Process for Revision of Technical or Documentation Procedures

There are several types of quality assurance documents.

*The Quality Assurance Manual provides the overall policy for the laboratory.

*Standard Operating Procedures (SOP's) are detailed instructions outlining a specific routine task performed in the laboratory.

*Project Specific Manuals may be prepared where a project requires unique or different quality assurance requirements or when they ar required by regulatory agencies. These documents are frequently called Quality Assurance Project Plans (QAPP's).

All of the Quality Assurance Documents listed above are approved and controlled documents as spelled out in this paragraph. The Quality Assurance Manual requires the approval of the QA Officer an the Scientific Director before changes are issued. SOP's approved by the Quality Assurance Office and the Scientific Director require the approval of the Laboratory Manager. These documents must be signed and dated by these responsible individuals before issuance.

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appropriate locations around the laboratory (i.e. equipment logs and laboratory SOP notebooks). All finalized SOPs are On-line in the SOP directory.

Only certified copies or On-line View Only Procedures of the original document shall be used in the laboratories.

When revised certified copies of SOPs are issued, all old copies are collected by the Document Control and destroyed.

All SOP's which are determined to be no longer appropriate will be withdrawn.

After obsolescence is determined, the original SOP and all contents from the historical file (when applicable) are stamped with the word "WITHDRAWN," initialed, and dated by Regulatory Affairs. The entire contents are then transferred to the Withdrawn File.

All copies of the SOP, as documented by the SOP distribution list, are collected and destroyed.

The On-Line version is deleted from the system.

The assigned number of the Withdrawn SOP is not be reused.

All WITHDRAWN documents are listed on a Withdrawn Index. This Index can be accessed On-line in Wordperfect 5.1 under Quality System directory.

All SOP files (Master, Historical, and Withdrawn files) are maintained in numerical order.

SOP Master File: The SOP Master File located in Regulatory Affairs, is a permanent, chronological history of Standard Operating Procedures (SOPs), SOP reviews, and distribution lists.

SOP Historical File: A separate file is kept of all historical SOPs. This includes all prior original and/or subsequent SOP revisions and the dates of the revisions.

SOP WITHDRAWN File: A third file is kept for all withdrawn SOPs and their appropriate documentation.

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4. Analytical Methodology

4.1 Calibration Procedures and Frequency

This section describes the calibration procedures and the frequency of calibration for the quantitative analysis of organic, metallic, and other inorganic analyte. All reference materials used for instrument calibration, internal standards, and surrogate standards are of the highest purity attainable (>98%) through certified sources.

4.1.1 Gas Chromatography/Mass Spectroscopy

The identity of analytes used in instrument tuning, as internal standards, as surrogate compounds, as System Performance Check Compounds (SPCC), and as Calibration Check Compounds (CCC) will vary with the method. Nevertheless, the principle applies to all GC/MS methods. Tuning BFB/DFTPP is performed on a daily basis. Examples of tuning criteria with acceptance/ rejection ranges are listed in Tables 4-1 and 4-2.

Five standard solutions are analyzed and quantified using the internal standard method. The area response for the characteristic ions is tabulated against concentration for each analyte and internal standard, and the Relative Response Factors (RRFs) calculated as follows:

 $A_x = Area of the characteristic ion for the analyte of interest$

 A_{is} = Area of the characteristic ion for the specific internal standard

 C_{is} = Concentration of the internal standard

 C_x = Concentration of the analyte of interest

The average RRF is calculated for each standard analyte. Before the calibration curve may be used, a system

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Mixture must be greater than or equal to 60.0% of the height of the shorter peak.

<u>Performance Evaluation Mixture</u>

All peaks in both of the Performance Evaluation Mixtures must be 100 percent resolved on both columns.

The absolute retention times of each of the single component pesticides and surrogates in both the PEMs must be within the retention time windows determined from the initial calibration.

The relative percent difference of the calculated amount and the true amount for each of the single component pesticides and surrogates in both of the PEMs must be less than or equivalent to 25.0 percent.

The breakdown of DDT and Endrin in both of the Performance Evaluation Mixtures must be less than 20.0 percent, and the combined breakdown of DDT and Endrin must be less than 30.0 percent.

Continuing Calibration

Three types of analyses are used to verify the calibration and evaluate instrument performance. The analyses of instrument blanks, Performance Evaluation Mixtures (PEM), and the mid point concentration of individual Standard Mixtures A and B constitute the continuing calibration.

All single component pesticides and surrogates in the Performance Evaluation Mixtures used to demonstrate continuing calibration must be 100 percent resolved. The resolution between any two adjacent peaks in the midpoint concentrations of Individual Standard Mixtures A and B in the initial calibration must be greater than or equal to 90.0 percent.

The absolute retention time for each of the single component pesticides and surrogates in the PEMs and mid point concentration of the Individual Standard Mixtures used to demonstrate continuing calibration must be within the retention time window determined for the initial calibration.

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The relative percent difference of the calculated amount and the true amount for each of the single component pesticides and surrogates in the PEM and mid point concentration of the Individual Standard Mixtures used to demonstrate continuing calibration must be less than or equal to 10.0 percent.

The percent breakdown of DDT and Endrin in the PEM must be less than or equal to 20.0 percent on both columns. The combined breakdown of DDT and Endrin must be less than or equal to 30.0 percent on both columns.

4.1.3 Metals

All ICP metals are calibrated with a 3-point calibration when a new standard is prepared. All successive calibrations are done by performing an update on this 3-point calibration with the high and the low standard for each run. All GFAA and CVAA metals are calibrated using a 5-point calibration which is verified using a regression analysis. The calibration is verified at the beginning of each working day (ICV) and every 10 analyses thereafter (CCV) using standards from a different source than that of the calibration being verified. The recovery of the ICV and CCV must be 80%-120% for mercury and 90%-110% for all other metals.

When a certified solution of an analyte is not available from EPA or similar source, analyses are conducted on an independent standard at a concentration other than that used for calibration but within the calibration range. When measurements for the certified elements exceed control limits, the analysis is terminated, the problem corrected, the instrument re-calibrated and the calibration re-verified.

If a continuing calibration verification fails to pass acceptance criteria for any parameter, those samples analyzed after the last acceptable calibration verification will be repeated.

4.1.4 Balances

Each balance is checked daily for accuracy and precision using Class S weights. Any deviation greater than that specified for the instrument is considered unacceptable and the balance is serviced.

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Tables 4-3 through 4-5 detail the instrument calibration parameters that should be recorded for all sample runs relative to inorganics, GC/MS, GC and metals analyses. These logs are maintained by the analysts and reviewed daily by department supervisors.

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Table 4-1

GC/MS Standard P-Bromofluorobenzene (BFB) Tune Criteria for Volatiles, 20 ng Level or Less

m/e	Ion Abundance Criteria
50	15-40% of the base peak
75	30-60% of the base peak
95	Base peak, 100% rel. abundance
96	5-9% of the base peak
173	Less than 1% of the base peak
174	Greater than 50% of the base peak
175	5-9% of mass 174
176	95-101% of mass 174
177	5-9% of mass 176

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Table 4-2

GC/MS Standard Decafluorotriphenylphosphine (DFTPP) Tune

Criteria for Semivolatiles, 20 ng Level or Less

m/e	Ion Abundance Criteria
51	30-60% of mass 198
68	Less than 2.0% of mass 69
69	Mass 69 relative abundance
70	Less than 2.0% of mass 69
127	40-60% of mass 198
197	Less than 1.0% of mass 198
198	Base peak, 100% rel. abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	Greater than 1.0% of mass 198
441	Present, but less than mass 443
442	40-100% of mass 198
443	15-23 of mass 442

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		Inst	Table 4-3 Instrument Calibration (Metals)	4-3 ation (Met	als)		
Instrument	Standard Source	# Standards Init. Calib.	Acceptance/ Rejection Criteria Init. Cali.	Frequency	# Standards Cont. Calib	Acceptance/ Rejection Criteria Cont. Cali	Frequency
ICP (6010A)	Commer- cial Supplier (Leeman labs)	E)	ICV within +/-5% True Value	Daily or on failure of ICV/CCV	г .	+/- 10% True Value	Initial & every 10 Samples
AAS (CVAA hg)	Spex Fisher	S	ICV within +/- 20% True Value	Daily or on failure of ICV/CCV	1	+/-20% True Value	Initial & every 10 Samples
GFAA (7000A	Fisher EPA	ហ	ICV within +/- 10% True Value	Daily or on failure of ICV/CCV	T	+/- 20% True value	Initial & every 10 Sample

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	į	Ins	Tablestrument Calib	Table 4-4 Instrument Calibration - Organics	,		
				6.			
		# Standards	Acceptance/ Rejection		***	Acceptance/ Rejection	
Instrument	Standard Source	Init. Calib.	Init. 'Cali.*	Frequency	Standards Init. Calib.	Criteria Cont. Cali.*	Frequency
GC/MS	Commercial	5-6	% RSD and	Failure of		o t	
82408 82608	Supplier		minimum RF	continuous	1	Difference	Carty.
1			individual	calibration		Minimum RF	
			method			as per individual	
						method	
8270B	Commercial	ហ	% RSD and	Failure of	П	8F2	Dailv
	zer redens		minimum RF	continuous		Difference	7
			individual	calloration		minimum RF	
			method			individual	
						method	
* = Crite	* = Criteria apply to all Standard Sources	all Standard S	ources				

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		Instru	Table 4-5 Instrument Calibration - Organics (GC)	. 4-5 ion - Organics	(00)		
Instrument	Standard Source	# Standards Init. Calib.	Acceptance/ Rejection Criteria Init. Cali.*,	Frequency	# Standards Init. Calib.	Acceptance/ Rejection Criteria Cont.	Frequency
GC VOA Extractable Organics	Commercial Supplier	ហ	% RSD per individual method	Failure of continuous calibration	ri .	% D per individual method	Daily

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4.2 Sample Preparation / Extraction Procedures

Detailed instructions for each sample preparation / extraction procedure are listed in the individual standard operating procedures. However, brief summaries of the methods used are detailed in this section.

METALS

Method 3005A

Method 3005A is an acid digestion procedure used to prepare surface and ground water samples for analysis by inductively coupled argon plasma spectroscopy (ICP).

Total recoverable metals - The entire sample is acidified at the time of collection with nitric acid. At the time of analysis the sample is heated with acid and substantially reduced in volume. The digestate is filtered and diluted to volume, and is then ready for analysis.

Dissolved metals - The sample is filtered through a 0.45-um filter at the time of collection and the liquid phase is then acidified at the time of collection with nitric acid. Samples for dissolved metals do not need to be digested as long as the acid concentrations have been adjusted to the same concentration as in the standard.

Method 3010A

This digestion procedure is used for the preparation of agueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis, or by inductively coupled argon plasma spectroscopy (ICP). The procedure is used to determine total metals.

A mixture of nitric acid and the material to be analyzed is refluxed in a covered Griffin beaker. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is refluxed with hydrochloric acid and brought up to volume. If sample should go to dryness, it must be discarded and the sample reprepared.

Method 3015

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This digestion procedure is used for the preparation of aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for analysis, by graphite furnace absorption spectroscopy (GFAA) or inductively coupled argon plasma spectroscopy (ICP). The procedure is a hot acid leach for determining available metals using microwave heating.

A representative 45 mL aqueous sample is digested in 4 mL of concentrated nitric acid in a fluorocarbon (PFA or TFM) digestion vessel for 20 minutes using microwave heating. After the digestion process, the sample is cooled, and then filtered, centrifuged, or allowed to settle in a clean sample bottle prior to analysis.

Method 3020A

This digestion procedure is used for the preparation of aqueous samples, mobility procedure extracts, and wastes that contain suspended solids for analysis by furnace atomic absorption spectroscopy (GFAA) for the metals listed below. The procedure is used to determine the total amount of the metal in the sample:

A mixture of nitric acid and the material to be analyzed is refluxed in a covered Griffin beaker. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has been stabilized. After the digestate has been brought to a low volume, it is cooled and brought up in dilute nitric acid such that the final dilution contains 3% (v/v) nitric acid. This percentage will vary depending on the amount of acid used to complete the digestion. If the sample contains suspended solids, it must be centrifuged, filtered, or allowed to settle.

Method 3050A

This method is an acid digestion procedure used to prepare sediments, sludges and soil samples for analysis by furnace atomic absorption spectroscopy (GFAA) or by inductively coupled argon plasma spectroscopy (ICP).

A representative 1- to 2-g (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid or hydrochloric acid. Hydrochloric acid is used for ICP analyses and nitric acid is used for furnace AA work. Dilute hydrochloric acid is used as the final reflux acid for (1) the ICP analysis of As and Se,

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and (2) ICP analysis of Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn Mo, Na, Ni, Os, Pb, Tl, V, and Zn. Dilute nitric acid is employed as the final dilution acid for the furnace AA analysis of As, Be, Cd, Cr, Co, Fe, Pb, Mo, Se, Tl, and V.

Method 3051

This method is applicable to the microwave assisted acid digestion of sludges, sediments, soils, and oils.

This method is provided as an alternative to Method 3050. It is intended to provide a rapid multi-element acid leach digestion prior to analysis so that decisions can be made about site cleanup levels, the need for TCLP testing of a waste, and whether a BDAT process is providing acceptable performance. Digests produced by the method are suitable for analysis by graphite furnace atomic absorption (GRAA) or inductively coupled plasma emission spectroscopy (ICP-ES).

A representative sample of up to 0.5 g is digested in 10 mL of concentrated nitric acid for 10 min using microwave heating with a suitable laboratory microwave unit. The sample and acid are placed in a fluorocarbon (PFA or TFM) microwave vessel. The vessel is capped and heated in the microwave unit. After cooling, the vessel contents are filtered, centrifuged, or allowed to settle and then diluted to volume and analyzed by the appropriate SW-846 method.

ORGANICS

Method 3510B_

A measured volume of sample, usually 1 liter, at a specified pH is serially extracted with methylene chloride using a separatory funnel. The extract is dried, concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the cleanup or determinative method to be used. This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

Method 3540B

Method 3540 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils,

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sludges, and wastes. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

The solid sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble or between two plugs of glass wool, and extracted using an appropriate solvent in a Soxhlet extractor. The extract is then dried, concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.

Method 3550A

Method 3550 is a procedure for extracting nonvolatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

The method is divided into two sections, based on the expected concentration or organics in the sample. The low concentration method (individual organic compounds of less than 20 mg/kg) uses a larger sample size and a more rigorous extraction procedure (lower concentrations are more difficult to extract). The medium/high concentration method (individual organic components of more than 20 mg/kg) is much simpler and therefore faster.

Low concentration method - A 30 g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted three times using ultrasonic extraction. The extract is separated from the sample by vacuum filtration or centrifugation. The extract is ready for cleanup and/or analysis following concentration.

Medium / high concentration method - A 2 g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted once using ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis.

Method 5030

Method 5030 can be used for most volatile organic compounds that have boiling points below 200 degrees Celsius and are insoluble or slightly soluble in water. Volatile water-

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soluble compounds can be included in this analytical technique: however, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency. The method is also limited to compounds that elute as sharp peaks from a GC column packed with graphitized carbon lightly coated with a carbowax or a coated capillary column. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

The purge-and-trap process: An inert gas is bubbled through the solution at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are absorbed. After purging is completed, the sorbent column is heated and back flushed with inert gas to desorb the components onto a gas chromatographic column.

If the sample introduction technique above is not applicable, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water in a specially designed purging chamber. It is then analyzed by purge and trap GC following the normal water method.

