NEW YORK STATE | Department of Environmental Conservation

KATHY HOCHUL
Governor

AMANDA LEFTON
Commissioner

Sent via email to:
jonathan.blaine@hbfuller.com

May 5, 2026

Jonathan Blaine
H.B. Fuller Company
1200 Willow Lake Boulevard
St. Paul, Minnesota 55110

Re: Revised Site Management Plan (SMP) - Approval
Former H.B. Fuller- Monarch Div. Prop. Site
City of Geneva, Ontario County, New York
NYSDEC Site Number: V00119

Dear Jonathan Blaine:

The New York State Department of Environmental Conservation (NYSDEC) and New York State Department of Health (NYSDOH), collectively referred to as the "Departments", have reviewed the revised Site Management Plan (SMP), dated April 28, 2026, prepared by H&A of New York Engineering and Geology, LLP (H&A) for the Site listed above.

In accordance with Title 6 of the New York Codes, Rules, and Regulations (NYCRR) Part 375-1.6, the SMP is hereby approved as revised. Please manage all activities at the site in accordance with this approved SMP.

Please contact me at kathryn.lovell@dec.ny.gov or at (585) 438-5280 to discuss any questions or concerns you may have.

Sincerely,

Kathryn Lovell
Project Manager

ec:
Chase Romney (Fortress Investment Group)
William Turner (Fortress Investment Group)
Mark Kleiman (Univar)
Jeff Rowe (Univar)
Santa McKenna (H&A)
Scott Anderson (H&A)
Glenn White (H&A)
Justin Deming (NYSDOH)
Julia Kenney (NYSDOH)
David Pratt (NYSDEC)
Mackenzie Rees (NYSDEC)

FORMER MONARCH CHEMICALS FACILITY

ONTARIO COUNTY

GENEVA, NEW YORK

SITE MANAGEMENT PLAN

NYSDEC Site Number: VCA NO.V00119-8

Prepared for:

H.B. Fuller Company

1200 Willow Lake Boulevard

St. Paul, Minnesota 55164-0683

Prepared by:

H & A of New York Engineering and Geology, LLP

260 E Main St

Suite 2100

Rochester, NY 14604

(585) 359-9000

Revisions to Final Approved Site Management Plan:

Revision No.	Date Submitted	Summary of Revision	NYSDEC Approval Date
1	April, 2016	Initial report	
2	March 31, 2017	Addition of Groundwater Monitoring	
3	July 7, 2017	Revisions to Language, EC and Monitoring	
4	September, 2017	Addition of indoor air sampling, data management	
5	September 10, 2024	Removal of air monitoring and well network reduction	
6	April 28, 2026	Convert to updated NYSDEC template	

APRIL 2026

CERTIFICATION STATEMENT

I SANTA E. MCKENNA certify that I am currently a Qualified Environmental Professional in defined in 6 NYCRR Part 375 and that this Site Management Plan was prepared in accordance with all applicable statutes and regulations and in substantial conformance with the DER Technical Guidance for Site Investigation and Remediation (DER-10) and Green Remediation (DER-31).

SANTA E. MCKENNA, P.G. QEP

4/28/2026 DATE

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FORMER MONARCH CHEMICALS FACILITY
ONTARIO COUNTY
GENEVA, NEW YORK

SITE MANAGEMENT PLAN

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List of Acronyms

µg/kg	Micrograms per Kilogram
µg/L	Micrograms per Liter
11DCA	1,1-Dichloroethane
111TCA	1,1,1-Trichloroethane
AS	Air Sparging
ASP	Analytical Services Protocol
AST	Aboveground Storage Tank
BCA	Brownfield Cleanup Agreement
BCP	Brownfield Cleanup Program
BGS	Below Ground Surface
BMP	Best Management Practice
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CAMP	Community Air Monitoring Plan
cDCE	cis-1,2-Dichloroethene
C/D	Construction and Demolition
CFR	Code of Federal Regulation
CLP	Contract Laboratory Program
COC	Certificate of Completion
COPC	Constituents of Primary Concern
CO2	Carbon Dioxide
CP	Commissioner Policy
DER	Division of Environmental Remediation
DUSR	Data Usability Summary Report
EC	Engineering Control
ECL	Environmental Conservation Law
ERD	Enhanced Reductive Dechlorination
ELAP	Environmental Laboratory Approval Program
ERP	Environmental Restoration Program
EWP	Excavation Work Plan
GHG	Greenhouse Gas
GWE&T	Groundwater Extraction and Treatment
IC	Institutional Control
IRM	Interim Remedial Measures
MEK	Methyl ethyl ketone
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
NYCRR	New York Codes, Rules and Regulations
O&M	Operation and Maintenance
OM&M	Operation, Maintenance and Monitoring
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PCE	Tetrachloroethene
P.E. or PE	Professional Engineer

PFAS	Per- and Polyfluoroalkyl Substances
PID	Photoionization Detector
PRP	Potentially Responsible Party
PRR	Periodic Review Report
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QEP	Qualified Environmental Professional
RAO	Remedial Action Objective
RAWP	Remedial Action Work Plan
RCRA	Resource Conservation and Recovery Act
RI/FS	Remedial Investigation/Feasibility Study
ROD	Record of Decision
RP	Remedial Party
RSO	Remedial System Optimization
SAC	State Assistance Contract
SCG	Standards, Criteria and Guidelines
SCO	Soil Cleanup Objective
SHSP	Site-Specific Health & Safety Plan
SMP	Site Management Plan
SOP	Standard Operating Procedures
SOW	Statement of Work
SPDES	State Pollutant Discharge Elimination System
SSD	Sub-slab Depressurization
SVE	Soil Vapor Extraction
SVI	Soil Vapor Intrusion
TAL	Target Analyte List
TCE	Trichloroethene
TCL	Target Compound List
TCLP	Toxicity Characteristic Leachate Procedure
USEPA	United States Environmental Protection Agency
UST	Underground Storage Tank
VC	Vinyl Chloride
VCA	Voluntary Cleanup Agreement
VCP	Voluntary Cleanup Program
VOC	Volatile Organic Compound

ES EXECUTIVE SUMMARY

The following provides a brief summary of the controls implemented for the Site, as well as the inspections, monitoring, maintenance and reporting activities required by this Site Management Plan:

Site Identification:	Site No. V00119-8, Monarch Chemical Facility, 61 Gates Avenue, Geneva, Ontario County, New York 14456
Institutional Controls:	1. The property may only be used for industrial use;
	2. All ECs must be inspected at a frequency and in a manner defined in the SMP;
	3. The use of groundwater underlying the property is prohibited without necessary water quality treatment as determined by the NYSDOH or the Ontario County Department of Health to render it safe for use as drinking water or for industrial purposes, and the user must first notify and obtain written approval to do so from the Department;
	4. Groundwater and other environmental or public health monitoring must be performed as defined in this SMP;
	5. Data and information pertinent to site management must be reported at the frequency and in a manner as defined in this SMP;
	6. All future activities that will disturb remaining contaminated material must be conducted in accordance with the Excavation Work Plan and this SMP;
	7. Monitoring to assess the performance and effectiveness of the remedy must be performed as defined in this SMP;
	8. Operation, maintenance, monitoring, inspection, and reporting of any mechanical or physical component of the remedy shall be performed as defined in this SMP;
	9. Access to the site must be provided to agents, employees or other representatives of the State of New York with reasonable prior notice to the property owner to assure compliance with the restrictions identified by the Deed Restriction;

Site Identification:	Site No. V00119-8, Monarch Chemical Facility, 61 Gates Avenue, Geneva, Ontario County, New York 14456	
	10. The potential for vapor intrusion must be evaluated for any buildings developed in the area within the IC boundaries, and any potential impacts that are identified must be monitored or mitigated;	
	11. Vegetable gardens and farming on the site are prohibited.	
	12. An evaluation shall be performed to determine the need for further investigation and remediation should large scale redevelopment occur, if any of the existing structures are demolished, or if the subsurface is otherwise made accessible; and	
	13. The Site owner or remedial party will submit to NYSDEC a written statement that certifies, under penalty of perjury, that: (1) land use and groundwater restrictions employed at the Property are unchanged from the previous certification or that any changes to the restrictions were approved by NYSDEC; and (2) nothing has occurred that impairs the ability of the controls to protect public health and environment or that constitute a violation or failure to comply with the SMP. NYSDEC retains the right to access the Property at any time in order to evaluate the continued maintenance of any and all controls. This certification shall be submitted annually, or an alternate period of time that NYSDEC may allow and will be made by an expert that NYSDEC finds acceptable.	
Engineering Controls:	1. Cover system	
Inspections:		Frequency
1. Sitewide Inspection		Annually
2. Screening/segregating soil samples per EWP. Confirmation samples per excavation dimensions.		As-needed; if disturbed >1’ bgs
Monitoring:		
1. Groundwater		Biannually
2. Well and soil probe abandonment		One-time event
3. Soil Vapor Intrusion Evaluation for New Buildings		As needed

Site Identification:	Site No. V00119-8, Monarch Chemical Facility, 61 Gates Avenue, Geneva, Ontario County, New York 14456
Maintenance:	
1. None	
Reporting:	
1. Periodic Review Report	Annually
2. Soil screening for excavations disturbing VOC impacted soils > one (1) ft bgs	Within ninety (90) days after completion of excavation

Further descriptions of the above requirements are provided in detail in the latter sections of this Site Management Plan.

1.0 INTRODUCTION

1.1 General

This Site Management Plan (SMP) is a required element of the remedial program for the Former Monarch Chemicals Facility located in Geneva, New York (hereinafter referred to as the "Site"). See Figure 1. The Site is currently in the New York State (NYS) Voluntary Cleanup Program (VCP), Site No. No. V00119-8, which is administered by New York State Department of Environmental Conservation (NYSDEC or Department).

H.B. Fuller Company (H.B. Fuller) entered into a Voluntary Cleanup Agreement (VCA) on March 4, 2002, with the NYSDEC to remediate the site. A figure showing the site location and boundaries of this site is provided in Figure 2. The boundaries of the site are more fully described in the metes and bounds site description that is part of the Deed Restriction provided in Appendix A.

After completion of the remedial work, some contamination was left at this site, which is hereafter referred to as "remaining contamination". Institutional Controls and Engineering Controls (ICs & ECs) have been incorporated into the site remedy to control exposure to remaining contamination to ensure protection of public health and the environment. An Deed Restriction granted to the NYSDEC, and recorded with the Ontario County Clerk, requires compliance with this SMP and all ECs and ICs placed on the site.

This SMP was prepared to manage remaining contamination at the site until the Deed Restriction is extinguished in accordance with ECL Article 71, Title 36. This plan has been approved by the NYSDEC, and compliance with this plan is required by the grantor of the Deed Restriction and the grantor's successors and assigns. This SMP may only be revised with the approval of the NYSDEC.

It is important to note that:

- This SMP details the site-specific implementation procedures that are required by the Deed Restriction. Failure to properly implement the SMP is a violation of the Deed Restriction, which is grounds for revocation of the Certificate of Completion (COC); and
- Failure to comply with this SMP is also a violation of Environmental Conservation Law, 6 NYCRR Part 375 and the VCA No. V00119-8 for the site, and thereby subject to applicable penalties.

All reports associated with the site can be viewed by contacting the NYSDEC or its successor agency managing environmental issues in New York State. A list of contacts for persons involved with the site is provided in Appendix B of this SMP.

This SMP was prepared by Pinnacle Engineering, Inc (Pinnacle), revised by H & A of New York Engineering and Geology, LLP (Haley & Aldrich), on behalf of H.B. Fuller in accordance with the requirements of the NYSDEC's DER-10 ("Technical Guidance for Site Investigation and Remediation"), dated May 2010 and the guidelines provided by the NYSDEC. This SMP addresses the means for implementing the ICs and/or ECs that are required by the Deed Restriction for the site.

1.2 Revisions and Alterations

Revisions and alterations to this plan will be proposed in writing to the NYSDEC's project manager. The NYSDEC can also make changes to the SMP or request revisions from the remedial party. Revisions will be necessary upon, but not limited to, the following occurring: a change in media monitoring requirements, upgrades to or shutdown of a remedial system, post-remedial removal of contaminated sediment or soil, or other significant change to the site conditions. All approved alterations must conform with Article 145 Section 7209 of the Education Law regarding the application of professional seals and alterations. For example, any changes to as-built drawings must be stamped by a New York State Professional Engineer. In accordance with the Deed Restriction for the

site, the NYSDEC project manager will provide a notice of any approved changes to the SMP, and append these notices to the SMP that is retained in its files.