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Table 4-6 Sample Preparation Table (Metals)

Sample Prep. Method number		Matrix	Sample prep. for these methods
3010	Acid Digestion	Aqueous sample Extracts.	es/ ICP
3005	Acid Digestion	Water (Dissolv	red) ICP
3020	Acid Digestion	Aqueous sample Extracts.	es/ GFAA
3050	Acid/peroxide Digestion	Soil/Sludge	GFAA
200/4.1.1-4	Acid Digestion	Aqueous sample (Drinking Wate	es GFAA er)
7470/7471	Acid/Permanga & Soil Digest	nate Water ion	7470/7471-Hg
1311	TCLP	All	200.7, 6010 (ICP)

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Table 4-7
Sample Preparation Table (Organics)

Sample Prep. Method number	Description :		ole prep. for se methods
1311:	Toxicity Characteristic Leaching Procedure	Liquid, Solid, Multiphasic Waste	TCLP
3510	Liquid/Liquid Extraction	Water, Wastewater.	
3550	Sonication Extraction	Soil, Sludge	8270, 8010 8080, 8020.
3540	Soxhlet Extraction	Soil, Sludge	8270, 8010 8080, 8020.
5030	Purge & Trap Method	Water, Soil, Sludge	8240.

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4.3 Sample Analysis Procedures

Detailed instructions for each analytical method are listed in the individual standard operating procedures. However, brief summaries of the methods used are detailed in this section.

VOLATILES

Water Samples

An inert gas is bubbled through a 5 mL sample contained in a specifically designed purging chamber at ambient temperature. The purgeable compounds are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeable compounds onto a gas chromatographic column. The gas chromatograph is temperature-programmed to separate the purgeable compounds which are then detected with a mass spectrometer.

Low Soil Samples

An inert gas is bubbled through a mixture of reagent water and 5 g of sample contained in a specifically designed purging chamber that is held at an elevated temperature. The purgeable compounds are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbet column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeable compounds onto a gas chromatographic column. The gas chromatograph is temperature-programmed to separate the purgeable compounds which are then detected with a mass spectrometer.

Medium Soil Samples

A measured amount of soil is extracted with methanol. A portion of the methanol extract is diluted to 5mL with reagent water. An inert gas is bubbled through this solution in a specifically designed purging chamber at ambient temperature. The purgeable compounds are effectively transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and

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backflushed with the inert gas to desorb the purgeable compounds onto a gas chromatographic column. The gas chromatograph is temperature-programmed to separate the purgeable compounds which are then detected with a mass spectrometer.

Water Samples

Arone liter aliquot of sample is acidified to pH 2.0 and extracted with methylene chloride using a continuous liquid-liquid extractor. Separatory funnel extraction is NOT permitted. The methylene chloride extract is dried with sodium sulfate, concentrated, subjected to GPC (GPC is required when higher molecular weight compounds are present that interfere with the analyses of target compounds; GPC is optional for all other circumstances), and analyzed by GC/MS for extractable organics.

Low Soil/Sediment Samples

A thirty (30) gram portion of soil/sediment is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone solution using an ultrasonic probe. If the low level screen is used, a portion of this dilute extract is concentrated fivefold and screened by GC/FID or GC/MS. If peaks are present at greater than 10,000 ug/kg, discard the extract and prepare the sample by the medium level method. If no peaks are present at greater than 10,000 ug/kg, the entire extract is concentrated, subjected to GPC cleanup, and analyzed by GC/MS for extracable organics.

Medium Soil/Sediment Samples

Approximately one gram portion of soil/sediment is mixed with anhydrous powdered sodium sulfate in a vial and extracted with methylene chloride. The methylene chloride extract can be screened for extractable organics by GC/FID or GC/MS. If organic compounds are detected by the screen, the methylene chloride extract is subjected to GPC cleanup and analyzed by GC/MS for extractable organics. If no organic compounds are detected by the medium level screen, then a low level sample preparation is required.

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PESTICIDES / AROCLORS

Water Samples

Continuous liquid-liquid or separatory funnel extraction procedures are employed for aqueous samples. A 1 L volume of sample is spiked with the surrogate solution and extracted with methylene chloride using a separatory funnel or a continuous extractor. The methylene chloride extract is dried with anhydrous sodium sulfate, concentrated and cleaned up by GPC (GPC is required when higher molecular weight compounds are present that interfere with the analyses of target compounds; GPC is optional for all other circumstances). The extract is then solvent-exchanged into hexane, cleaned up by Florisil cartridges, and the final volume adjusted to 1 mL or 2 mL. The extract is analyzed using a dual column wide-bore Chromatography/Electron Capture Gas capillary technique.

Soil/Sediment Samples

A 30 g aliquot of sample is spiked with the surrogate and then mixed with anhydrous sodium sulfate and extracted with a 1:1 acetone/methylene chloride solvent mixture by sonication. The methylene chloride extract is then cleaned up by GPC (mandatory), solvent-exchanged into hexane, cleaned up by Florisil cartridge, and adjusted to a final volume of 1 mL or 2 mL. The extract is analyzed using a dual column wide-bore capillary Gas Chromatography/Electron Capture (GC/EC) technique.

METALS

<u>Inductively Coupled Plasma - Atomic Emission Spectrometric</u> Method

The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the line are monitored by photomultiplier tubes. The photo currents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to

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the determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

Atomic Absorption Furnace Methods

In the furnace technique, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. As a greater percentage of available analyte atom is vaporized and dissociated for absorption in the tube rather than the flame, the use of smaller sample volumes or detection of lower concentrations of elements is possible. The principle is essentially the same as with direct aspiration atomic absorption, except that a furnace, rather than a flame, is used to atomize the sample. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. intensity of the transmitted radiation decreases in proportion to the amount of the ground state element in the vapor. metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the characteristic radiation from the hollow cathode lamp or electrodeless discharge lamp, and a photo sensitive device measures the attenuated transmitted radiation.

MERCURY

Water Samples

The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized and the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

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Soil / Sediment Samples

A weighed portion of the sample is acid digested for 2 minutes at 95 degrees Celsius, followed by oxidation with potassium permanganate and potassium persulfate. Mercury in the digested sample is then measured by the conventional cold vapor technique.

CYANIDE

The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetrically.

In the colorimetric measurement the cyanide is converted to cyanogen chloride, CNCI, by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-pyrazolone or pyridine-barbituric acid reagent. The absorbance is read at 620 nm when using pyridine-pyrazolone or 578 nm for pyridine-barbituric acid. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

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Superfund Target Compound List (TCL) Quantitation Limits Table 4-8

Quantitation Limits

Volatiles	Water ug/L	Low Soil ug/Kg	Med Soil ug/Kg	
Chloromethane	. 10.0	10	1200	
Vinyl Chloride	10.0	10	1200 1200	
Bromomethane	10.0	10	1200	
Chloroethane	10.0	10	1200	
Trichlorofluoromethane	10.0	10	1200	
1,1-Dichloroethene	10.0	10	1200	
Methylene Chloride	10.0	10	1200	
1,1-Dichloroethane	10.0	10	1200	
Trans-1,1-Dichloroethane	10.0	10	1200	
Chloroform	10.0	10	1200	
1,2-Dichloroethane	10.0	10	1200	
1-2-Trichlorofluoromethane	10.0	10	1200	
Carbon Tetrachloride	10.0	10	1200	
Benzene 💃	10.0	10	1200	
1,1-Dichloropropene	10.0	10	1200	
2,1-Dichloropropane	10.0	10	1200	
Trichloroethene	10.0	10	1200	
1,2-Dichloropropane	10.0	10	1200	
Bromodichloromethane	10.0	10	1200	
Toluene	10.0	10	1200	
1-2-Trichloroethane	10.0	10	1200	
Tetrachloroethene	10.0	10	1200	
Methyl tert-butyl ether	10.0	10	1200	
Chlorobenzene	10.0	10	1200	
Ethylbenzen	10.0	10	1200	
p-Isopropyltoluene	10.0	10	1200	
m & p-Xylene	10.0	10	1200	
o-Xylene	10.0	10	1200	
1-2-Dichlorobenzene	10.0	10	1200	
1-3-Dichlorobenzene	10.0	10	1200	
1-4-Dichlorobenzene	10.0	10	1200	
Naphthalene	10.0	10	1200	
n-Propylbenzene 1-4-Dichlorobenzene	10.0	10	1200	

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Quantitiaton limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, as required by the protocol, will be higher.

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Superfund Target Compound List (TCL) Quantitation Limits Table 4-9

	Qu	antitatior Low	<u>Limits</u> Med
	Water	Soil	Soil
Semivolatiles	ug/L	ug/Kg	ug/Kg
2,4-Dinitrphenol	25	800	25,000
4-Nitrophenol	25	800	25,000
Dibenzofuran	10	330	10,000
2,4-Dinitroluene	10	330	10,000
Diethyphthalate	10	330	10,000
4-Chlorophenyl phenylether	10	330	10,000
Fluorene	10	330	10,000
4-Nitroaniline	25	800	25,000
4,6-Dinitro-2-methylphenol	25	800	25,000
N-nitrosodiphenylamine	10	330	10,000
4-Bromophenyl phenlyether	10	330	10,000
Hexachlorbenzene	10	330	10,000
Pentachloraophenol	25	800	25,000
Phenanthrene	10	330	10,000
Anthracene	10	330	10,000
Carbazole	10	330	10,000
Di-n-butyl phthalate	10	330	10,000
Fluoranthene	10	330	10,000
Pyrene .	10	330	10,000
Butyl benzyl phthalate	10	330	10,000
3,3'-Dichlorobenzidine	10	330	10,000
Ben[a]anthacene	10	330	10,000
Chrysene	10	330	10,000
bis-(2-ethylhexyl)phthalate	10	330	10,000
Di-n-octyl phthalate	10	330	10,000
Benzo[b]fluoranthene	10	330	10,000
Benzo[k]fluoranthene	10	330	10,000
Benzo[a]pyrene	10	330	10,000
Ideno(1,2,3-cd)pyrene	10	330	10,000
Dibenz[a,h]anthracene	10	330	10,000
Benzo(g,h,i)perylene	10	330	10,000

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*Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight as required by Protocol, will be higher.

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Superfund Target Compound List (TCL) Quantitation Limits Table 4-10

	<u>Qu</u> Water	antitation Low Soil	<u>Limits</u> Med So i l
Semivolatiles	ug/L	ug/Kg	ug/Kg
Phenol .	10	330	10,000
bis(2-chloroethyl)ether	10	330	10,000
2-Chlorophenol	10	330	10,000
1,3-Dichlorobenzene	10	330	10,000
1,4-Dichlorobenzene	10	330	10,000
1,2-Dichlorobenzene	10	330	10,000
2-Methylphenol	10	330	10,000
2,2'-Oxybis(1-Chloropropane)#	10	330	10,000
4-Methylphenol	10	330	10,000
N-Nitroso-di-n-propylamine	10	330	10,000
Hexachloroethane ,	10	330	10,000
Nitrobenzene	10	330	10,000
Isophorone	10	330	10,000
2-Nitrophenol	10	330	10,000
2,4-Dimethylphenol	10	330	10,000
bis(2-Chloroethoxy) methane	10	330	10,000
2,4-Dichlorophenol	10	330	10,000
1,2,4-Trichlorobenzene	10	330	10,000
Naphthalene	10	330	10,000
4-Chloroaniline	10	330	10,000
Hexachlorobutadiene	10	330	10,000
4-Chloro-3-methylphenol	10	330	10,000
2-Methylnaphthalene	10	330	10,000
Hexachlororcyclopentadiene	10	330	10,000
2,4,6-Trichlorophenol	10	330	10,000
2,4,5-Trichlorophenol	25	800	25,000
2-Chloronaphthalene	10	330	10,000
2-Nitroaniline	25	800	25,000
Dimethyl phthalate	10	330	10,000
Acenaphthylene	10	330	10,000
2,6-Dinitrotuene	10	330	10,000

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3-Nitroaniline Acenaphthene 25 10 800 330 25,000 10,000

Previously known by the name bis(2-Chloroisopropyl) ether

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Superfund Target Compound List (TCL) Quantitation Limits Table 4-11

Quantitation Limits*

Pesticides/Aroclors	Water ug/L	Soil ug/Kg	
alpha-BHC	0.05	1.7	
beta-BHC .	0.05	1.7	
delta-BHC	0.05	1.7	
qamma-BHC	0.05	1.7	
Heptachlor	0.05	1.7	
Aldrin	0.05	1.7	
Heptachlor epoxide	0.05	1.7	
Endosulfan 1	0.05	1.7	
Dieldrin	0.10	3.3	
4,4 ¹ -DDE	0.10	3.3	
Endrin	0.10	3.3	
Endosulfan II ;	0.10	3.3	
4,4 ¹ -DDD	0.10	3.3	
Endosulfan sulfate	0.10	3.3	
4,4;-DDT	0.10	3.3	
Methoxychlor	0.50	17.0	
Endrin ketone	0.10	3.3	
Endrin-aldehyde	0.10	3.3	
alpha-Chlordane	0.05	1.7	
Toxaphene	5.0	170.0	
AROCLOR-1016	1.0	33.0	
AROCLOR-1221	2.0	67.0	
AROCLOR-1232	1.0	33.0	
AROCLOR-1242	1.0	33.0	
AROCLOR-1248	1.0	33.0	
AROCLOR-1254	1.0	33.0	
AROCLOR-1260	1.0	33.0	

^{*} Quantitation Limits for soil/sediment are based on wet weight. The quantitation limits calculated by the Laboratory for soil/sediment, calculate on dry weight basis, as required by the Protocol, will be higher.

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Superfund Target Compound List (TCL) Quantitation Limits Table 4-11

Parameter		Quantitation Level ug/L
Aluminum		200
Antimony		60
Arsenic		10
Barium		200
Beryllium		5
Cadmium	general control of the control of th	5
Calcium		5000
Chromium	\dot{x}	10
Cobalt		50 25
Copper		100
Iron		3
Lead		5000
Magnesium		15
Manganese		0.2
Mercury		40
Nickel		5000
Potassium	44	5
Selenium		10
Silver		5000
Sodium Thallium		10
Thaillum Vanadium		50
Zinc		20
Cyanide		10

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Metals Quantitation Limits 40 CFR Part 136 Parameters Table 4-13

Parameter		Quantitation Level ug/L
Aluminum		
Antimony		
Arsenic		10
Barium		200
Beryllium		5
Cadmium		5
Calcium	• • •	5000
Chromium		10
Cobalt	I .	50
Copper		25
Iron		100
Lead		5
Magnesium		5000
Manganese		15
Mercury		0.2
Nickel		40
Potassium		5000
Selenium	•.	5
Silver		10
Sodium		5000
Thallium		10
Vanadium		50
Zinc		20

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Conventionals Quantitation Limits 40CFR Part 136 Parameters Table 4-12

Parameter	Quantation Level ug/L
Biochemical Oxygen Demand (BODS) Chemical Oxygen Demand (COD) Ammonia, as N Total Kjeldahl Nitrogen, as N Nitrate-Nitrite; Total Phosphorous Sulfate Oil and Grease Total Organic Carbon Total Phenols Chloride	2,000 1,000 50 100 100 50 5,000 5,000 2,000 10 5,000 5,000
Fluoride Cyanide	10

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RCRA Target Compound List Volatile Organics (Method 8240) Quantitation Limits Table 4-14

10	INTE 4-14	
Volatile Organics	Low Water ug/L	Quantitation Limits Low Soil/Sediment ug/L
Chloromethane	. 10	10
Vinyl Chloride	10	10
Bromomethane	10	10
Chloroethane	10	10
Trichlorofluoromethane	10	10
Acetone	10	10
1,1-Dichloroethene	10	10
Methylene Chloride	10	10
Carbon Disulfide	10	10
1,1-Dichloroethane	10	10
Vinyl Acetate	10	10
2-Butanone	10	10
Chloroform /	10	10
1,1,1-Trichloroethane	10	10
Carbon Tetrachloride	10	10
1,2-Dichloroethane	10	10
Benzene	10	10
Trichloroethene	10	10
1,2 Dichloropropane	10	10
Bromodichloromethane	10	10
2-Chloroethylvinyl ether	10	10
4-Methyl-2-pentanone	10	10
Cis-1,3-Dichloropropene	10	10
Toluene	10	10
Trans-1,3-Dichloropropene	10	10
1,1,2-Trichloroethane	10	10
2-Hexanone	10	10
Tetrachloroethene	10	10
Chlorobenzene	10	10
Ethylbenzene	10	10
m & p-Xylene	10	10
Styrene	10	10
Bromoform	10	10
1,1,2,2-Tetrachloroethane	10	10
Dibromochloromethane	10	10
Trans-1,2-Dichlorethane	10	10

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RCRA Target Compound List Semivolatile Organics (Method 8270) Quantitation Limits Table 4-15

Parameter	Low Water ug/L	Quantitation Limits Low Soil/Sediment ug/L
Acenaphthene	10	330
Acenaphthylene	10	330
Anthracene	10	330
Benzoic acid	150	1700
Benzo(a) anthracene	10	330
Benzo(b) fluoranthene	10	330
Benzo(a) pyrene	10	330
Benzo(a/pyrene	10	330
Benzo(g,h,i)perylene	10	330
Benzyl alcohol	10	330
Bis (2-chloroethyl) ether	10	330
Bis (2-chloroethoxy) methane	10	330
Bis(2-chloroisopropyl)ether	10	330
4-Bromophenyl-phenylether	10	330
Butylbenzylphthalate	10	330
2-Chloronaphthalene	10	330
4-chlorophenyl-phenylether	10	330
Chrysene	10	330
Dibenz(a,h)anthracene	10	330
Dibenzofuran	10	330
Di-n-bytylphthalate	10	330
1,2-Dichlorobenzene	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	20	660
3,3'-dichlorobenzidine	10	330
2,4-Dichlorophenyl	10	330
Diethylphthalate	10	330
Diethylphthalate	25	800
4,6-Dinitro-2-methylphenol	10	330
N-Nitrosodimethylamine	10	550

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RCRA Target Compound List Semivolatile Organics (Method 8270) Quantitation Limits Table 4-15 (Continued)

Parameter	Low Water ug/L	Quantitation Limits Low Soil/Sediment ug/L
2,4-Dinitrophenol	. 25	800
2,4-Dimitrotoluene	10	330
2,6-Dinitrotoluene	10	330
Di-n-octylphthalate	10	330
Fluoranthene	10	330
Fluorene	10	330
Hexachlorobenzene	10	330
Hexachlorobutadiene	10	330
Hexachlorocyclopentadiene	10	330
Hexachloroethane	10	330
Indeno(1,2,3-cd)pyrene	10	330
Isophorone	10	330
2-Methylnaphthalené	10	330
2-Methylphenol (o-cresol)	10	. 330
4-Methylphenol (p-cresol)	10	330
Naphthalene	10	330
2-Nitroaniline	25	800
3-Nitroaniline	25	800
4-Nitroaniline	25	800
Nitrobenzene	10	330
2-Nitrophenol	10	330
4-Nitrophenol	25	800
N-Nitrosodiphenylamine	10	330
N-Nitrosodi-n-propylamine	10	330
Pentachlorophenol	25	800
Phenanthrene	10	330
Phenol	10	330
Pyrene	10	330
1,2,4-Trichlorobenzene	10	330
2,4,5-Trichlorophenol	10	330
2,4,6-Trichlorophenol	10	330
4-Chloroaniline	10 10	330
4-Chloro-3-methylphenol	10	330 ⁻ 330
2-Chlorophenol	10	330

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RCRA Target Compound List Pesticides/Aroclors (Method 8080) Quantitation Limit Table 4-16

Parameter	CAS	Table	Low Water (ug/L)	Quantitation Limits Low Soil/Sediments (ug/Kg)
			0.05	8.0
Aldrin			0.5	80
AROCLOR-1016 AROCLOR-1221			0.5	80
AROCLOR-1221 AROCLOR-1232			0.5	80
AROCLOR-1232 AROCLOR-1242			0.5	80
AROCLOR-1242 AROCLOR-1248			0.5	80
AROCLOR-1254			1.0	160
AROCLOR-1254 AROCLOR-1260			1.1	160
alpha-BHC	_		0.05	8.0
beta-BHC		•	0.05	8.0
delta-BHC			0.05	8.0
gamma-BHC (Lindane)			0.05	8.0
Chlordane (Total)			0.5	80
4,4'-DDD			0.10	16
4,4'-DDE			0.10	16
4,4'-DDT			0.10	16
Diedrin			0.10	16
Endosulfan I			0.10	16
Endosulfan II			0.10	.16.
Endosulfan Sulfate			0.10	16
Endrin	٠.		0.10	16
Endrin Adelyde			0.20	32
Heptachlor			0.05	8.0
Heptachlor epoxide			0.05	8.0 80
Methoxychlor			0.05	160
Toxaphene			1.1	190

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Figure 4-1

Reference	ID:		Page
		REFERENCE ST	CANDARD LOG FORM
REFERENCE	STANDARD	INFORMATION	
Standard T Manufactur			Manufacturer: Physical State:
Manufactur Lot #:	er's Expi	ration Date: Cer	Received: tificate Supplied: Y:N:
DILUENT IN	FORMATION		
Diligent: % Purity:			Mfg.Lot#:
PREPARATIO	N OF STAN	DARD	
Prepped By/On	Volume ; Used (mL)	Diluted To (mL)	Parameters, Expected Values, & Range Limits
	 		

The Reference Standards will be identified upon receipt with a reference standard (RS) ID number. The number begins with RS and is followed by the year, the month of receipt (or entry in the log), and a three digit sequential number followed by a WC. i.e. RS8910003

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PRIMARY STANDARDS QA FORM Figure 4-2

Primary Standard		_ Primary Standard ID#
Prepared by/on		Expiration Date
Neat Chemical		_ csc#
Weight/Volume	Balance Used	
Diluent		ID#
Total Volume	4.	Final Conc
Calculations:		•
		_ Primary Standard ID#
Prepared by/on		_ Expiration Date
Neat Chemical		_ csc#
Weight/Volume	Balance Used	
Diluent		ID#
m		nil oana
Total Volume		Final Conc.

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WORKING STANDARDS QA FORM Figure 4-3

Working Standard	Working Standard ID#		
Prepared by/on	Expiration Date		
Primary Std. ID#			
Conc.			
Date Prepared	Exp. Date		
Diluent	ID#		
Conc. of 1º Std.	Vol. of 1º Std. Total Vol. Conc.		
Working Standard	Working Standard ID#		
Prepared by/on	Expiration Date		
Primary Std. ID#			
	· · · · · · · · · · · · · · · · · · ·		
	Exp. Date		
Diluent	ID#		
Conc. of 1º Std.	Vol. of 1º Std. Total Vol. Conc.		

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MULTIELEMENT WORKING STANDARDS QA FORM Figure 4-4

Working Standard	Working Standard ID#
Prepared by/on	m to the day to the ten
Primary Std. ID#	
Conc	
Date Prepared	Exp. Date
Diluent	ID#
	Vol. of 1º Std. Total Vol. Conc.
	·

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4.4 Standards Preparation Procedures

Supervisors of each laboratory section are responsible for receiving the purchased standards with appropriate packing slip, purchase order and certificates relative to the standard's purity and traceability from the supplier. Labeling includes the identity of the material, date received, expiration date (if any) beyond which the standard may not be used and a Standard ID #.

Each laboratory section is responsible for maintaining the primary standards, working standards (calibrants) and reference standards for the analyses performed. Lab supervisors are responsible for taking the precautions to prevent loss, contamination, or change.

Primary standard material will be traceable to a recognized source. If no recognized source is available, a secondary source may be used at the discretion of the Laboratory Manager. All commercially available primary standards other than the test substance will be purchased with certification from the source. In addition, reference standards are used primarily to test the accuracy and validity of a procedure (i.e. as QC samples such as EMSL's) and are not used as calibrants.

When standards are used within a laboratory section, labeling for the standard should include at least the following:

- o Identity of the material
- o Concentration and date prepared
- o Standard ID # assigned and,
- o Expiration date beyond which the standard may not be used.

Primary Standards, Working Standards, and Reference Standards for the purpose of record keeping are identified as PS, WS, and RS respectively. Working Standards may also refer to Internal Standards and Surrogates, which are cross-referenced to Batch QC numbers for the purposes of data reporting.

The laboratories are identified as WC-Wet Chem, ME-Metals, GC-Organics, OE-Organic Extraction, MS-Mass Spec, PR-Pesticide Residue, MB-Microbiology and OS-Outside Service respectively for the purposes of standards labelling.

A Primary Standards QA Form (see table 4-2) is used to detail the source of a primary standard or its preparation if it is

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made from a neat chemical. These forms bound in notebook style comprise the primary standards logbook for each department and each page are numbered sequentially. The logbook also contains any certificates relative to the standard's purity and traceability from the supplier. If a primary standard must be made from a neat chemical, only ACS grade reagents are used. Traceability and preparation is fully documented on this form. Each Primary Standard is given a sequential number starting with the standard abbreviation, year designation, month, three sequential digits, and the department within which it was prepared. For example, the first primary standard prepared in January 1996 by the Metals department would be labelled PS-96-01-001-ME, the second as PS-96-01-002-ME, etc. Each month, the designation of month will change and the three digit sequence will begin again. Therefore, the first primary standard for 1996 in February would be PS-96-02-001-ME. This facilitates month to month data and performance audits rather than continuing sequentially for an entire year.

Working standards will be made from Primary Standards only and traced using the Primary Standard reference number. The Working Standards QA form (Tables 4-3 and 4-4) contains information needed to document the preparation of Working Standards from Primary Standards. This information is recorded. Working standards are identified as described for primary standards except that WS is substituted for PS.

Reference standards are used as QC samples and usually are supplied by EPA, USP or other commercial reference sources. These standards are not traceable to in-house primary standards. Figure 4-1 is an example of the reference standard log form. For the purpose of traceability and to ease the burden of transcribing multi-element information on to the Commercially Prepared Standards form, certificates of manufacture are attached. These certificates document traceability to a certified source and state analyte concentrations.

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4.5 Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action

Individuals responsible for assessing each QC measure include the following:

- a. Lab analyst must check to see that QA/QC data are within defined limits.
- Laboratory supervisor responsible for verifying sample analysis and QA/QC data.
- c. Lab manager responsible for verifying QA/QC data and sample data.
- d. QA personnel, along with lab manager, verify QA/QC data and sample data.

Corrective action items can be initiated by either b, c, or d. Approval of corrective action items for Quality Assurance/Quality Control criteria is the responsibility of the Laboratory Director.

The specific types of corrective action to be taken for each type of quality control measure are listed in Tables 4-17 through 4-19.

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Table 4-17 Corrective Action (Organics)

	rective accion (organi	
QA/QC Samples	Acceptance Criteria	Corrective Action
Holding Blank	< Reporting Limit	Recheck all samples within that holding space.
	٠	Verify with second sample from other holding spike.
		Rerun blank check for column contamination.
Laboratory Control Sample	In-house Limits	Investigate cause of contamination and reanalyze.
Initial Calibration Standard Curves	Method Specific	Check Instrument conditions, if okay, check standards.
		Rerun - remake standards and rerun
Continuing (GC/MS) Calibration Standards	Method Specific	Reanalyze. If not, acceptable, recalibrate
CCV (GC)	Method Specific	Reanalyze. If not acceptable, recalibrate and rerun sample batch to last passing CCV.
Surrogate Standard Compounds	In-house Limits; or Method Specific	Double check spiking and preparation of data. Rerun or re-extract.
Matrix Spike (MS) MS Duplicate	In-house Limits or Project Specific	Check LCS recovery; rerun or flag data.

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Table 4-18
Corrective Action (Metals)

QA/QC Samples	Acceptance Criteria	Corrective Action
Preparation Blank	Reporting Limit or Method Specific	Investigate cause of blank problem. Redigest and rerun.
Laboratory Control Sample	In-house Limits	Investigate cause and reanalzye.
Initial Calibration	Method Specified	Rerun calibration. Remake standards. Check instrument response.
Duplicate	In-house Limits or Project Specific	Check for error; Flag data.
Spike Recovery	In-house Limits or Project Specific	Reanalyze. Remake laboratory samples. Check primary and secondary standard logs. Prepare & analyze a post-spike solution.
Continuing Calibration Verification	Method Specific	Rerun samples up to last passing CCV.

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Table 4-19 Corrective Action (Inorganics)

Corrective Action (Inolganics)			
QA/QC Samples	Acceptance Criteria	Corrective Action	
Calibration Blank	< MDL	Investigate cause of blank problem. Rerun.	
Digestion Blank	< MDL .	Investigate cause of blank contamination. Redigest and reanalzye.	
Laboratory Control Sample	In-house Limits	Investigate cause of contamination and reanalyze.	
Initial Calibration Verification	90%-110%	Rerun calibration. Remake standards. Check instrument response.	
Continuing Calibration Verification	90%-110%	Rerun samples to last passing CCV.	
Spike Recovery	In-house Limits or Project Specific	Investigate cause, check LCS.	
Digestion Duplicate	In-house Limits or Project Specific	Reanalyze samples. Flag results.	

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5. Data Generation

5.1 Data Collection Procedures

All data transcribed on forms by reading off of the instrument directly, reading off of an instrument printout (includes chromatograms) and recorded after calculation will have at a minimum: sample ID number, Analyst's initials, method number of analysis, reporting units and results. Data summaries generated on software like Lotus 123, etc. are also included in this category. Figure 5.1 is an example of a manual data entry form. The file with all manual data entry forms is with the QA Department.

Analytical data is generated by the GC/MS computer software; the GC computer; the ICAP computer; AA, ion chromatograph; and associated laboratory instrumentation. Outputs include identifications of compounds, concentrations, retention times, and comparisons to standards. Outputs are in graphic form (chromatograms), bar graph (spectra) and printed tabular form, and are in standard formats specified for each analysis.

All data on an instrument printout (such as Mass Spec. printouts) will have at a minimum: Operator initials, Method #, Sample ID and results.

All data forms, whether manual, automated or electronic are archived and maintained for at least three years. All raw data, chromatograms, equipment run logs, equipment calibration, QA/QC records, chain of custody records, label information and shipping information are archived. They are stored in water-proof, fire-proof secure areas. Accessibility to these records is limited to only assigned personnel such as the QA Unit and/or lab manger.