1.3 Notifications

Notifications will be submitted by the property owner to the NYSDEC, as needed, in accordance with NYSDEC's DER – 10 for the following reasons:

1. 60-day advance notice of any proposed changes in site use that are required under the terms of the VCA No.V00119-8 6 NYCRR Part 375 and/or Environmental Conservation Law.
2. 7-day advance notice of any field activity associated with the remedial program.
3. 15-day advance notice of any proposed ground-intrusive activity pursuant to the Excavation Work Plan. If the ground-intrusive activity qualifies as a change of use as defined in 6 NYCRR Part 375, the above-mentioned 60-day advance notice is also required.
4. Notice within 48 hours of any damage or defect to the foundation, structures or EC that reduces or has the potential to reduce the effectiveness of an EC, and likewise, any action to be taken to mitigate the damage or defect.
5. Notice within 48 hours of any non-routine maintenance activities.
6. Verbal notice by noon of the following day of any emergency, such as a fire; flood; or earthquake that reduces or has the potential to reduce the effectiveness of ECs in place at the site, with written confirmation within 7 days that includes a summary of actions taken, or to be taken, and the potential impact to the environment and the public.
7. Follow-up status reports on actions taken to respond to any emergency event requiring ongoing responsive action submitted to the NYSDEC within 45 days describing and documenting actions taken to restore the effectiveness of the ECs.

Any change in the ownership of the site or the responsibility for implementing this SMP will include the following notifications:

1. At least 60 days prior to the change, the NYSDEC will be notified in writing of the proposed change. This will include a certification that the prospective purchaser/Remedial Party has been provided with a copy of the VCA No. V00119-8, and all approved work plans and reports, including this SMP.

2. Within 15 days after the transfer of all or part of the site, the new owner's name, contact representative, and contact information will be confirmed in writing to the NYSDEC.

Table 1 on the following page includes contact information for the above notifications. The information on this table will be updated as necessary to provide accurate contact information. A full listing of site-related contact information is provided in Appendix B.

Table 1: Notifications*

Name	**Contact Information**	**Required Notification****
NYSDEC's Project Manager Kathryn Lovell, GIT	585-438-5280 kathryn.lovell@dec.ny.gov	All Notifications
NYSDEC Project Manager's Supervisor NYSDEC DER Regional HW Engineer David Pratt, P.E.	585-226-5449 david.pratt@dec.ny.gov	All Notifications
NYSDEC Site Control Kelly A. Lewandowski, P.E.	518-402-9547 kelly.lewandowski@dec.ny.gov	Notifications 1 and 8
NYSDOH Project Manager Julia Kenney, P.G.	518-402-7873 julia.kenney@health.ny.gov	Notifications 4, 6, and 7

* Note: Notifications are subject to change and will be updated as necessary.

** Note: Numbers in this column reference the numbered bullets in the notification list in this section.

2.0 SUMMARY OF PREVIOUS INVESTIGATIONS AND REMEDIAL ACTIONS

2.1 Site Location and Description

The site is located in City of Geneva, Ontario County, New York and is identified as Section 90.15 Block 4 and Lot 68.100 on the Ontario County Tax Map. The site is an approximately 1.713-acre area and is bounded by a commercial manufacturer to the north; vacant manufacturing facilities and residential developments to the south and west; railroad tracks and easements of the Finger Lakes Railroad, an intermittent stream contained in a drainage ditch adjacent to vacant lots and residences to the east. (see Figure 3 – Property Map). The owner(s) of the site parcel(s) at the time of issuance of this SMP is/are:

Fortress Investment Group LLC

The tenant/operator(s) of the site parcel(s) at the time of issuance of this SMP is/are:

Univar Solutions

2.2 Physical Setting

2.2.1 Land Use

The Site consists of the following: a building, loading and unloading areas, parking lots, and an above ground storage tank (AST) containment area. The Site is zoned industrial and is currently a chemical manufacturing plant.

The properties adjoining the Site and in the neighborhood surrounding the Site primarily include commercial, industrial, and residential properties. The properties immediately south and west of the Site include vacant manufacturing facilities and residential development properties; the properties immediately north of the Site include commercial manufacturer property; the properties immediately east of the Site include vacant lots and

residential properties with an active railroad line and accompanying easement and intermittent stream.

2.2.2 Geology

The on-Site surficial geology indicates lacustrine silt and clay predominate the shallow soils. This generally consists of laminated clay and silt deposited in pro-glacial lakes, extending in thickness up to 155 feet (Muller et al, 1991). Borings completed on Site show the near surface sediments range between 0 and 4 feet below ground surface (bgs), consisting of a mixed fill (gravel to silt) material which extends across the Site. This fill layer is underlain by native silt and clays ranging in thickness from 0 to 8 feet thick. A sand and sandy loam underlies this unit and ranges from 2 to 10 feet thick. Each of these units is variable in composition, and interbedded laterally with other discontinuous layers, common of a deltaic or fluvio-lacustrine depositional environment. Beneath these granular sediments is a clay and silty clay unit encountered between 9 and 14 feet bgs. This lower confining unit is not continuous and was not encountered in the east-central portion of the Site, or at off-site locations. Beneath the glacial sediments, a crystalline gray amorphous limestone and chert was encountered at approximately 20 feet bgs off Site and east of the drainage ditch. On Site, bedrock is encountered more than 24 feet bgs. These Site investigative programs have advanced more than 72 soil borings and monitoring wells on the 1.83-acre property and adjacent parcels. An east-west cross section of the surficial geology transecting north of the BCS plant is provided in Figure 4.

The bedrock formation encountered beneath the glacial sediments on Site is the Onondaga limestone (Richard et al., 1970). The Onondaga limestone is lower than the middle Devonian in age, and consists of the Seneca, Morehouse (cherty) and Nedrow limestone members, and the Edgecliff cherty limestone member. Because of its resistance, the Onondaga limestone often forms an escarpment. The outcrop of the Onondaga limestone can be traced in a general east-west direction from just east of Detroit, Michigan and through Ontario and eastward to the Hudson River. Throughout this trend, it varies in thickness from 100 to 500 feet and represents a broad, carbonate platform facies deposited

during early to middle Eifelian time, with the carbonates characterized by calcarenitic to cherty to argillaceous limestones and minor shales deposited in a shallow epicontinental sea (Brett et al., 1994). Site specific boring logs are provided in Appendix H.

2.2.3 Hydrogeology

These shallow hydrogeological conditions are a function of the properties of the upper geologic formations - the surficial glacial sediments and the underlying limestone bedrock. The water table is encountered in unconsolidated surficial sediments in the intergranular pore spaces. The upper section of these surficial sediments are seasonally unsaturated, and the water table is unconfined and generally encountered in the glacial sediments. Shallow groundwater at the Site forms a recharge-to-discharge flow system where the water table mimics the surface topography, and upland areas serve as recharge (infiltration) areas, and streams, ditches, springs, and lakes often serve as discharge locations.

Bedrock forms the second water-bearing unit and can be under unconfined or confined conditions. In this limestone, groundwater is generally located in fractures and bedding planes, and to a lesser degree within the pore spaces of the limestone. On Site, at all locations where drilling into limestone, fracture densities were low enough that the bedrock was dry, and no water-bearing features or saturated bedding planes were encountered.