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	Fi	igure	5-1
	PES	TICID	E/PCE
RGANICS	ANALYSIS	DATA	SHEE
	DDOTECT #	<i>t</i> •	

	NICS ANALYSIS DAT. PROJECT #:	A SHEET	(PaGe 1 of 2) BATCH QC#:	
CLIENT:	COLUMN ID#		DETECTOR:	ECD
INSTRUMENT ID		MPLE TYP		
UNITS:		MEDL III	DATE	
METHOD:	VERIFIED BY:	E ID#		
	SAMPL	E ID#		
			M)L
Parameter				ug/L ug/Kg
alpha-BHC				
gamma-BHC				
(Lindane)				
beta-BHC				··········
Heptachlor				
delta-BHC				
Aldrin				
Heptachlor				
epoxide				
Endosulfan I				
4,41 - DDE				
Dieldrin				
Endrin				
4,4 ¹ -DDD				
Endosulfan II				
				
4,41-DDT				
Endrin aldehyde				
Endosulfan				
sulfate				
Chlordane				
Toxaphene				
Methoxychlor				
Arochlor 1016				
Arochlor 1221				
Arochlor 1232				
Arochlor 1242				
Arochlor 1248				
Arochlor 1254				
Arochlor 1260				
Arochlor 1262				
Arochlor 1268				
SURROGATE				
(DSC)				
Dilution				
Factor				
Reporting				•

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Units	•
Date Run	
Date Extracted	
Initials	

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5.2 Data Reduction Procedures

Data is reduced within each laboratory by each analyst that performs the specific analysis. Each analyst is responsible for:

- Documenting all reagents/lot #'s used in the analysis.
- Calibrating and documenting all instrumentation used in the analysis.
- Running all appropriate quality control/quality assurance documentation.
- Appropriate labelling of all chromatograms and completing all test data sheets.

Lab notebooks are used to record reduction. Temperature compensation is not calculated since it is automatically adjusted by the instruments.

Formulas used to calculate final values are taken directly from the published methodologies. In general, no adjustments are made to the data. If any adjustments or corrections are made to the data such as blank subtractions, this would be documented in the final report.

Otherwise, the regression analysis used for the generation of standard curve equations for a straight line is:

$$y = B^0 + B^i x$$

Where:

 $B^0 = y intercept$

 $B^i = slope$

This equation is programmed into Lotus 123 and technicians input values from analyses and generate the equation which is used to calculate results. The straight line regression analysis is standardly used in each laboratory.

Dilution Factors

Dilution factors are calculated as follows:

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DF = <u>Volume diluted to</u> Volume used

The calculated value is multiplied by the dilution factor and reported as a final value.

5.2.1 Inorganics/Wet Chemistry

Results are generated by various instruments. Calculations used for converting results not otherwise designated in each respective methodology for solid samples are as follows:

 $mg/L \times 1000 \times Sample Volume (L) = mg/Kg$ Sample Weight (g) s % Dry Weight

ug/L x 1000 x Sample Volume (L) = mg/Kg
Sample Weight (q) s % Dry Weight

5.2.2 Metals

Results are generated by the instrument (A.A., ICP, CVAA) which converts absorbance units into results in mg/L by linear regression automatically.

Final values for water samples are calculated as follows and take into account dilution factors.

Metal Concentration $(mg/L) = A \times B$

Where:

A = Concentration of metal in digested solution, mg/L.

B = Final volume of digested solution, mL.

C = Initial sample volume, mL.

Final results for soil samples are calculated as follows on a dry weight basis.

Metal Concentration $(mg/L) = A \times B \times 100$ g Sample C

Where.

A = Concentration of metal in digested solution, mg/L.

B = Final volume of digested solution, L.

C = Total solids, %.

5.2.3 Organics

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GC analysis calculations include the following formulas calculated from the chromatograms using the external standard method of calculation. In general, calculations are performed as follows:

Water

Concentration (u/L) = (A1) (I) V1(A2) (V2) (V3)

Where:

A1 = Response for the parameter to be measured.

A2 = Response for the external standard.

V1 = Volume of total extract (uL). Take into account any dilutions.

I = Amount of standard injected in nanograms (ng).

V2 = Volume of extract injected (uL).

V3 = Volume of water extracted (mL).

Sediment/Soil

Concentration
$$(ug/Kg) = (A1)(I)V1)$$

$$(A2)(V2)(W)(D)$$

Where:

A1, I, A2, V1 = As given in equation above (for water). V2 = Volume of low level total extract (Using 20,000 uL or a factor of this when dilutions are made other than those accounted for below:

- 1/20 total extract taken for pesticide analysis (derived from 0.5 mL of 10 mL extract).
- Final concentration to 1.0 mL for pesticide analysis.

- or -

V2 = Volume of medium level total extract (Using 10,000 uL or a factor of this when dilutions are made).

$$D = 100 - % Moisture = Dry Weight$$

W = Weight of sample extracted (g).

For multicomponent analytes, retention times of peaks in

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the standards are matched with peaks in the sample. Every identifiable peak is quantitated (>50% of the total area must be used) unless interference with individual peaks persist after cleanup. Peak height or peak area of each identified peak in the chromatogram is added. Total response in the sample versus total response in the standard is calculated.

Calculation for surrogate and matrix spike recoveries.

Percent Recovery = $Q \times 100\%$

Where:

Q = Quantity determined by analysis.

S = Quantity added to sample.

All dilutions should be taken into account. Soil/Sediment has a 20-fold dilution factor built into the method when accounting for one-twentieth of extract taken for pesticide analysis and final dilution to 1 mL.

5.2.4 Organics GC/MS:

EPA Method # 8240/624 - (Water & Water-Miscible Waste/Waste water):

$$ug/L = (A^{x}) (I^{s})$$

$$((A^{is}) (RF) V^{o})$$

Where:

Ax = Area of Characteristic ion for target compound. Is = Amount of internal standard injected (ng)
Ais = Area of characteristic ion for internal standard
RF = Response factor for target compound
Vo = Volume of water purged (mL), taken into consideration any dilutions made

EPA Method # 8240 - Sediment (Soil, Sludge & Waste):
High-level:

$$ug/Kg = \frac{(A^{x}) (I^{s}) V^{r}}{(Ai^{s}) (RF) (V^{i}) (W^{s})}$$

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Low-level:

$$ug/Kg = \frac{(A^x) (I^s)}{(Ai^s) (RF) (W^s)}$$

Where

Ax, Is, Ais, RF = Same as for Water above.

Vt = Volume of total extract (uL) (use 10,000 uL or a factor of this when dilutions are made.

Vi = Volume of extract added (uL) for syringe.

Ws = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific application of the data.

EPA Method # 8270 - (Water):

$$ug/Kg = \frac{(A^{x}) (I^{s}) (V^{r})}{(Ai^{s}) (RF) (V^{0}) (V^{1})}$$

Where:

Ax = Area of characteristic ion for target compound.

Is = Amount of internal standard injected (ng).

Vt = Volume of total extract, taking into account
 dilution.

Ais = Area of characteristic ion for internal standard.

RF = Response factor for compound being measured.

Vo = Volume of water extracted (mL).

Vi = Volume of extract injected (uL).

EPA Method # 8270 - Sediment/Soil Sludge (on dry-weight basis) & Waste (on wet-weight basis).

$$ug/Kg = \frac{(A^{x}) (I^{s}) (V^{r})}{(Ai^{s}) (RF) (V^{i}) (W^{s}) (D)}$$

Where:

Ax, Is, Vt, Ais, RF, Vi = Same as for water above.

Ws = Weight of sample extracted or diluted in grams D =
 (100 - % moisture in sample) /100, or 1 for a water
 weight basis.

Calculations for surrogate and matrix spike recoveries are the same as for GC analysis.

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5.3 Data Validation Procedures

During data generation the managerial staff closely observes and supervises the technique of the analyst. In addition to the adequate training of the analytical staff, careful attention is paid to the following aspects of data generation.

- a. Recording of important observations.
- b. Methods direction adherence/proper methodology.
- c. Reading instrument data and volumetric measurements.
- d. Transcription of data.
- e. Key punching in data handling systems.
- f. Data reduction.
 - g. Retention times and peak heights.
 - h. Standardization and traceability.
 - i. Purity and traceability of reagents.
 - j. Clean glassware.
 - k. Traceability and verification of primary standards to secondary standards.
 - 1. Proper documentation of concentration values and conversions thereof.
 - m. Equipment calibration and preventive maintenance.
- n. Holding time and preservation checks.

A list of activities used to verify data integrity by the Laboratory Supervisor includes the following:

- a. Check raw data entries and calculations at a frequency of 10 %.
- b. Check extraction and preparation logs.
- c. Check column logs and instrument logs.
- d. Checking standard curves and instrument initial

calibration verification and continuing calibration verification records. Reviewing control charts for systematic or random problems related to instrument sensitivity and linearity.

e. Internal chain of custody is verified by the lab supervisor daily for all work orders.

Chromatograms are identified by the information provided as follows:

Sample	No
Operator Analysis	Date

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The carrier gas pressures and column condition (i.e. temperature) are preset before an analytical run, as are conditions of the integrators.

Test data sheets must include all calculations, QA/QC documentation, dates, and initials of analysts.

Computer record identification must include at a minimum the run #, method, work order #, and must be signed and dated by the analyst.

Data are calculated on the raw data form (test data sheet) associated with each test and the equations are provided with each data form. If computer data is required for standard curves and related correlation coefficients, a computer generated linear or logarithmic regression program is utilized. Single point reference calculations are generated both manually and with the use of Lotus 1-2-3 for simple calculations and miscellaneous equations. Data entry from paper format is entered into the SAM (laboratory management network) by the analyst generating the data or the data manager.

Quality Assurance Personnel check the following items;

- a. Calculations checks.
- b. Dilution checks.
- c. Precision and accuracy checks of spikes, duplicates, calibration standards, blanks.
- d. Completeness checks.
- e. Representativeness checks.
- f. Comparability checks.
- f. Statistical checks (regression analysis etc.).
- h. Transcriptional checks.
- i. Sample identification and matrix checks.
- j. Review of all chain of custody information and verification of sample identification and holding times.

5.4 Data Reporting and Authorization Procedures

Final reports are generated from the laboratory network data system (SAM).

Data within the SAM system cannot be written and transmitted to a final report without a data verification step (computerized). Computerized data verification can be performed only by the lab manager, lab supervisor, or the QA

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Officer.

Once the final report is completed, it is printed out and compared with the raw data. A form is signed which is part of each work order as follows:

LABORATORY DATA VERIFICATION EVIDENCE

Work Order #:	
Wet Chemistry:	Date:
Verified by	
Metals:	Date:
Verified by	
Organics:	Date:
,	-
LABORATORY	
MANAGER:	Date:

The Laboratory Information Management System (LIMS) at Toxikon (called SAM) assures security and data integrity by using user privileges, which defines various levels of accessibility. Figure 5.2 is an example of the user privileges that are employed. Only authorized personnel such as the lab manager, QA officer and data reduction personnel can access information that can be edited.

Once a project is logged into SAM, test codes specific to the project are entered for that project number by the data reduction personnel. The system automatically provided screens for data entry by the lab supervisors or his/her designee. Each of these screens has a space provided for the initials of the lab supervisor, to indicate that they were verified after data entry. Once the results for a project have been entered, the Lab Manager and/or the QA Officer verify that the information is correct. The system then requires that the Lab Manager/ QA Officer enter their initials on a special screen provided for the purpose, indicating that the results have been verified. Only after the verification step can a copy of the final results be printed out and sent to the client.

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Figure 5.2

User name: BIFF

Password:

Allowed to create, modify or delete subcontractors -- N
Allowed to create, modify or delete tests -- N
Allowed to create, modify or delete global jobs -- N
Allowed to create, modify or delete clients -- N
Allowed to create, modify or delete projects -- N
Allowed to create or modify work orders -- N
Allowed to create, modify or delete fractions -- N
Allowed to transmit work orders -- N
Allowed to enter/review results on work orders -- N
Allowed to alter work status via the COMP program -- N
Allowed to verify completed work orders -- N
Allowed to reopen, roll-up, reduce of delete work orders -- N
Allowed to access confidential information -- N
Allowed to see and/or set QA/QC flag -- N

Write this account? (y if yes) [N]

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6. Quality Control

6.1 Solvent, Reagent and Adsorbent Check Analysis

Reagent and Solvent Blanks are prepared in the laboratory from method solvents and reagents, these blanks assist in eliminating contamination from commercially supplied chemicals. Each new lot or batch is checked as it is used. Method reagent blanks are prepared and analyzed at a rate of one per sample set.

Adsorbent performance checks are described in the individual method SOP's and are required for each batch or lot of adsorbent or cartridges.

6.2 Reference Material Analysis

The purity of a compound can sometimes be misrepresented by a chemical supply house. Since knowledge of purity is needed to calculate the concentration of solute in a solution standard, it is the Laboratory's responsibility to have analytical documentation ascertaining that the purity of each compound is correctly stated.

Mis-identification of compounds occasionally occurs and it is possible that a mislabeled compound may be received from a chemical supply house. It is the Laboratory's responsibility to have analytical documentation ascertaining that all compounds used in the preparation of solution standards be correctly identified. Identification confirmation, when performed, should use, gas chromatographic/mass spectrometry analysis on at least two different analytical columns, or other appropriate techniques.

Solutions of analytical reference standards can be purchased by Laboratories provided that the laboratories maintain the following documentation to verify the integrity of the standard solutions they purchase:

- mass spectral identification confirmation of the neat material
- 2) purity confirmation of the neat material
- 3) chromatographic and quantitative documentation that the solution standard was QC checked

It is the responsibility of Toxicon to maintain the necessary

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documentation to show that the chemical standards they have used in the performance of ASP analysis conform to the requirements previously listed. Weighing logbooks, calculations, chromatograms, mass spectra, etc., whether produced by the Laboratory or purchased from chemical supply houses, must be maintained by the Laboratory and may be subject to review during On-Site inspection visits. In those cases where the documentation is supportive of the analytical results of data packages sent to NYSDEC, such documentation is to be kept on file by the Laboratory for a period of one year.

6.3 Internal Quality Control Checks

The objectives of the Quality Assurance Program are to provide analytical chemistry data of known quality and defensibility. Daily, routine analyses of blank, replicate and spiked samples (or surrogate compounds) provides precision, accuracy and contaminated data for assessing the validity of analytical results. Method Detection Limits (MDLs) are determined by procedures outlined in 40 CFR Part 136, Appendix B.

For ASP, after award of a contract and before the first sample results are due and on a semiannual basis thereafter, using standard reference materials, Toxicon will determine the analytical instrument detection limits (IDL's).

The general quality control measures and the frequency with which they are employed are tabulated in this section. However, a comprehensive listing is beyond the scope of this document as they are method-dependent and will vary depending on the analyses chosen. A protocol or SOP used for a particular analysis will contain a detailed description of the quality control measures to be employed. Usually, QC check standards are analyzed at a continuing frequency of 5% of samples in the analytical set or analyzed at the start of the run to verify the standard curve.

The integrity of data produced by the laboratory is ensured through the analysis of blanks, spikes, and duplicates. The quality of organic analyses is further ensured by the analyses of surrogate compounds and quantitation of unknowns. Additional QC checks (eg. reagent purity checks) are analyzed if required by method. Analysis of QC samples is interspersed with that of unknown samples at a regular interval. Continuous calibration standards are analyzed at a rate of one every ten samples in an analytical set.

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Trip (Travel) Blank Analyses

Volatile organics samples are susceptible to contamination by diffusion of organic contaminants through the Teflon-faced silicon rubber septum of the sample vial. Trip blanks, prepared by filling two 40-ml VOA vials with organic free water, are shipped with the field kit, and follow the sample bottles through the field collection and shipment to the laboratory. Trip blanks are analyzed with the sample set.

Equipment Blanks

An equipment blank is a volume of deionized water or organic solvent that is used to rinse a sampling tool which contacts multiple samples to demonstrate that there is no residual contamination remaining on the tool to carry over to succeeding samples. Equipment blanks are analyzed with each sample set that a sampler sends.

Field Blank Analyses

A field blank is a volume of deionized water, or purified soil, that is placed into sample containers at the site by the sample takers and is shipped with the field samples. Field blanks are analyzed with each sample set that a sampler submits.

Reagent and Solvent Blanks

Reagent and Solvent Blanks are prepared in the laboratory from method solvents and reagents. These blanks assist in identifying contamination from commercially supplied chemicals. Each new lot or batch is checked as it is used. Method reagent blanks are prepared and analyzed at a rate of one per sample set.

Method Blank Analyses

A method blank is a volume of deionized water or sample of purified soil (heated in a muffle furnace for 18 hours at 400°C) that is carried through the entire analytical procedure to verify that interferences caused by contaminants in the solvents, reagents, glassware, etc. are known and minimized. A method blank is analyzed with each group of samples.

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Laboratory Holding Blank

A laboratory holding blank is prepared from laboratory deionized water. These blanks are placed in cold storage with the volatile organics samples. A holding blank is analyzed weekly.

Blank Spike Analyses (Laboratory Control Sample)

An LCS is a volume of deionized water or a sample of purified soil that is spiked with the analytes of interest and carried through the entire analytical procedure to demonstrate that the laboratory techniques for this method are in control. This sample is recommended in conjunction with matrix spike/matrix spike duplicate samples where severe matrix interferences are anticipated. If the matrix spike/matrix spike duplicate shows poor recoveries while the blank spike sample is acceptable, this is strong evidence that the method has been performed correctly by the laboratory but that matrix interferences have affected the results of these samples.

<u>Duplicate Analysis</u>

Duplicate samples are analyzed at a rate equivalent to 5% of all samples analyzed in the lab for an analytical batch. The relative percent difference between the samples is calculated and used to assess analytical precision.

Matrix Spike Analysis

Matrix spikes are routinely used in the analysis of inorganic and organic matrices for an analytical batch. In matrix spike analysis, predetermined quantities of certain analytes are added to a sample matrix prior to sample extraction/digestion and analysis.

Laboratory Matrix Spike Duplicate Analyses

A separate aliquot sample is spiked with the analyte(s) of interest and analyzed with the associated sample and sample matrix spike.

Surrogate Spike Analysis

The surrogate is a non-method-analyte spiked into a sample aliquot prior to extraction and is measured with the same procedures used to measure other sample components. These compounds are spiked into all blanks, standards, samples and

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spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates are used to monitor the performance of the method for each sample.

Internal Standard

Samples are spiked with the internal standard immediately prior to analysis to assist in quantitation and to indicate short-term instrument performance. The concentration of the unknown is calculated using the general formula:

$$Concentration = [As] [Cis]$$
 $[Ais] [RF]$

Where:

As = Response for the parameter to be measured Ais = Response for the internal standard Cis = Concentration of the internal standard RF = Response Factor from the initial calibration

Continuing Calibration Check

Because standards and calibration curves are subject to change, a midpoint standard or check standard is frequently analyzed with each group of samples to verify the curve.

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Frequency of QA Checks

Organics

Table 6-1

Quality Control Measure	Frequency
Holding Blank (for VOA*)	One per week
Holding Blank (for von)	The Para
Laboratory Method Blank (VOA)	One per 12 hours (or batch analysis
Continuing Calibration Check	One per 12 hours or as specified in meth
Surrogate Standard	Added to each sample, bla and standard
Matrix Spike/Matrix Spike Duplicate	One set per 20 samples
GC/MS Tune	Once per day (600 Series) Every 12 hours (80
Series)	•

*VOA = Volatiles

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Frequency of QA Checks

Metals

Table 6-2

Quality Control Measure	Frequency
Preparation Blank	One per group of 20 or fewer samples
Matrix Spike	One per group of 20 or fewer samples
Duplicate	One per group of 20 or fewer samples
Laboratory Control Spike	One per group of 20 or fewer samples
Initial Calibration Verification	Daily or each time instrument is calibrated
Continuing Calibration Verification	One per every 10 analyses

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6.4 Corrective Action and Determination of QC Limit Procedures

Individuals responsible for assessing each QC measure include the following:

- a. Lab analyst must check to see that QA/QC data are within defined limits.
- b. Laboratory supervisor responsible for verifying sample analysis and QA/QC data.
- c. Lab manager responsible for verifying QA/QC data and sample data.
- d. QA personnel, along with lab manager, verify QA/QC data and sample data.

Corrective action items can be initiated by either b, c, or d.

External sources are also used to check laboratory performance. Corrective action can be initiated by results of EPA WS/WP results, split samples, trip/field blanks, etc. In addition, the internal QA/QC laboratory system and performance audit results can be used to initiate corrective action if, deficiencies are noted at the time of the audits. Outside audits by clients/regulatory agencies can also be used to initiate corrective action items.

Presently, Toxikon is employing control charts in the laboratory to control the variability or precision between duplicate analyses and to control the accuracy for spike recovery measurements.

The procedures used for establishing and updating control limits are contained in a separate, detailed SOP. As stated in this SOP, control limits for bias are based on the historical mean recovery plus or minus three standard deviation units, and control limits for precision range from zero (no difference between duplicate control samples) to the historical mean RPD plus three standard deviation units. For methods that contain specific control limits, those limits are used in place of historical in-house limits.

Precision and accuracy are monitored over the long term using control charts. Control charting provides a means for separating or analyzing the determinate (systematic) from the indeterminate (random) sources of variation within data sets.

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Precision:

The degree of agreement between repeated measurement of the same property under similar conditions. Using duplicate analytical results, precision is expressed as Relative Percent Difference (RPD) and calculated as follows:

RPD =
$$/D1 - D2/ \times 100$$

(D1 + D2) ÷ 2

Where:

RPD = Relative Percent Difference

D1 = First Duplicate Value D2 = Second Duplicate Value

Upper Warning Limit = RPD + 2 SD Upper Control Limit = RPD + 3 SD

For multiple results, Relative Standard Deviation (RSD) is used. RSD is calculated by:

$$RSD = \underbrace{S}_{X} \times 100$$

Where:

S = Standard Deviation of a set of numbers.

X = Mean of the set of numbers

Accuracy: The degree of agreement between an experimentally determined value and the accepted reference value. Analytical accuracy is measured by calculating the recovery of spiked or reference sample analytes.

Percent recovery is calculated as follows:

From replicate analyses, average percent recovery and standard deviation are calculated. The control limits are set as follows:

Upper Control Limit = Average percent recovery + 3 SD Lower Control Limit = Average percent recovery - 3 SD

Upper Warning Limit = Average percent recovery + 2 SD Lower Warning Limit = Average percent recovery - 2 SD

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6.5 Responsibility Designation

The functional units responsible for data review include:

Analyst
Laboratory Manager
Quality Assurance Unit (QAU)

The first level of Quality Control lies with the trained bench analysts conducting the analyses. Proper documentation and peer review are important aspects of laboratory management at this level.

The Laboratory Manager is responsible for ensuring that adequate facilities and equipment are available to the analysts to ensure the production of scientifically and technically valid data. The Manager interacts closely with the analysts and ensures that the laboratory Quality Control (QC) procedures are strictly adhered to.

The QAU is responsible for auditing the laboratory facilities, procedures, study conduct, and raw data. The results of these audits are presented to the Laboratory Director and may be used, when required, to decide upon corrective action. The QAU assists in upgrading the management systems and analytical procedures in the laboratory through performance and system audits.

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7.0 Quality Assurance

7.1 Documentation of Appropriate Laboratory Certifications

Toxikon currently holds certifications from the states of Connecticut, Florida, Maine, Maryland, Massachusetts, New Hampshire, New Jersey, New York, North Carolina, Pennsylvania, Rhode Island, and South Carolina.

TOXIKON CORPORATION (Laboratory ID 88002) Certifying Authority: NY Certificate Number: 88002001 Date of Issue: 07/08/1997 Expiration Date: 08/15/1997 CLEAN WATER ACT

SEMI-VOLATILES

POLYNUCLEAR AROMATIC HYDROCAR. EPA 610

VOLATILES (VOCS)

PURGEABLE AROMATICS PURGEABLE HALOCARBONS EPA 602 EPA 601

INORGANIC - TRACE METAL

LEAD EPA 200.7 EPA 239.2 LEAD

INORGANIC - MISCELLANEOUS

OIL & GREASE EPA 413.1 TOXIKON CORPORATION (Laboratory ID 88002)
Certifying Authority: VY

Certificate Number: 38002001

Date of Issue: 07/08/1997 Expiration Date: 08/15/1997

EPA 3080A

EPA 3140

SOLID & HAZARDOUS WASTES

· ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~	PCBS	AND	PESTICIDES	3
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ORGANO. PEST. & PCBS
ORGANOPHOSPHATE PESTICIDES

HERBICIDES

CHLOROPHENOXY ACID HERBICIDES EPA 3150B

SEMI-VOLATILES

PHENOLS (ORGANICS) EP.A 3040A

VOLATILES (VOCS)

ACROLEIN & ACRYLONITRILE EPA 8030A

ENORGANIC - TRACE METAL

YNOMITAL EPA 6010A ANTIMONY EPA 7040 ARSENIC EPA 6010A ARSENIC EPA 7060A BARIUM EPA 6010A CADMIUM EPA 6010A CADMIUM EPA 7131A CHROMIUM EPA 6010A CHROMIUM, HEXAVALENT EPA 6010A LEAD EPA 6010A LEAD EPA 7421 MERCURY EPA 7470A MCKEL EPA 6010A SELENIUM EPA 6010A SELENIUM EPA 7740 SILVER EPA 6010A

INORGANIC - MINERAL

HYDROGEN-ION CONCENTRATION EPA 9040B

INORGANIC - MISCELLANEOUS

CYANIDE EPA 9010A SULFIDE EPA 9030A

INORGANIC - HAZARDOUS WASTE

IGNITABILITY (PENSKY MARTENS)

REACTIVITY - CYANIDE

REACTIVITY - SULFIDE

TCLP - BOTTLE EXTRACTION

EPA 1010

S.7.3. - SW846

EPA 1311

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7.2 Data Quality Assurance

7.2.1 Toxikon will maintain a written Quality Assurance Plan which describes the general and specific procedures used within the laboratory to achieve scientifically sound and legally defensible data.

7.2.2 The quality of data produced by the laboratory is assessed by the evaluation of precision, accuracy, representativeness, completeness, comparability and sensitivity.

7.2.2.1 Precision

Precision is the degree of agreement between repeated measurement of the same property under similar conditions. For the chemical analysis of environmental samples, precision is commonly determined from laboratory duplicate samples (i.e., matrix spike/matrix spike duplicates, or matrix duplicate samples). To establish the precision of a given analytical method, the laboratory control sample would typically be used. Using duplicate, analytical results, precision is expressed as Relative Percent Difference (RPD) and calculated as follows:

RPD =
$$\frac{D1-D2}{x 100}$$
 x 100 (D1+D2)/2

RPD = relative percent difference

D1 = first duplicate value

D2 = second duplicate value

For multiple results, Relative Standard Deviation (RSD) is used. RSD is calculated by:

where, S = Standard Deviation of a set of numbers X = Mean of the set of numbers

Control limits are defined as:

Upper Control Limit = RSD + 3 SD Lower Control Limit = RSD - 3 SD

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Upper Warning Limit = RSD + 2 SD Lower Warning Limit = RSD - 2 SD

7.2.2.2 Accuracy

Accuracy is the degree of agreement between an experimentally determined value and the accepted reference value. Accuracy is commonly determined in the laboratory from spiked samples (i.e., matrix spikes, laboratory control samples, surrogate spiked, etc.) or performance evaluation samples. Accuracy is measured by calculating the recovery of the spiked or reference sample analyte.

Percent recovery is calculated as follows:

P = Observed Value x 100 Known Value

From replicate analyses, average percent recovery and standard deviation are calculated. The control limits are set as follows:

Upper Control Limit - Average percent recovery + 3 SD Lower Control Limit - Average percent recovery - 3 SD

Upper Warning Limit = Average percent recovery + 2 SD Lower Warning Limit = Average percent recovery - 2 SD

7.2.2.3 Representativeness

Representativeness refers to the degree to which sample data accurately and precisely describes the characteristics of a population of samples. The representativeness criteria is best satisfied in the laboratory by making certain that all subsamples taken from a given sample are representative of the entire sample. This would include sample premixing and the discarding of obvious foreign objects. Samples requiring volatiles analysis should not undergo any premixing or homogenization. Samples that are not properly preserved or are analyzed beyond acceptable holding times should not be considered to provide representative data.

7.2.2.4 Completeness

Completeness is the percentage of a set of

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measurements made which are judged to be valid.

7.2.2.5 Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable for similar samples and sample conditions. This goal is achieved through the use of standard techniques to collect and analyze representative samples and reporting analytical results with appropriate units.

7.2.2.6.1 Method Detection Limit

The method detection limit (MDL) is the minimum concentration of a substance 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 The laboratory will perform MDL analyte. MDLs are estimated in an studies annually. interference-free matrix, typically reagent water for water methods and a purified solid matrix (e.g., sand) for soil/sediment methods. Method detection limits are estimated for each method target analyte using the procedures presented in 40 CFR, Par 136, Appendix B. The MDLs are extraction/digestion method-specific and shall include any clean-up methods used. the MDLs must be demonstrated on individual instruments, or on individual detectors if appropriate, when multiple instruments are used for any given method.

7.2.2.6.2 Method Quantitation Limit

The method quantitation Limit (MQL) is the lowest calibration standard and should be no lower than ten times the standard deviation as determined from the MDL study. In the absence of project-specific requirements, the lowest calibration standard used for initial calibration is set at between three to ten times the MDL for each method target analyte. The low standard will not be set at a value that is lower than the MQL.

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7.2.2.6.3 Method Reporting Limit

In the absence of project-specific reporting limits, the Method Reporting Limit (MRL) will be set at the value of the low standard used during initial calibration. The low standard would typically be set at one half the project-specific action levels or regulatory levels. The low standard could not be set at higher concentration than the projectspecific action levels. Analyte values reported below the low standard shall be reported as estimated (flagged) values based on the analyst's judgment. MRLs are adjusted based on the sample matrix and any necessary sample dilutions. The definition of MRLs must be declared in each data package.

- 7.2.3 Toxikon will maintain written, approved laboratory specific standard operating procedures (SOPs) for all methods and general operations that fully detail the actual procedures and documentation used to implement the methods. Copies of the appropriate SOPs will located in each section of the laboratory (Volatiles, Semivolatiles, Metals, etc.) Laboratory SOPs are controlled documents with unique ID numbers which are reviewed annually and updated as necessary. Retired SOPs are maintained by the laboratory in case quality questions arise later.
- 7.2.4 For each method performed, the laboratory will maintain documentation of the employee's ability to perform the method. This will require that the employee analyze at least four aliquots of a laboratory control sample and meet the method's precision and accuracy limits.
 - 7.2.5 Toxikon will work with clients to assure that samples are collected in the appropriate containers and are properly preserved. Toxikon will maintain documentation that all sample holding times for extraction and analysis have been met. All samples and their associated extracts will be stored under conditions that will ensure their integrity. Samples will not be stored with standards. Samples designated for volatile organics testing will be segregated from other samples.
 - 7.2.6 Toxikon's building is protected by a fully integrated, state-of the art security system (ADT Focus

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100 D). Employees must place security cards in computer keypads to enter the building. Visitors are escorted at all times. Samples are stored in a locked room with a full time sample custodian. further, to ensure security, locked refrigerators are provided for the storage of samples.