The primary hydrogeologic units of interest are shallow Quaternary-age surficial glacial sediments. Groundwater is found in the shallow unconfined sands and sandy clay units below the Site. The depth to groundwater generally follows the surface topography, typical of shallow unconfined glacial sediments in this region. The area of greatest depth to groundwater is on the eastern portion of the property near the drainage ditch where the hydraulic gradient steepens near the drainage channel. Groundwater flow is somewhat radially outward from the Basic Chemical Solutions (BCS) plant, with a general easterly to northeasterly and southeasterly flow across the Site. As part of Site investigations, over

72 borings and 56 wells or piezometers were installed to evaluate and monitor groundwater conditions. At the completion of the Remedial Investigation (RI), 10 groundwater monitoring wells remain, including two sets of nested wells.

The depth to groundwater varies across the Site, particularly on the eastern edge of the property near the drainage ditch. On the western portion of the Site at MW-117, seasonal groundwater levels can be as shallow as 2.6 feet bgs and seasonally drop to as low as 8.8 feet bgs, and to the north and east at MW-115 in sands at 7 feet bgs to more than 12 feet bgs in the silty clays, to MW-114 where water levels are generally restricted to sands and generally range 7.5 to 13 feet bgs. In the central portion of the Site at MW-118, water levels are generally contained in the fine sands and seasonally range from less than 5 feet to more than 9 feet bgs, and at MW-119 the water levels are generally encountered in brown silt and clays with seasonal water tables ranging from 4 feet bgs to approximately 8 feet bgs. To the southeast at MW-120, the water levels generally are encountered 9 to 13 feet bgs and are located in silt and clays, and at MW-121 the water levels are relatively consistent at 12 to 13 feet bgs and located in interbedded silty, clayey sands. Along the railroad tracks in the central portion of the Site at MW-122, the water levels range from 8 to 10.5 feet bgs and are located in fine sands. At MW-124s, the water levels seasonally range from 2.5 to 7.5 feet bgs, located in clays and fine sands, with the deeper well MW-124d the water levels ranging from 13 to 19 feet bgs in fine grained sandy clays.

A groundwater contour map is shown in Figure 5. Groundwater elevation data is provided in Table 2. Groundwater monitoring well construction logs are provided in Appendix H.

2.3 Investigation and Remedial History

The following narrative provides a remedial history timeline and a brief summary of the available project records to document key investigative and remedial milestones for the Site. Full titles for each of the reports referenced below are provided in Section 8.0 – References.

The Site is listed under the Classification Code A - in the New York State Voluntary Cleanup Program. Classification Code A is the classification assigned to a non-registry site in any remedial program where work is underway, but not yet complete.

The first recorded building on the property was a portion of the existing manufacturing plant, constructed in 1900. The earliest manufacturing records indicate Vance Boiler Works operated on Site from 1903 through 1912 (*Geneva Advertiser-Gazette*, February 13, 1913), with the south portion of the building occupied by the boiler shop, and the northern portion of the building a warehouse, with adjacent properties occupied by cutlery manufacturing, can manufacturing, and food storage facilities (ESC - May 1996). In 1915, Geneva Boiler Works (Geneva Times, November 26, 1934) occupied the property up through 1940, with similar operations to the earlier Vance Boiler Works. Burnham Boiler succeeded Geneva Boiler Works between 1941 and 1945. Following Burnham Boiler and up through December 1951, William C. Leggott operated a wholesale beer business on Site. On October 5, 1951, the McLean Precision Products Co., Inc., purchased the William C. Leggott warehouse at 61 Gates Avenue, with plans for defense and aerospace manufacturing beginning in January 1952. McLean Precision Products assumed production of the defense-related work that Premier Mill Corporation was conducting on Site. In January 1952, the portion of the plant operated by Premier Mill Corporation modified processes to manufacture colloid mills and mixers. In January 1962, Magnetic Components, Inc. purchased the property and began circuit board manufacturing operations and impregnated capacitors and associated board components in the northern portion of the plant. Magnetic Components, Inc. became part of Nytronics Inc., which by May 1972 was manufacturing vertical output and small-and-large power transformers for the entertainment and computer markets, employing more than 130 workers at the plant (The Geneva Times, May 3, 1972). In mid-December 1973, Nytronics, Inc. closed operations on the property. Between 1974 and 1977 the Site was occupied by the KILT Company.

In 1979, Monarch Chemical Company (Monarch), a former operating division of H.B. Fuller, bought Antical Chemical, which produced dairy-related cleaning products and

supplies on Site. Monarch continued utilizing the Site to manufacture similar food-grade cleaners and sanitizers used in the dairy and related industries. Chlorinated solvents and degreasers were not utilized by Monarch in the manufacturing of food-grade cleaners and sanitizers for dairy and related industries.

In 1996, H.B. Fuller sold the property and business to HoltraChem Distribution Inc. (HoltraChem). HoltraChem continued to engage in similar production activities employed by Monarch. In the late 1990s, HoltraChem was acquired by Basic Chemical Solutions, LLC (BCS). BCS is a global distributor of commodity chemicals, with a primary focus on inorganic mineral acids and bases such as caustic soda, bleach, and sulfuric and hydrochloric acid. On December 20, 2010, Univar Solutions (Univar) completed the acquisition of BCS. Fortress Investment Group LLC acquired the Site from Univar in December 2024. Univar continues to operate at the Site as a tenant/operator.

Currently, Univar mixes, and packages powdered and liquid cleaners and sanitizers on Site. The components of this operation include acids, caustics, sodium hypochlorite, and surfactants. Bulk liquids are delivered by tanker truck, connected to piping located outside the east portion of the plant within a concrete secondary containment area. Additional raw materials are delivered via truck in drums and totes and unloaded at a dock along the southeast corner of the manufacturing building. The mixing tanks are connected via piping to the bulk loading area along the northeast portion of the manufacturing plant.

Several subsurface investigations have been conducted at the Site to evaluate soil and groundwater quality. Previous subsurface investigations performed by others at the Site include:

- *Phase I Environmental Assessment of Monarch Facility* (Environmental Strategies Corporation [ESC] - May 1996);
- *Phase II Site Assessment, Monarch Chemicals Division* (Legette, Brashears & Graham [LBG] - July 1996);
- *Remedial Investigation Report, Former Monarch Chemicals Division* (Delta Environmental Consultants [Delta]- December 1998);

- *Compilation of Site Data/Recommendations for No Further Action, Former Monarch Chemicals Division* (Delta - August 2000);
- *Passive Soil Gas Survey Findings and Recommendations, Former Monarch Chemicals Division* (Delta - September 2003);
- *Limited Site Investigation Report* (Delta - November 2005);
- *Supplemental Remedial Investigation, Former Monarch Chemicals Facility* (Delta - March 17, 2006); and,
- *Baseline Soil Vapor Intrusion Report* (Delta - June 12, 2007).

Historical investigations have established the presence of petroleum hydrocarbons and chlorinated VOCs on-Site, constituting the following Site-related Constituents of Primary Concern (COPCs):

- Tetrachloroethene (PCE)
- Acetone
- Carbon disulfide
- Trichloroethene (TCE)
- Methylene chloride (MEC)
- Chlorobenzene
- 1,1-Dichloroethene (11 DCE)
- Chloroform
- Chloroethane
- cis-1,2-Dichloroethene (cDCE)
- Methyl ethyl ketone (MEK)
- Cyclohexane
- 1,1,2-Trichloroethane
- Toluene
- Chloromethane
- 1,1-Dichloroethane (11DCA)
- Methyl acetate
- Isopropylbenzene
- 1,1,1-Trichloroethane (111TCA)
- Trans-1,2-dichloroethene
- Benzene
- Methyl isobutyl ketone (MIBK)
- Ethylbenzene
- Xylenes (BTEX)
- 1,2-Dichloroethane (12 DCA)
- Vinyl chloride (VC)
- 2-Hexanone

2.3.1 Soil

Several investigations were completed between 1996 and 2016. The 1996 investigations indicated the primary VOC impacts were located at GP-02 in the AST Containment Area (where MW-118 would be later installed), and west (upgradient) at MW-117. The highest PCE concentration in this area, the only VOC identified above NYSDEC Unrestricted Soil Use standards, was detected below the water table at 14 feet bgs at GP-02, which was installed in the AST Containment Area. Subsequent soil investigations completed in October 2005 north of the BCS plant collected soil samples that did not exhibit VOC concentrations in soil exceeding Soil Cleanup Objectives. North of the BCS plant in the MW-117 area, VOCs were detected in soil in October 2005. This data did not exhibit VOC or semi-volatile organic compound concentrations in soil exceeding Soil Cleanup Objectives

A shallow excavation in the fall of 2016 related to the BCS construction\replacement of the truck scale and canopy in the northeast corner of the plant between former monitoring wells MW-119 and MW-120, unearthed a pipe emanating beneath the AST area, deadheading several feet outside and below the containment wall. Excavation management of materials was completed by ARCADIS on behalf of Univar through an *Interim Site Management Plan* approved by NYSDEC in August 2016. These data are contained in the *Final Construction Report* (ARCADIS, January 2017).

2.3.2 Groundwater

The baseline groundwater VOC concentrations used to develop the corrective action were established using groundwater data collected in October 2005, prior to implementation of the Interim Remedial Measures (IRM). The historic groundwater flow indicated a radial flow pattern in the upgradient portion of the Site, with groundwater flow from the MW-117 and MW-118 Source Areas migrating eastward toward the drainage ditch east of the property.

PCE in groundwater originates in a source area at MW-118 with moderate concentrations extending downgradient to MW-116 and the drainage ditch. Similarly, TCE in groundwater has shown a similar footprint and migration pathway, as does the final primary solvent, 111TCA in nature and extent, and in downgradient magnitude. Groundwater impacts detected east of the drainage ditch appeared to be related to a sampling event cross contamination issue, and the accepted area of groundwater impacted with VOCs on Site. Throughout the history of the study of this release, the VOC concentrations in groundwater have decreased over time, with the general exception of breakdown products during enhanced reductive dechlorination (ERD), including cDCE, 11DCA, and VC. Over time, ERD reduced primary chlorinated solvents PCE, TCE, and 111TCA as their footprint and concentration trends decreased. Following implementation of the IRM, the primary chlorinated VOCs continued to decrease in concentrations to below, or near, NYSDEC Groundwater Cleanup Standards.

2.3.3 Surface Water

Surface water sampling has been completed in the drainage ditch adjacent to the Site. Monitoring surface water\groundwater interactions indicate vertical gradients result in groundwater seepage (gaining stream) along the streambank into the drainage ditch throughout most of the stream reach during all but dry summer months (July through September), after which time the stream becomes a losing stream. During this time, if water is flowing through the channel, discharge occurs from surface water to groundwater.

Monitoring locations included SW-1, located upstream of the property, SW-2 located directly downgradient of MW-118, and SW-3 located on the downstream side of the property. More than a dozen surface water sampling events indicated two occasions where VC exceeding 2 micrograms per liter (µg/L) were detected in the drainage ditch. Both sampling events occurred after the implementation of the IRM at SW-2 in the channel segment directly downgradient from the MW-118 Source Area. Other surface water sampling events detected VOCs in low µg/L range.

2.3.4 Sediment

Sediment sampling was completed in July 2005 in the drainage ditches at SED-1, SED-2, and SED-3, which were co-located with SW-1, SW-2, and SW-3, respectively. Sediment samples were collected in the channel from 0 to 0.5 feet bgs. The highest concentration of total VOCs (51 µg/kg) were detected at SED-3 in July 2005, with acetone detected at 31 B µg/kg, carbon disulfide detected at 3 J µg/kg, methylene chloride detected at 10 J µg/kg, 11DCE detected at 6 J µg/kg, MEK detected at 11 J µg/kg, TCE detected at 5 J µg/kg, PCE detected at 4 J µg/kg, cDCE detected at 10 J µg/kg, and dichlorodifluoromethane detected at 2 J µg/kg. These analytes detected in the sediments are similar in nature to groundwater and surface water compounds, and it is expected these impacts are attributable to Site and upstream operations which contribute sediment, groundwater, and surface water discharge to the drainage ditch. However, none of the VOCs detected exceeded NYSDEC sediment screening values

2.3.5 Soil Vapor

In March 2007, in accordance with NYSDEC criteria, a soil vapor intrusion (SVI) monitoring program was initiated with sampling four indoor air (IA) locations in the BCS plant, co-located with four soil vapor sub-slab (SS) points, and one outdoor air (OA) sample collected upwind of the plant. The results of the initial SVI sampling round indicated VOCs detected in the indoor air samples that were not detected below the manufacturing plant floor (SS). TCE and PCE were detected in both the sub-slab soil vapor samples and in indoor air, but below the 2017 New York State Department of Health (NYSDOH) Indoor Air Guidelines of 2 micrograms per cubic meter (µg/m^3) and 30 µg/m^3, respectively.

Based on the historical trend for sub-slab vapor, on September 10, 2024, NYSDEC approved discontinuing the sub-slab soil vapor and indoor air monitoring program from the SMP. The program would only be reinstated if any alterations to the on-Site building foundation or new building construction occur.