7.3 Internal Audits

7.3.1 System Audits

Quarterly, the Quality Assurance Unit conducts an audit of the Environmental Laboratory. This system audit is an on-site qualitative check of the quality assurance systems and physical facilities in the laboratory for sampling, calibration and quantitative measurement. Systems audits determine that each element within an activity of functioning appropriately and within the approved methodology, of appropriate quidelines procedures and QA plan. Some of the components of a system audit include personnel, facilities, and equipment custody procedures, instrument chain -of calibration and maintenance, standards preparation and verification, analytical procedures, quality control procedures, data handling procedures, and documentation control procedures. If any of these components are found to be deficient a corrective action is immediately taken to improve or modify the system.

7.3.2 Corrective Action Audits

Audits may be conducted on a particular aspect, area or method in the laboratory if routine data review or the review of correction action forms indicates that a problem or deficiency may be present. An audit may also be triggered by questions from clients about their sample data or reports.

7.3.3 Blind Performance Evaluation Samples

Due to the number of state certifications that Toxikon currently holds, the laboratory analyzes a number of performance evaluation samples submitted by the states. However, if there is a significant period of time over which no performance samples are received, laboratory management or the quality assurance unit may choose to introduce blind performance evaluation samples into the Toxikon laboratory. Correction action is triggered if

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any laboratory results are outside of the acceptance limits.

7.4 Performance/External Audits

7.4.1 State Audits

Toxikon currently holds certifications form the States of Connecticut, Florida, Maine, Maryland, Massachusetts, New Hampshire, New Jersey, New York, North Carolina, Pennsylvania, Rhode Island, and South Carolina. Toxikon analyzes performance evaluation samples submitted by these states and is subject to on-site audits by these states.

7.4.2 Federal Agency Audits

Toxikon is subject to audits for the Environmental Protection Agency. When applying for certification from agencies such as the USACOE, Toxikon is subject to indepth on-site audits and the successful analysis of performance evaluation samples prior to initiation of work on any project.

7.4.3 Client Audits

Prior to the start of a major project, engineering firms or industrial clients may submit performance evaluation samples or schedule on-site audits.

7.5 Corrective Action Procedures

When errors, deficiencies, or out-or-control situations exist, the laboratory's QA program shall provide systematic procedures, called 'corrective actions,' to resolve problems and restore proper functioning to the analytical system(s).

7.5.1 Corrective Action by the Analyst

Corrective actions are often handled at the bench level by the analyst, who reviews the sample preparation procedures for possible errors, checks the instrument calibration, spike, and calibration mixes, instrument sensitivity, etc. Analysts are alerted that corrective actions are necessary if: (1) QC data are outside the acceptable windows for precision and accuracy; (2) blanks or laboratory control samples contain contaminants above

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acceptable levels; or (3) undesirable trends are detected n spike recoveries or RPD between duplicates.

7.5.2 Corrective Action by Management or the Quality Assurance Unit

If a problem persists or cannot be identified at the analyst level, then laboratory management or the quality assurance unit must initiate a Corrective Action Report Form (Fig. 7.1) to investigate and find a solution to the problem.

The basic outline of the correction action program is:

- Define the problem
- Assign responsibility for properly investigating the problem
- Determine corrective action to eliminate the problem
- Assign responsibility for implementing the correction action
- Establish effectiveness of the corrective action
- Verify that the corrective action has eliminated the problem
- Fully document the corrective action items and responses

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Figure 7-1

CORRECTIVE ACTION REPORT

Project Number:		
Client:		
Parameter (s):		
Date Sample (s) received:		
Date Analyzed: Date of Data Package Audit:		
Date of System Audit:		
Date of System Audit:		
Observation (s):		
	1.49	
Corrective Action Require	ed/Reason:	
Corrective Action Due Dat	e:	
Responsible Personnel:		-
		-
QAU Auditor		Date
Received and Reviewed by:		
Received and Reviewed by.		
	Lab Director	Date
		*
	Lab Manager	Date
	Responsible Perso	nnel Date
	Responsible Perso	nnei Date
Response/Corrective Action		
,		
Date:		
FINAL REVIEW		D 1
Acceptable Unacceptable	leQAU int	Date

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7.6 Quality Assurance Reporting Procedures

7.6.1 A Quality Assurance Report is submitted to the Scientific Director and the V.P. Regulatory Affairs at the conclusion of each quarterly Systems Audit. Copies are circulated to the Laboratory Manager, the Technical Director, and group Supervisors. Any deficiencies noted during this quality audit require that the Quality Assurance Unit initiate a Corrective Action Report Form for each deficiency. The results of Corrective Action Audits are documented in the monthly report sent to the Scientific Director by the QAU. Any deficiencies noted also require the initiation of a Corrective Action Report Form.

7.6.2 External Audits

On-site audits require two written reports. The first report written by either the inspecting agency or Toxikon's QAU must list any deficiencies noted during the audit. The second report written by laboratory management or the QAU must specifically state the action(s) taken to eliminate or correct the deficiencies noted during the audit.

7.6.3 All reports on System or External Audits will be maintained for a minimum of three years.

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			Figure 1
			-
			-

State of Connecticut, Department of Public Health

Approved Environmental Laboratory

APPLICABLE PROVISIONS OF THE PUBLIC HEALTH CODE AND GENERAL STATUTES OF CONNECTICUT, FOR MAKING THE EXAMINATIONS, THIS IS TO CERTHY THAT THE LABORATORY DESCRIBED BELOW HAS REEN APPROVED BY THE STATE DEPARTMENT OF PUBLIC HEALTH PURSUANT TO DETERMINATIONS, OR TESTS SPECIFIED BELOW WHICH HAVE BEEN AUTHORIZED IN WRITING BY THAT DEPARTMENT

TOXIKON CORPORATION

THIS CERTIFICATE IS ISSUED IN THE NAME OF AND REGISTERED IN THE NAME OF LOCATED AT ..15 Wiggins Avenue.....IN ...I.AXMAN S. DESAL..... ..I.AXMAN S. DESIA..... ... Bedford, Massachuseus 01730.

DESIGNATED BY THE REGISTRANT TO BE IN CHARGE OF THE LABORATORY WORK COVERED BY THIS CERTIFICATE OF APPROVAL

POTABLE WATER, WASTEWATER AND/OR TRADE WASTE, SEWAGE AND/OR EFFLUENT, SOIL Examination for:

Inorganic Chemicals
Organic Chemicals

SEE COMPUTER PRINT-OUT FOR SPECIFIC TESTS APPROVED

THIS CERTIFICATE EXPIRES JUNE 30, 1999 AND IS REVOCABLE FOR CAUSE BY THE STATE DEPARTMENT OF PUBLIC HEALTH DATED AT HARTFORD, CONNECTICUT, THIS 31st · ; · · · DAY OF. · · · July . · 1997 .



PI-I-0563

Eaux Shur

DIRECTOR, DIVISION OF ENVIRONMENTA! "JALT"

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140		CHRUMIUM TOTAL	
	TEST 278	CHROMITMORES	
	TEST 250	CUBALT COBBER	
**	753T 282	ISON	
	<u> </u>	MAGRESIUM	
	TEST 255	MANGANESE	
<u> </u>	TEST 207	AERCURY MOLYREDWIM	
. <u> </u>	<u> </u>	N'CKSL	
	T=ST 259	SELENIUM	
	TEST 291	311 VE 3	
	TEST 292 TEST 293	SODIUM	
	TEST 294	THALL TIM VANAD IUM	
	TEST 297	7.7.5	
	1EST 311	ORGANIC CHEMICALS PRAGRAME HALDCARHONS	
	T2 ST 317	PURGEABLE ARCHAILUS	
-	153T 313 153T 314	>=STTC1)E3	
	7 = ST 310	565 TM 37!	
	7887 317 7887 316	SCIMDREIC BASTALE SCIMDREIC S.O. S.	
	TEST 320	OIL AND GREASE	
	TEST 331		
			
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	CONNECTION STATE DEPARTMENT OF HEALTH
- YENDAY AUGUST	LABORATORY DIVISION 11 1997 2:30 PM
	REGISTRATION DATE U7/01
PHOSOJ - TOXIKOM CORPORATI	
REGISTRANT LAXMA	N S. DESAI DIRECTOR LAXMAN S. DESAI
-RECMUN ERACICEP	INTERSTATE NUMBER-
	S 44 TS4
TEST 201 WASTER	ATER AND/OR TRADE WASTE
TEST 203 3011	14-1-1-4
TEST 231	INDREAMIC CHEMICALS
T=3T 233	COLOR
7537 235	TURSIDITY
780T 236 780T 237	
758T 239	COMBUCTIVITY MINERALS
7887 241	ALKALINITY
TEST 247	CHLORIDE
TEST 249	CHLORINE CHLORINE
TEGT 251	AMMONIA
7837 384 7837 255	BTTATTE STINTER
7537 250 7637 257	23740-PH00PHATE
	TOTAL PHOSPHORUS
TEST 259	TOTAL SOLIDS
7=37 231 	TOTAL VOLATILE SOLIDS
TEST 203	CYANIDE
TEST 207	30D
TEST 208	T3C
	ALUMINUM
	1 + 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 -
TEST 274 TEST 275	ARSENIC

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State of Florida Department of Health BUREAU OF LABORATORIES ENVIRONMENTAL WATER

This is to certify that



E87143
Toxikon Corporation - MA

15 Wiggins Avenue Bedford, MA 01730

has complied with Florida Administrative Code 64t. I, Part II, for the examination of environmental water in the following entegories:

Metals, Hazardons Waste Characterization, Pesticides/Herbicides/PCB's (GC), Purgeable Organics (GC, GC/MS), Extractable Organics (GC, GC/MS), Nutrients

Specific methods, parameters, and analytes certified are on file at the Burgan of Jaboratories, P. O. Box 210, Jacksonville, Horida 32231

THE HARPARA

шстиг — ДЛУ 1, 1997

THROUGH JUNE 30, 1998

Eldert C. Hartwig, Jr., Sc. Jr., Adv.H.
Bureau Chief, Bureau of aboratorics
Horida Department of Health
DH Form 1697, 3/97
NON TRANSFERABLE

CERTIFICATE No.: 97207

AUGUSTA, MAINE II STATE HOUSE STATION DEPARTMENT OF HUMAN SERVICES STATE OF MAINE

1100-655+0

SOVERNOR HUGUS S. KING, LF.

COMMESICAES KENIN M. CONCANNON certifies that

TOXIKON Corporation

15 Higgins Avenue

Bedford, Massachusetta 01730

Laboratory Certification and is hereby granted 157-A and the rules for Comprehensive Environmental Elimination System and as required by 22 M.R.S.A., Chapter analytes as defined by the Mational Pollution Discharge Has demonstrated the capability to analyze WASTE WATER

and Certified Parameter List. See Massachusetts Certificate FULL CERTIFICATION FOR:

.tail retemers beilitred bas See Massachusetts Certificate PROVISIONAL CERTIFICATION FOR:

Issue Date: 06/02/97 Exprestion Date: 05/04/98

Certification No.: MAG64

DISPLAY IN A PROMINENT LOCATION Michael C. Sodano, Certification Officer, Department of Human Services



ANGUS S. KING, JA

KEVIN W. CONCANNON COMMISSIONER

certifies that

TOXIKON Corporation

15 Wiggins Avenue

Bedford, Massachusetts 01730

Has demonstrated the capability to analyze DRINKING WATER as required by 22 M.R.S.A., Chapter 157-A and the rules for Comprehensive Environmental Laboratory Certification and the

Safe Drinking Water Act and is hereby granted

FULL CERTIFICATION FOR:

See Massachusetts Certificate

and Certified Parameter List.

PROVISIONAL CERTIFICATION FOR:
See Massachusetts Certificate
and Certified Parameter List.

Certification No.: MA064

Issue Date: 06/02/97

Expiration Date: 05/04/98

Michael C. Scdano, Certification Officer, Department of Human Services

DISPLAY IN A PROMINENT LOCATION





STATE OF MARYLAND

DEPARTMENT OF HEALTH AND MENTAL HYGIENE LABORATORIES ADMINISTRATION

15 Wiggins Avenue, Bedford, Massachusetts 01730 TOXIKON CORPORATION Certifies That

And Standards Of Performance In Accordance With Regulations Governing Laboratory Certification The Annotated Code of Maryland, having duly met the requirements of the is hereby approved as a

State Certified Water Quality Laboratory

To perform the analyses indicated on the Annual Certified Parameter List,

Approved Analyses: Trace Metals 1,2; Inorganics 1,2,3; THM; VOCs 1,2

185

July 30, 1997

June 30, 1998

(Not Transferable)

Director, Laporatyfies Administration

This certification is subject to unannounced laboratory inspections

CONSPICUOUSLY DISPLAY IN THE LABORATORY WITH THE ARM OF THE ATABLE FRIE.

DATE HIRED:

06/27/94 [08/15/88 - 08/21/90)]

RESUME DATE:

06/27/95 001

REVISION:

CAROL LEEDOM WET CHEMISTRY SUPERVISOR

M.S. 1988 Water Resource Sciences, University of Michigan, Ann Arbor, MI

B.S. 1980 Environmental Science, Purdue University, West Lafayette, IN

1994 - Present/ 1988 - 1990 Toxikon Corporation, Woburn, Massachusetts

As the Wet Chemistry Supervisor, Ms. Leedom is involved in extraction and preparation of She handles a wide range of inorganic analyses, e.g. UV/Vis samples for inorganic analyses. and FTIR Spectroscopy for GLP protocols under EPA methodologies. responsible for scheduling the tests and oversees the work of the inorganic extractions department staff.

1983 - 1985 University of Michigan, Ann Arbor, Michigan

As a Research Assisting in the Department of Environmental Engineering, Ms. Leedom assisted with gel chromatography, collected, extracted and prepared natural surface water and landfill drainage samples for GC/MS analyses and assisted with rate studies of priority pollutants onto sediments and DOC.

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DATE HIRED: RESUME DATE:

11/16/87 06/27/95

REVISION:

001

DAVID BLACKWELL ORGANIC EXTRACTION SUPERVISOR

B.S. Chemistry, University of Lowell, Lowell, Massachusetts (1986)

1987 - Present

Toxikon Corporation, Woburn, Massachusetts

Since joining Toxikon, Mr. Blackwell has been working in the organics extraction department, primarily running all organic extraction procedures, specifically method 608, 625, 8270, 8080 and Herbicides. His responsibilities include record keeping, equipment calibration and preventive maintenance, documentation of data and special events that may affect data integrity. His work is reviewed by the Laboratory Director. He directs actiities of the Organics Extraction Department.

1987

Cambridge Analytical Associates, Bedford, Massachusetts

Mr. Blackwell spent approximately 6 months at Cambridge Analytical Associates performing organic extracts, data entry, and routine QA/QC.

	; ************************************





Department of Environmental Protection

Division of Environmental Analysis Senator William X. Wall Experiment Station

certifies

MMA064 Toxikon Corporation 15 Wiggins Ave. Bedford, MA 01730

Laboratory Director: Laxman S. Desai

for the Chemical Analysis of Potable and Non-Potable Water

pursuant to 310 CMR 42.00

This certificate supersedes all previous Massachusetts certificates issued to this laboratory. The laboratory is regulated by and shall be responsible for being in compliance with Massachusetts regulations at 310 CMR 42.00.

This certificate is valid only when accompanied by the latest dated Certified Parameter List as issued by the Massachusetts D.E.P.

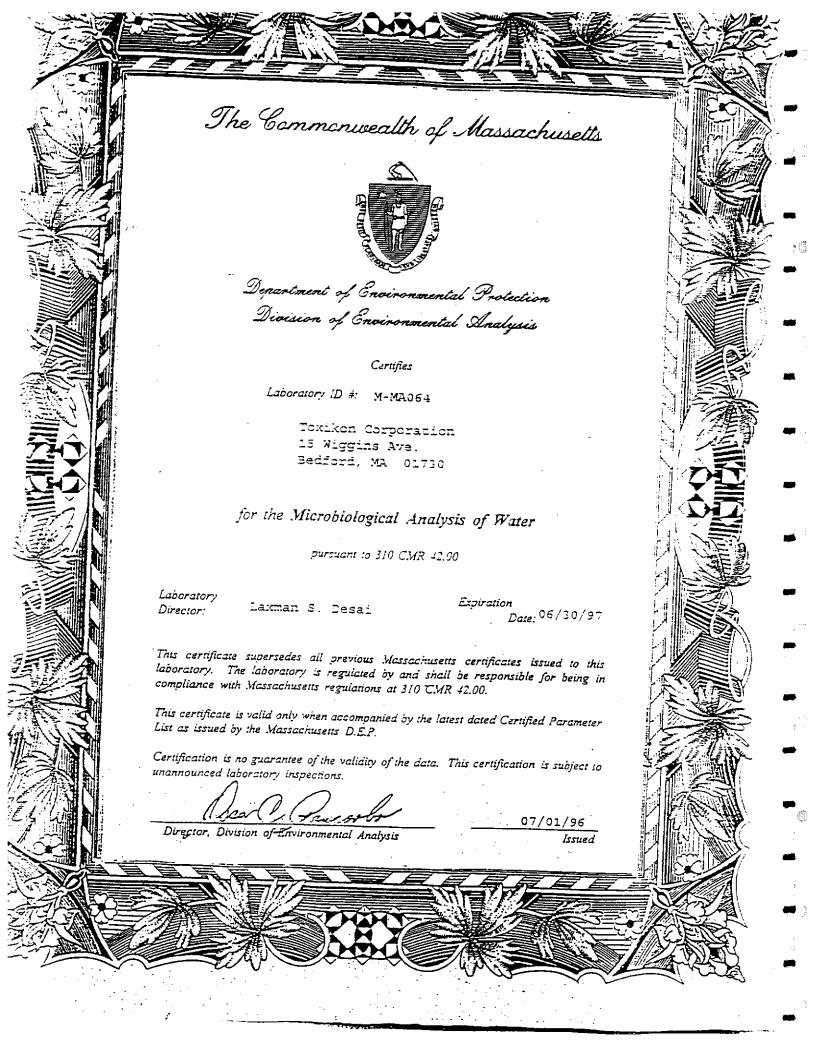
Certification is no guarantee of the validity of the data. This certification is subject to unannounced laboratory inspections.

(1 sear of presents

Issued: 07/01/97

Director, Division of Environmental Analysis

Expires: 06/30/98





CERTIFICATE OF APPROVAL Drinking Water Analysis

Issued to
Toxikon Laboratory

Located at

15 Wiggins Anenue, Bedford, MA

Under the provisions of the Regulations in Env-C300 for the following analyses:

FULL CERTIFICATION: Metals by Graphite Furnace, Metals by ICP, Mercuric Nitrite-N, Fluoride, Orthophosphate, Residual Free Chlorine, Turbidity, Total Filterable Residue, Calcium Hardness, pH, Alkalinity, Sodium, Sulfate Insecticides (Limited List), Trihalomethanes, Volatile Organics, Viny Chloride, DBCP, and EDB.

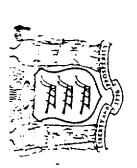
PROVISIONAL CERTIFICATION: Nitrate-N.

CERTIFICATE NUMBER: 204097-A

DATE OF ISSUE: July 24, 1997

EXPIRATION DATE: July 23, 1998

Certifying Officer



ULITAHIIMLNI OF ENVIRONMENTAL PROTECTION

Certifies That
Twiken
225 Wildwood Avenue
Woburn, M. 01801



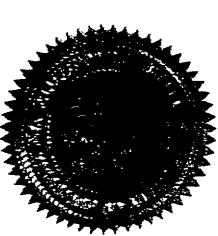
having duly met the requirements of the

Regulations Governing Laboratory Certification And Standards Of Performance NJ.A.C. 7:18 et. seq.

is hereby approved as a

State Certified Water Laboratory

To perform the analyses as indicated on the Annual Certified Parameter List which must accompany this certificate to be valid



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Carried A. Charre

Prodavije skrije armanije June 7. 1990 Darg

N.J.A.C. 7:18-2.11(d) and agreed to by the Laboratory Manager on filing the application This certification is subject to unannounced laboratory inspections as specified by

TO BE CONSPICUOUSLY DISPLAYED AT THE LABORATORY WITH THE ANNUAL CERTIFIED PARAMETER LIST.

State of Fem Fersey

Theistine Toda Whitman. Il eenor Department of Environmental Protection

Rabert C. Shinn Tr. Commissioner

Office of Quality Assurance
9 Ewing Street, 2.0, 20x 414
Trenton, New Tursey 98615
Tel. (609) 633-3840
Fee. (609) 777-1774

TOXIKON CORPORATION IS WIGGINS AVENUE BEDFORD, MA 01730 Lab IO # 59538 Arm: LAXMAN S. DESAL

July 21, 1997

Dear Laboratory Manager:

The Office of Quality Assurance (OQA) will not be issuing a Certificate or an Annual Certified Parameter List at this time. New Certificates will be issued as soon as they become available. Annual Certified Parameter Lists will be generated and forwarded once our Environmental Laboratory Certification Program completes the development of its computedized data base.

Effective with the receipt of this letter your laboratory's certification status is temporarily extended until December 31, 1997 or until new Certificates and Annual Certified Parameter Lists become available, whichever is earlier. To determine your laboratory's current certification status please use the temporary Annual Certified Parameter List issued August 1, 1996, and all written notices of certifications and suspensions received by your laboratory since that time.

Additionally, laboratories that have submitted an administratively complete application to the OQA by July 1, 1997 for certification in categories SHW1 through SHW12 and CLP1 through CLP7, are given temporary approval. Part III of your Fiscal/Certification Year 1997 Renewal Application should be used to determine your approval status. For those laboratories submitting a completed Fiscal/Certification Year 1998 Renewal Application, Part III of that application should be used in place of the 1997 Renewal Application.

As always, we are available to discuss any comments or questions. Please do not hesitate to contact me or one of the laboratory certification officers.

Sincerery,

Joseph F. Aiello, Chief

EPA MA

DERTIFICATION ID 59538

LAB NAME TOXIKON CORPORATION

```
ADDRESS 15 WIGGINS AVENUE
                   CITY BEDFORD
                                                  STATE MA ZIP 01730
                   MANAGER LAXMAN S. DESAI
                  PHONE 517-275-3330
  3. WATER POLLUTION CERTIFICATION: C
  :. MICROSICLOGY(WP):
       74055 F.Cali 74054 F.Strep
                                     74058 Heterotro PL IV. CRGANICS(WP): C
      74056 T.Cali 74057 Enterococci 74059 Pseudomonas
                                                         C 501 Purgeable Halocarbon, P&T/GC/HECO
 II. LIMITED CHEMISTRY(WP): C
                                             aerug.
                                                         C 502 Purgeable Aromatics, P&T/GC/PID
    0 00436 Acid 0 00500 75 0 00660 pp,204 01042 Cu,7
                                                          304 Phenois
    0 30410 Alkal C 70300 TDS 0 00671 oP,? 01045 Fe,T
                                                         A 508 Pesticides & PCB's
    C 508P Organochtorine Pesticides: Aldrin/Dietorin/
                                            01051 25,7
    0 30310 3CD5/20A 30545 SS,V 0 00615 NO2
                                            00927 Mg,T
                                                               DDD/DDE/DDT/Heptachlor/Chlordane
    608P9 PC3's only
    C 00948 CL
               0 00505 TVals 2 00680 Ord,T
                                            01067 Nf, T
                                                         A STO PAHIS
    0,300 C 2nd 2 00095 Cond 2 08681 Grc,0
                                            00937 K, T
                                                         512 Chlorinated Hydrocarbons, Extr.w GC/ECO
   0 00080 Cation 0 00945 S04 0 00605 GnW 0 00955 Si02
                                                         C b24 Purgeables,P&T w GC/MS
    0 00720 CM, T 0 00745 S-- 01105 AL,T
                                          00929 Na,T
                                                       0 525 3/N, Acids, Pesticides, Extr. W. GC/MS
   0 00722 CM,012 0 00740 803
                            31802 As, T
                                          01087 V, T
     00300 00,WIN 0 38260 Sunf 01012 Be,T
                                          01092 Zn,T
   0 00299 00,31 00010 Temp 01022 3, 7
                                         00320 C3CD V. 31CASSAY(WP):
   0 00951 F,70T .0 00076 Turb 01027 04,7
                                          (5 & 20 DAY)
                                                         99004 Acute Toxicity
   0 00900 Hardh 0 32730 Phen 00916 Ca, T
   3 30400 H+,pH 3 30650 P,PG4 A 31032 GF,V!
                                                   71. Raden(WP):
   0 30625 TKN 0 30665 P,P 01034 Cn,T
                                                          32303 Radon
IIII.METALS(WP): 0
     01210 AL,AA/GF 0 01035 Co,10AP 01210 Pd,AA/GF
                                                     C. AIR
   0 01105 At, 10AP | 0 01042 Cd, AA/GF | 01171 Pt, AA/GF
                                                    I. Radon/Radon Progeny
   0 01097 Sb,AA/GF 0 01040 GU,CGAP 4 28201 Rh,AA/GF
                                                         451,Ra,MCharceal CampisterM
    01095 Sb,1GAP 71910 Au,AA/GF 27901 Ru,AA/GF
                                                         452,Ra,MAiona-TrackM
  S 01002 As, AA/GF 01046 Fe, AA/GF 0 01147 Se, AA/GF
                                                         453,Ra,meump_Carcon_Grabm
  $ 31000 As,10AP | 0 01045 Fe,10AP | 0 01145 Se,10AP
                                                         454,Ra,MRPISUM
  0 31007 Ba,AA/GF 0 01051 Pb,AA/GF 01077 Ag,AA/GF
                                                         455,Ra,"Charcoal_Liquid_Scintillation"
  0 01005 Ba, ICAP 0 01049 Pb, ICAP 0 01075 Ag, ICAP
  C 01012 3e,AA/GF C 00937 K,AA
                                 00930 Na.AA
                                                     Continuous Monitoring
  C 00929 Na,1CAP
                                                        Ha THOS 16400
  4 01020 B,:CP
                C 00927 Mg,AA
                                 00956 SiG2, ICAP
                                                        00011 CONT Temperature
  50061 CONT CL2 Residual
  C 01025 Cd, (CAP A 01056 Mm, AA
                                 01057 TL, TCAP
                                                        00091 CONT Conductivity .
  C 00916 Ca,AA
                C 01055 Mm, CAP C 01102 Sm, AA/GF
  C 00915 Ca,:CAP | C 71900 Hg,CV
                                 01152 Ti,AA/GF
 C G1932 CrVI,AA A 01062 Mo,AA/GF 01087 V,AA/GF A 01104 Sn, ICP
   01034 Cr,AA/GF C 01060 Mo,ICAP C 01085 V,ICAP
 C 01030 Cr,ICAP A 01067 NI,AA/GF 01092 Zn,AA/GF
                                                   Legend: C (Certified); A (Applied); S (Suspended)
 C 01037 Co,AA/GF C 01065 Ni,ICAP C 01090 Zm,ICAP
                                                            c (Certified by Capillary Column GC)
```



Expires 12:01 AM April ISSUED April 1, 1997 REVISED November 20, 1337

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

ID No.: 10778

Director: DR. LAXMAN DESAI

Lab Name: TOXIKON CORPORATION

Address : 15 WIGGINS AVENUE

FEDFORD MA 01730

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES/ POTABLE WATER

All approved subcategories and/or analytes are listed below:

Wer Metals I : r, total otal ing, fotal m, fotal

Trinking Water Mon-Hecals : Alfalining Talonum Hariness Chioride Cyanide Color

Orinking Tater Tribalomethana (1111) Microextractables (1111)
Tolatile Arcmatics (1111) Tolatile Balocarbons (1111) Tolatile arcmatics (ALL)

Corrosidiy Titrate (as I)
Eydrogen Ton (pH)
Solids, Total)Issolved

5 rial No.: 101604

Wadsworth Center

Property of the New York State Department of Health. Valid only at the address shown. I ist be conspicuously posted. Valid certificate has a red serial number.

I H-3317 (3/97)



Expires 12:01 AM April ISSUED April 1, 1397 REVISED November 20, 1397

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

Lab ID No.: 10773

Director: DR. LAXMAN DESAI

Lab Name: TOXIKON CORPORATION

Address : 15 WIGGINS AVENUE

BEDFORD MA 01730

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES/SOLID AND HAZARDOUS WASTE

All approved subcategories and/or analytes are listed below:

ictaristic Tasting : Ignitability Reactivity Miscallaneous : Cyanide, Total Tydrogen Ion (pH) Suifide (as 3) Purgaable Arcaatics (ALL)

Accolein and Accylonitalia (ALL) Maloethers (ALL) Metals II (ALL) Polychlorinated Hiphenyls (ALL) Chlorinated Sydrocarbons (111), Metals I (ALL) Polymuclear Area, Sydrocarbon (ALL) Priority Pollutant Phenois (ALL)

Serial No.: 101605

Wadsworth Center

Property of the New York State Department of Health. Valid only at the address shown. Must be conspicuously posted. Valid certificate has a red serial number.