2.4 Remedial Action Objectives

The Remedial Action Objectives (RAOs) for the Site as listed in the Decision Document dated-July, 16, 2016 are as follows:

2.4.1 Groundwater

RAOs for Public Health Protection

- Prevent ingestion of groundwater with contaminant levels exceeding drinking water standards.
- Prevent contact with, or inhalation of, volatiles from contaminated groundwater.

RAOs for Environmental Protection

- Restore ground water aquifer to pre-disposal/pre-release conditions, to the extent practicable.
- Prevent the discharge of contaminants to surface water.
- Remove the source of ground or surface water contamination.

2.4.2 Soil

RAOs for Public Health Protection

- Prevent ingestion/direct contact with contaminated soil.
- Prevent inhalation of or exposure from contaminants volatilizing from contaminants in soil.

RAOs for Environmental Protection

- Prevent migration of contaminants that would result in groundwater or surface water contamination.

- Prevent impacts to biota from ingestion/direct contact with soil causing toxicity or impacts from bioaccumulation through the terrestrial food chain.

2.5 Remaining Contamination

2.5.1 Soil

One soil sample from the Site investigation contained one VOC that exceeded the Unrestricted Use SCOs, the sample was collected from soil boring GP-02 (see Figure 6 below). The soil sample from GP-02 was collected from 14 feet bgs, which is approximately 8 feet below the groundwater table, and it contained PCE at a concentration of 6,700 µg/kg. Soil boring GP-02 is located within the concrete AST Containment Area. This area is known as the MW-118 Source Area and was the subject of the IRM. There were no off-Site locations detecting VOCs in soil exceeding Unrestricted Use SCOs.

Based on the follow-up groundwater sampling that has been conducted over nearly a decade, it has been concluded that the remediation conducted in the MW-118 Source Area successfully reduced groundwater impacts.

Figure 6: Remaining Soil Sample Exceedance

Table 3 summarizes the VOC detections in the sample from GP-02 that exceed the Unrestricted Use SCO for PCE.

2.5.2 Sediment

No VOCs were detected in off-Site sediments exceeding NYSDEC screening values.

2.5.3 Groundwater

Groundwater monitoring wells in the current monitoring network have been sampled routinely since 2005. Over this time period, the average depth to groundwater is 8.2 feet bgs. VOC concentration trends continue to decrease, and the groundwater monitoring program will continue per Section 4 of this SMP.

Table 4 and Figure 7 summarize the results of all samples of groundwater that exceed the SCGs after completion of the remedial action.

2.5.4 Surface Water

There are no remaining surface water monitoring locations detecting VOCs in excess of 6 NYCRR Part 375 Soil Cleanup Objectives.

2.5.5 Soil Vapor

The SVI evaluation program was initiated in March 2007. Each of five sub-slab SVI monitoring events were accompanied by pre-sampling building inspection and chemical and storage inventories at these established and agreed-upon historic sampling locations.

On September 10, 2024, NYSDEC approved eliminating the sub-slab soil vapor and indoor air monitoring program from the SMP. The program would only be reinstated if any alterations to the on-Site building foundation or new building construction occur.

3.0 INSTITUTIONAL AND ENGINEERING CONTROL PLAN

3.1 General

Since remaining contamination exists at the site, Institutional Controls (ICs) and Engineering Controls (ECs) are required to protect human health and the environment. This IC/EC Plan describes the procedures for the implementation and management of all IC/ECs at the site. The IC/EC Plan is one component of the SMP and is subject to revision by the NYSDEC project manager.

This plan provides:

- A description of all IC/ECs on the site;
- The basic implementation and intended role of each IC/EC;
- A description of the key components of the ICs set forth in the Deed Restriction;
- A description of the controls to be evaluated during each required inspection and periodic review;
- A description of plans and procedures to be followed for implementation of IC/ECs, such as the implementation of the Excavation Work Plan (EWP) (as provided in Appendix C for the proper handling of remaining contamination that may be disturbed during maintenance or redevelopment work on the site; and
- Any other provisions necessary to identify or establish methods for implementing the IC/ECs required by the site remedy, as determined by the NYSDEC project manager.

3.2 Institutional Controls

A series of ICs is required by the Decision Document (NYSDEC, July 2016) to: (1) implement, maintain and monitor Engineering Control systems; (2) prevent future exposure to remaining contamination; and, (3) limit the use and development of the site to industrial and commercial uses only. Adherence to these ICs on the site is required by the

Deed Restriction and will be implemented under this SMP. ICs identified in the Deed Restriction may not be discontinued without an amendment to or extinguishment of the Deed restriction. The IC boundaries are shown on Figure 1. These ICs are:

- The property may be used for: industrial use;
- All ECs must be operated and maintained as specified in this SMP;
- All ECs must be inspected at a frequency and in a manner defined in the SMP;
- The use of groundwater underlying the property is prohibited without necessary water quality treatment as determined by the NYSDOH or the Ontario County Department of Health to render it safe for use as drinking water or for industrial purposes, and the user must first notify and obtain written approval to do so from the Department;
- Groundwater and other environmental or public health monitoring must be performed as defined in this SMP;
- Data and information pertinent to site management must be reported at the frequency and in a manner as defined in this SMP;
- All future activities that will disturb remaining contaminated material must be conducted in accordance with this SMP;
- Monitoring to assess the performance and effectiveness of the remedy must be performed as defined in this SMP;
- Operation, maintenance, monitoring, inspection, and reporting of any mechanical or physical component of the remedy shall be performed as defined in this SMP;
- Access to the site must be provided to agents, employees or other representatives of the State of New York with reasonable prior notice to the property owner to assure compliance with the restrictions identified by the Deed Restriction;
- The potential for vapor intrusion must be evaluated for any buildings developed in the area within the IC boundaries noted on Figure 1, and any potential impacts that are identified must be monitored or mitigated;
- Vegetable gardens and farming on the site are prohibited;
- An evaluation shall be performed to determine the need for further investigation and remediation should large scale redevelopment occur, if any of the existing structures are demolished, or if the subsurface is otherwise made accessible.

3.2.1 Excavation Work Plan

The existing cover system will be managed to restrict excavation below the existing cover system, including pavement and buildings. Excavated soil will be tested, properly handled to protect the health and safety of workers and the nearby community, and will be properly managed consistently with state regulations. Imported fill material brought on Site will meet the requirements for the identified site use as set forth in 6NYCRR Part 375-6.7(d). The EWP is provided in Appendix C.

The Site has been remediated for restricted industrial/commercial use. Future ground-intrusive work that will potentially encounter or disturb remaining impacts in soil will be performed in compliance with the EWP (Appendix C). This generally includes soil encountered at or below 1-foot bgs. Any work conducted pursuant to the EWP must also be conducted in accordance with the procedures defined in the Site-Specific Health & Safety Plan (SHSP) and Community Air Monitoring Plan (CAMP) prepared for the Site. A sample SHSP is referenced in Appendix D of this SMP. Based on future changes to state and federal health and safety requirements, and specific methods employed by future contractors, the SHSP and CAMP may be updated and re-submitted with the notification provided in Section B-1 of the EWP. Any intrusive construction work will be performed in compliance with the EWP, SHSP, and CAMP, and will be included in the periodic inspection and certification reports submitted under the Site Management Reporting Plan (Section 7).

The Site owner and associated parties preparing remedial documents submitted to the state, and parties performing this work, are completely responsible for the safe performance of all intrusive work, the structural integrity of excavations, proper disposal of excavated soil and excavation fluids, control of run-off from and into open excavations, and for structures that may be affected by excavations (such as building foundations, parking lots, railroad infrastructure). The Site owner/operator (Appendix G) will ensure that Site development activities will not interfere with, or otherwise impair or compromise, the IECs described in this SMP.

3.2.2 Groundwater Use

The use of groundwater underlying the property is prohibited without necessary water quality treatment as determined by NYSDOH or Ontario Department of Health to render it safe for use as drinking water or for industrial purposes, and the user must first notify and obtain written approval to do so from the Department.

3.2.3 Soil Vapor Use Intrusion

On September 10, 2024, NYSDEC approved eliminating the sub-slab soil vapor and indoor air monitoring program from the SMP. The program would only be reinstated if any alterations to the on-Site building foundation or new building construction occur. If there are any alterations or new construction, the NYSDEC and NYSDOH project managers will be notified, and it may be necessary to reinstate the soil vapor intrusion program at the Site.

3.3 Engineering Controls

3.3.1 Cover

Exposure to remaining contamination at the site is prevented by a cover system. This cover system is comprised of a minimum of 12 inches of a combination of gravel fill and native soils, asphalt pavement, concrete-covered sidewalks, and concrete building slabs. Figure 8 presents the location of the cover system The Excavation Work Plan (EWP) provided in Appendix C outlines the procedures required to be implemented in the event the cover system is breached, penetrated or temporarily removed. Procedures for the inspection of this cover are provided in the Monitoring and Sampling Plan included in Section 4.0 of this SMP. Any work conducted pursuant to the EWP must also be conducted in accordance with the procedures defined in a Site-Specific Health and Safety Plan (SHSP) and associated Community Air Monitoring Plan (CAMP) prepared for the site and provided in Appendix D and Appendix E. Any breach of the site's cover system must be

overseen by a Professional Engineer (PE) who is licensed and registered in New York State or a qualified person who directly reports to a PE who is licensed and registered in New York State.

3.3.2 Criteria for Completion of Remediation/Termination of Remedial Systems

Generally, remedial processes are considered completed when monitoring indicates that the remedy has achieved the remedial action objectives identified by the decision document. The framework for determining when remedial processes are complete is provided in Section 6.4 of NYSDEC DER-10. Unless waived by the NYSDEC, confirmation samples of applicable environmental media are required before terminating any remedial actions at the site. Confirmation samples require Category B deliverables and a Data Usability Summary Report (DUSR).

As discussed below, the NYSDEC may approve termination of a groundwater monitoring program. When a remedial party receives this approval, the remedial party will decommission all site-related monitoring, injection and recovery wells as per the NYSDEC CP-43 policy.

The remedial party will also conduct any needed site restoration activities, such as asphalt patching and decommissioning treatment system equipment. In addition, the remedial party will conduct any necessary restoration of vegetation coverage, trees and wetlands, and will comply with NYSDEC and United States Army Corps of Engineers regulations and guidance. Also, the remedial party will ensure that no ongoing erosion is occurring on the site.

3.3.2.1 - Cover (or Cap)

The composite cover system is a permanent control, and the quality and integrity of this system will be inspected at defined, regular intervals in accordance with this SMP in perpetuity.

3.3.2.2 - Monitoring Wells Associated with Monitored Natural Attenuation

Groundwater monitoring activities to assess natural attenuation will continue, as determined by the NYSDEC project manager in consultation with NYSDOH project manager, until residual groundwater concentrations are found to be consistently below ambient water quality standards, the site SCGs, or have become asymptotic at an acceptable level over an extended period. In the event that monitoring data indicates that monitoring for natural attenuation may no longer be required, a proposal to discontinue the monitoring will be submitted by the remedial party. Monitoring will continue until permission to discontinue is granted in writing by the NYSDEC project manager. If groundwater contaminant levels become asymptotic at a level that is not acceptable to the NYSDEC, additional source removal, treatment and/or control measures will be evaluated.

4.0 MONITORING AND SAMPLING PLAN

4.1 General

This Monitoring and Sampling Plan describes the measures for evaluating the overall performance and effectiveness of the remedy. This Monitoring and Sampling Plan may only be revised with the approval of the NYSDEC project manager. Details regarding the sampling procedures, data quality usability objectives, analytical methods, etc. for all samples collected as part of site management for the site are the Sample and Analysis Plan provided in Appendix F.

This Monitoring and Sampling Plan describes the methods to be used for:

- Sampling and analysis of all appropriate media (i.e., groundwater);
- Assessing compliance with applicable NYSDEC standards, criteria and guidance (SCGs), particularly groundwater standards and Part 375 SCOs for soil; and
- Evaluating site information periodically to confirm that the remedy continues to be effective in protecting public health and the environment.

To adequately address these issues, this Monitoring and Sampling Plan provides information on:

- Sampling locations, protocol and frequency;
- Analytical sampling program requirements;
- Inspection and maintenance requirements for monitoring wells;
- Monitoring well decommissioning procedures; and
- Annual inspection and periodic certification.

Reporting requirements are provided in Section 7.0 of this SMP.

4.2 Site-Wide Inspection

Site-wide inspections will be performed at a minimum of once per year. These periodic inspections must be conducted when the ground surface is visible (i.e. no snow cover). Site-wide inspections will be performed by a qualified environmental professional as defined in 6 NYCRR Part 375. Modification to the frequency or duration of the inspections will require approval from the NYSDEC project manager. Site-wide inspections will also be performed after all severe weather conditions that may affect ECs or monitoring devices. During these inspections, an inspection form will be completed as provided in Appendix I Site Management Forms. The form will compile–Site-wide inspections will obtain sufficient information to assess the following:

- Compliance with all ICs, including site usage;
- An evaluation of the condition and continued effectiveness of ECs;
- General site conditions at the time of the inspection;
- Whether stormwater management systems, such as basins and outfalls, are working as designed;
- The site management activities being conducted including, where appropriate, confirmation sampling and a health and safety inspection; and
- Confirm that site records are up to date.