OH-3317 (3/97)

EPA CERTIFICATION ID 59538 LAB NAME TOXIKON CORPORATION ADDRESS 15 WIGGINS AVENUE STATE MA EIP CITY BEDFORD MANAGER LAXMAN 3. DESAI FAX:517-271-1136 PHONE 517-275-3330 LOT/BLCCX JORINKING WATER CERTIFIC: C II(a).Limited Chem(Pb&Cu Rules & 7/30/92 Rules) HICROBICLOGY(OW): 319,E.c(NA+MUG) 001, Alk. "Titr" 006,5-P(P)"Auto Col" 020,NO2,Auto-R 303,7.c(mf) 309,7.c(CNPG) 321, E. = (CNPG) 002,Alk."E-Titr" 007,Si02 021, NO2, Cd-Red 305,7.a(ft) 315,7.a(70-->EC) 323,7.a(Calisure) 008,Temperature 023,802,9109 003,Ca,"Titr" 307, T.c(PA) 317, E.c(EC+MUG) 325,E.c(Calisure) 004,Canductiv." 018.Aspestos"TEM" 033,804,"Cater II. LIMITED CHEMISTRY(DW): C 005.b-P(P)Un/Cal 019,NO2"Spectrophot" 034,CN,"Spect" C 945,Chlorine Residual A 935, F, "Steptrode" 335,3-2(2),1031 936, F, "Distill_Color" 0.953, Handness IV. CRGANICS(DW):A 524.1 VCC's-GC/MS(Packed) 937, F, "Auto_Aliz_F_Blue" | C.951, pH, "Glass Electrode" A 524.2 VCC1s-GC/MS(Capillary) A 501.1, TOTAL THM'S 955, Socium 938, F, "Modified Auto" A 501.2, TOTAL THM'S 525.1 PAH/PEST*GC/MS* 940, F, "Zr_Erio_Cyanide" C 956, Sulfate 502.1, VCC P&T GC(Pack) 531.1 Carbamates(HPLC) 959, Sulfate, "Ion Chrom" 939, F, "Auto_Electrode" A 502.2 VCC P&T GC(Capil) 547 Glypnosate(HPLC) C 952, Total Dissolv Solids 931, F. "!on Chrom" 548 Endothat!(GC/ECD) 502.2T THM's only 2 944, Turbidity 0 932,Nitrate,"Cd_Red" 549 Diquat (HPLC) A 503.1 VCAC,PT/Arcm. 927, Capper 933, Nitrate, "Auto_Hydr" ייסטייקסנמטג פאנ 550 PAH, (HPLC) Lid/Lid A 504 928, Iron 0 934,Nitrate,"Auto_Cd_Red" 550.1 PAH (HPLO)SPE A 505 OrganoHal Pest/PC3 919, Nitrate, "Electrode" 929, Manganese 505P OrganCtPest"EMLT" 551 Disinfection Bioroducts 957, Mithate, "Ion Chrom" 930,25nc 5517 (Tribatomethanes only) 948, Jalan, "Pt_Ca" * 506 Adipat/Phtmal(PID) 925,As,"Ag_CEDT_Care" 507 Wysesest. "SC" 552.1 Halpacetic acid/Dalacom 950,0don,"Const_Series" 947,01, #4g_4g_NC3# 555 Chiominated Acids A 508 Cl-Pest."GC" 949,ASS/LAS,"Methylene 31" 953,01,41an Chrom# 1613 2,3,7,3 TCDD(Dicxin) 508A PC3's-Confimation 946,St."Potentiometric" 508P OrganoCl Pests"EMLT" III. METALS(DW): C # 515.1 Cht.Renbs."GC" A 515.1H Regul.Heros/PCP(2,4-0;Silvex;Dalacon) 910,As."Myd.Gen" 909,25,"AA,CLEL C 966,CU,"ICP" 904, Fe, "AA" 1 714.As. #GF1 0.918,25,497 V. Radiological(DW) 923, Fe, "GF" 2 P60,As.91029 C 912,8g,"MCV" 407 Ra-Tot 414 Co-60 40° Gross albha 913,8g,84CV8 C 967, Fe, "ICP" 901,3a,44A# ±15 Ru-106 402 Gross beta -33 i3 911, Se, "Hyd. Gen 905, Mn, "AA C.E." 915,3a,"SF" 409 U 416 In-63 924,86,4664 403 57-39 0 ₹6',3a,"100" 3 920, Se, "GF1 410 Ca+134 417 K 484 Sh490 907,05,"AA"C.E. 902,Ag,"AA" S 968, Mn, "ICP" 418 Ga+137 418 PU-239 405 Ra-226 C 921,Ag, "GF" 954, Na, "AA" 3 715,33,4379 4 9 3a- 33 412 (-131 40o-Ra-223 906, Zn, "AA" 968,05,"AA,C.E. 903,00,"AA" 925, Zn, "GF" C 969, Zn, "ICP" 917, Ch. #GF# 922, Qu, "GF" V[Radon(DW): 1 763,07,41024 A 970.Na."[CP" .(a),Metals(Pb&Cu Rules & 7/30/92 & Proposed193 Rules) 413 Radon 036,3e,"ICP" 022,5102"109" 024,55 "Hydr." 037,55,"10P/MS" 010,0aH:0PH Legend: "2M/401", as, 8E0 011,00"PitFun 025,85 "GF" C(Certified) A(Applied) S(Suspended) 039.3a,"[CP/MS"

c(Centified by Capillary Column GC)

CERTSUM DATABASE

027,8e "[CP/MS" | G40,Cd,"[CP/MS"

041,CF,"ICP/MS"

042,Mn,"ICP/MS" 043, Ni, "ICP/MS"

044.Se."[CP/MS"

045, Ag, "[CP/MS"

046, Zn, "ICP/MS"

009, CaMAAM

015, A L''GF"

916, ALHAAH -

S17, ALPICEP

019,AUTICP/MS

G13,95MPltFur

014,25"[C2/MS 028,NT "AA"

029,Ni "GF"

031,71 "GF"

030,xi "ICP"

032,TL "ICP/MS"

047, Hg "ICP/MS"



Expires 12:01 AM April ISSUED April 1, 1997 REVISED November 20, 1397

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 302 Public Health Law of New York State

Lab ID No.: 10773

Director: DR. LAKMAN DESAI

Lab Name: TOXIXON CORPORATION Address: 15 WIGGINS AVENUE

BEDECRD MA 01730

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES NON POTABLE WATER

All approved subcategories and/or analytes are listed below:

Facile Salocarbons: Resterd

Facinc Sections

Facinc Sect

Tastawatar Miscailaneous :

Boron, Total

Dramida, Total

Corrosivity

Phanois

Jil & Graasa Total Recoverable

Brancoan Ion (pH)

Spacific Canductanca

Clica, Dissolved

Suifila as 3)

Surfactant (MHS)

Iroanic Carbon, Total

Maiscans:

4-Brancobanyinhenyi ather

Bis 1-chioroachoxy/methana

Bis 1-chioroachoxy/methana

Bis 1-chioroachoxy/methana

Sis 1-chioroachox

Fascavacar Metals II :

Aluminum, Total
Armenic, Total
Bervilium, Total
Thromium TI
Mercury, Total
Linimoury, Total
Linimoury, Total
Linimoury, Total
Jone, Total
The John Total
Total Total
Total Total
Total Total
Total Total
Total Total
Total Indiano Themais (ALL)
Total Idditional Totalpounds (ALL)
Total Idditional Totalpounds (ALL)

Gastavatar detais III :
Gold. Jotai
Johait, Fotal
Johait, Fotal
Johait, Fotal
Jointenna, Fotal
Thailina, Fotal
Futerent :
Gieldani Mitrogen, Total
Associa (as #)
Mitrogen as #)
Mitrogen as #)
Grinophosphate (as #)
Grinop

Serial No.: 101603

Wadsworth Center

Property of the New York State Department of Health. Valid only at the address shown. Just be conspicuously posted. Valid certificate has a red serial number.

:OH-3317 (3/97)

Saje and Healthy Lives in Saje and Healthy Communities

24 June 1996

Mr. Layman DeSai Toxikon Corporation 15 Wiggins Avenue Bedford, Massachusetts 01730

RE: License Number 55

Dear Mr. DeSai:

This is to acknowledge acceptance of the Analytical Laboratory License Renewal Application for the period of July 1, 1996 until June 30, 1998.

In accordance with the provisions of Chapter 23-16.2-A/LAB of the Rhode Island General Laws 1956, as amended, the Rhode Island Department of Health hereby issues this license renewal for the period as specified above, authorizing the operation and maintenance of a Testing Facility known and operated as Toxikon Corporation.

This license covers the premise located at:

15 Wiggins Avenue Bedford, Massachusetts 01730

Sincerely,

Wayne I. Farrington

Chief

Division of Facilities Regulation

(401) 277-4566

Ig Enclosure wp.ltr/anl.lic.renew

South Carolina Department

Environmental Laboratory Certification Program of Health and Environmental Control

In accordance with the provisions of Regulation 61.81, entitled "State Environmental Laboratory Certification Regulation,"

TOXIKON CORPORATION (SSUUL) IS WIGGINS AVENUE BEDFORD, AIA 01730

certificate is the property of S.C. DHEC and must be surrendered upon demand. This certificate is non-transferable and is outly for the generated, but indicates the laboratory's adherence to pressoribed methodology, quality control, records keeping, and reporting procedures. This is hereby certified to perform analyses as documented on the attached parameter list(s). This certification does not guarantee calidity of data parameters and methodology listed on the attached parameter lists(s).

Certifying Authority: NY Date of Issue: 07/08/1997

Date of Expiration: 08/15/1997 Certificate Number: 88002001

(K' (Char Distris)

e Mice of Environmental Laboratory Pertification

4A-1459 (8/96)

STATE OF NORTH CAROLINA DEPARTMENT OF THE ENVIRONMENT, HEALTH & MATURAL RESOURCES

DIVISION OF ENVIRONMENTAL MANAGEMENT LABORATORY CERTIFICATION PROGRAM

In accordance with the provisions of N.C.G.S. 143-215.3 (a) (1), 143-215.3 (a)(10) and NCAC 211.0800;



TOXICON CORPORATION

Is hereby certified to perform mastemater analyses (as listed on inorganics attachment I or organics attachment II) and report monitoring duta to DRM for compliance with NPDES effluent, surface water, broundwater, and pretreatment regulations.

This certificate does not guaruntee validity of data generated, but indicates the methodology, equipment, quality control procedures, records, and proficiency of the laboratory have been examined and found to be acceptable.

This certificate shall be valid until 01.01.011011 31, 1998.

286 Certificate No. 1/2 1 7 THE (

('hief, Inbornabiry Section " Bernard H. Sims, PhD

North Carolina Wastewater/Groundwater Laboratory Cartification Certified Parameters Listing

Lap Name:

Toxikon Corporation

Address:

15 Wiggins Ave. Bedford, MA 01730 Certificate Number:

286

Effective Date:

1/1/96

Expiration Date:

12/31/98

Date of Last Amendment:

7/24/97

The above named laboratory, having duly met the requirement

	CERTIFIED PARAMETERS			
INORGANIC	RESIDUE, SUSPENDED			
3CD	SULFATE			
COD	SULFIDE			
CHLORIDE	SULFITE	#		
CHLORINE, TOTAL RESIDUAL	TCTAL ORGANIC CARBON	•		
CCLOR, PLATINUM COBALT	TURBIDITY			
CONDUCTIVITY	UTCLP	-		
CYANIDE	TCLP METALS			
LUCRIDE	TOLP ORGANICS			
ARCNESS, TOTAL	ORGANIC	4		
/BAS	PURGEABLE HALOCARBONS			

METALS I, REGULAR LEVEL

ALUMINUM

ARSENIC

BERYLLIUM CACMIUM

CHROMIUM, TOTAL

JOBALT

COPPER RCN

_EAD MANGANESE

VICKEL

BELENIUM /ANACIUM

INC ITALS II, REGULAR LEVEL

NTIMONY

ILVER HALLIUM

TALS I, LOW LEVEL RSENIC

EAD ELENIUM

TALS II, LOW LEVEL HALLIUM

RCURY **JONIA NITROGEN**

AL KJELDAHL NITROGEN **→ NO3 NITROGEN**

AL PHOSPHORUS HOPHOSPHATE & GREASE - WATER

!GANIC PHENOLS DUE, SETTLEABLE

L GREASE - EPA 9071

DUE, DISSOLVED 180 C

EPA 601

EPA 5030 + 8010 PURGEABLE ARCMATICS

EPA 602

EPA 5030 + 5020

CRGANCCHLORINE PESTICIDES & PCBs EPA 608

EPA 8080 - 3500 SERIES

POLYNUCLEAR ARCMATICS HYDROCARBONS

EPA 510

EPA 3100 + 3500 SERIES PURGABLE ORGANICS EPA 524

EPA 5030 + 8240 EP4 5030 + 8260

BASE NEUTRALIACID ORGANICS EPA 625

EPA 8270 + 3500 SERIES CRGANCPHOSPHORUS PESTICIDES

EPA 8140 + 3500 SERIES EPA 3141 + 3500 SERIES

TOTAL PETROLEUM HYDROCARBONS (TPH) CALIFORNIA METHOD

GASOLINE RANGE DIESEL RANGE

NONHALOGENATED VOLATILE **ORGANICS**

1.2. DIBROMOETHANE (EDB)

EPA 504

EPA 3015A

DUE, TOTAL

artification requires maintance of an acceptable quality assurance program, use of approved methodology, and satisfactory performance on evaluation samples. Laboratories pject to civil penalties and/or decertification for infractions as set forth in 15A NCAC 2H.0807.

DEPARTMENT OF ENVIRONMENTAL PROTECTION OFFICE OF MANAGEMENT AND TECHNICAL SERVICES

HUREAU OF LABORATORIES



Certifies that

TOXIKON CORPORATION 15 WIGGINS AVENUE BEDFORD MA 01730 having duly met the requirements of Chapter 109, Subchapter H, Safe Drinking Water Rules and Regulations issued under the Pennsylvania Safe Drinking Water Act of May 1, 1984 (P.L. 206, No. 43), (35 P.S. SS 721.1 - 721.17) is hereby approved as a

Certified Drinking Water Laboratory

to perform the following analyses:

Inorganic Trace Metals (Groups 1-3) Organic Total Trihalomethanes, Volatile Organic Chemicals (Groups 1-3)

Expiration Date: August 11, 1998

Certificate not transferable Surrender upon revocation

To Be Conspicuously Displayed at the Laboratory

Floyd Kefford, Bureau Director, Bureau of Laboratories

Trees and and probleme plantalions DEPARTMENT OF HEALTH

 $\Delta u dit = N_{\bar{0}}$



License No., 55

This is to certify that

15 WIggina Avenue Bedford, Манвасћивеtts — 01730 TOXIKON CORPORATION

Analytical Aahmatury

Toxicon Corporation

in conformity with Chapter 39 of Title 23 of the General Laws of Rhode Island, as amended.

It has demonstrated its proficiency in the performance of the following One. categories of laboratory tasts;

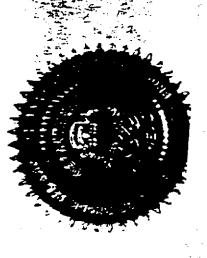
Chemintry

Surface Water

Wastewater

Potable Water

-Patricia a. Molan, 116, 11PM





2600 Buil Street Columbia, SC 29201-1708

July 3, 1997

KIMBERLY N PALMER TOXIKON CORPORATION 15 NIGGINS AVENUE BEDFORD MA 01730

Laboratory I. D. #88002

Dear Kimberly N. Palmer:

Based upon your laboratory's remittance of all outstanding fees and/or updated certification information. I am pleased to enclose your amended certificate and associated parameter list. The parameter list should be compared with your laboratory's original application to determine if any parameters have been added or omitted and that the correct method reference is listed. If problems are detected, please contact this office within ten(10) working days.

Please be informed that these documents now represent the certificate of record for your laboratory. Any certificate(s) and associated parameter list(s) received prior to your receipt of these documents are now null and void and should be destroyed. Please be reminded that all environmental data submitted to the Department is reviewed to ensure that the reporting laboratory possesses the necessary certification. Data reported by laboratories without the proper dertification will be addressed by the affected enforcement programs.

Any questions concerning the laboratory Cartification Program or the action; so taken may be addressed to mel Please accept my congratulations regarding this achievement.

Sincerely,

L'Elagre Dais

R. Wayne Davis, Director Office of Environmental Laboratory Certification Bureau of Environmental Services

RWD:ic

Enclosures

Office of Environmental Laboratory Certification
P. O. Box 72
State Park, South Carolina 29147
FAX # (803) 935-6859
(803) 935-7025

Certifying Authority: NY
Certificate Number: 88002001

CLEAN WATER ACT

Date of Issue: 07/08/1997

Expiration Date: 08/15/1997

SEMI-VOLATILES

POLYNUCLEAR AROMATIC HYDROCAR. EPA 610

VOLATILES (VOCS)

PURGEABLE AROMATICS EPA 602 PURGEABLE HALOCARBONS EPA 601

INORGANIC - TRACE METAL

LEAD EPA 200.7 LEAD EPA 239.2

INORGANIC - MISCELLANEOUS

OIL & GREASE EPA 413.1