Inspections of all remedial components installed at the site will be conducted. A comprehensive site-wide inspection will be conducted and documented according to the SMP schedule, regardless of the frequency of the Periodic Review Report. The inspections will determine and document the following:

- Whether ECs continue to perform as designed;
- If these controls continue to be protective of human health and the environment;

- Compliance with requirements of this SMP and the Deed Restriction;
- Achievement of remedial performance criteria; and
- If site records are complete and up to date.

Reporting requirements are outlined in Section 7.0 of this plan.

Inspections will also be performed in the event of an emergency. If an emergency, such as a natural disaster or an unforeseen failure of any of the ECs occurs that reduces or has the potential to reduce the effectiveness of ECs in place at the site, verbal notice to the NYSDEC project manager must be given by noon of the following day. In addition, an inspection of the site will be conducted within 5 days of the event to verify the effectiveness of the IC/ECs implemented at the site by a qualified environmental professional, as defined in 6 NYCCR Part 375. Written confirmation must be provided to the NYSDEC project manager within 7 days of the event that includes a summary of actions taken, or to be taken, and the potential impact to the environment and the public. The remedial party will submit follow-up status reports to the NYSDEC within 45 days of the event on actions taken to respond to any emergency event requiring ongoing responsive action, describing and documenting actions taken to restore the effectiveness of the ECs.

4.3 Post-Remediation Media Monitoring and Sampling

Samples shall be collected from the groundwater on a bi-annual basis. Sampling locations required analytical parameters and schedule are provided in Table 5 – Remedial System Sampling Requirements and Schedule below. Modification to the frequency or sampling requirements will require approval from the NYSDEC project manager.

Table 5 – Post Remediation Sampling Requirements and Schedule

	VOCs (EPA Method 8260D)	Schedule
MW-114	X	Bi-annual
MW-115	X	Bi-annual
MW-117	X	Bi-annual
MW-118	X	Bi-annual
MW-122	X	Bi-annual
MW-124d	X	Bi-annual

Detailed sample collection and analytical procedures and protocols are provided in Appendix F.

4.3.1 Groundwater Sampling

Groundwater monitoring will be performed biannually. Modification to the frequency or sampling requirements will require approval from the NYSDEC project manager.

Detailed sample collection and analytical procedures and protocols are provided in Appendix F. Historically, samples were collected using low-flow sampling techniques. However, based on historical water levels and insufficient water volume, conventional sampling techniques will be utilized beginning with the 2027 groundwater sampling event. Three well volumes will be purged from each monitoring well, or until dry. Sampling will be collected using a bailer upon recovery.

There are currently no complete human exposure pathways for Site contaminants. Groundwater monitoring wells in the sampling program included MW-114, MW-115, MW-115d, MW-116, MW-117, MW-118, MW-121, MW-122, MW-124s, and MW-124d. On September 10, 2024, NYSDEC approved the reduction on the groundwater sampling program to include MW-114, MW-115, MW-117, MW-118, MW-122, and MW-124d. Table 6 presents monitoring well construction details.

Monitoring well construction detail logs are included in Appendix H of this document.

If biofouling or silt accumulation occurs in the on-site and/or off-site monitoring wells, the wells will be physically agitated/surged and redeveloped. Additionally, monitoring wells will be properly decommissioned and replaced if an event renders the wells unusable.

Repairs and/or replacement of wells in the monitoring well network will be performed based on assessments of structural integrity and overall performance.

The NYSDEC project manager will be notified prior to any repair or decommissioning of any monitoring well for the purpose of replacement, and the repair or decommissioning and replacement process will be documented in the subsequent Periodic Review Report. Well decommissioning without replacement will be done only with the prior approval of the NYSDEC project manager. Well abandonment will be performed in accordance with NYSDEC's guidance entitled "CP-43: Groundwater Monitoring Well Decommissioning Procedures." Monitoring wells that are decommissioned because they have been rendered unusable will be replaced in kind in the nearest available location, unless otherwise approved by the NYSDEC project manager.

The sampling frequency may only be modified with the approval of the NYSDEC project manager. This SMP will be modified to reflect changes in sampling plans approved by the NYSDEC project manager.

Deliverables for the groundwater monitoring program are specified in Section 7.0 – Reporting Requirements.

4.3.2 Soil Vapor Intrusion Sampling

The Soil vapor intrusion sampling program was discontinued by NYSDEC on September 10, 2024 provided that no building expansions/renovations occur, or new structures are constructed at the Site. If any expansions, renovations, or new construction occurs at the Site, then sub-slab soil vapor and indoor air monitoring program will be re-instated.

Any alteration to the on-Site building's foundation, footprint, new building construction, etc. will require the sub-slab and indoor air monitoring program to be re-instated to ensure sub-slab soil vapor and indoor air concentrations have not changed and there are no exposures occurring due to SVI.

4.3.3 Monitoring and Sampling Protocol

All sampling activities will be recorded in a field book and associated sampling log Other observations (e.g., groundwater monitoring well integrity) will be noted on the sampling log. The sampling log will serve as the inspection form for the monitoring network. The sampling log is provided in Appendix F. Additional detail regarding monitoring and sampling protocols are provided in the site-specific Sample and Analysis Plan provided as Appendix F of this document.

5.0 OPERATION AND MAINTENANCE PLAN

The Site remedy does not rely on any mechanical systems, such as groundwater treatment systems, sub-slab depressurization systems, or air sparge/soil vapor extraction systems to protect public health and the environment. Therefore, the operation and maintenance of such components is not included in this SMP.

6.0 PERIODIC ASSESSMENTS/EVALUATIONS

6.1 Climate Change Vulnerability Assessment

Increases in both the severity and frequency of storms/weather events, an increase in sea level elevations along with accompanying flooding impacts, shifting precipitation patterns and wide temperature fluctuation, resulting from global climactic change and instability, have the potential to significantly impact the performance, effectiveness and protectiveness of a given site and associated remedial systems. Vulnerability assessments provide information so that the site and associated remedial systems are prepared for the impacts of the increasing frequency and intensity of severe storms/weather events and associated flooding.

This section provides a current vulnerability assessment that evaluates the vulnerability of the site and/or engineering controls to severe storms/weather events and associated flooding. This section also identifies vulnerability assessment updates that will be conducted for the site in Periodic Review Reports in the future.

- Flooding: The Site is mapped within Flood Zone C, identified as an area of minimal flooding, on FEMA Flood Insurance Rate Map (FIRM) for the City of Geneva, New York, Panel Number 360599-0001B (Effective April 15, 1982). Flood related impacts to Site institutional and engineering controls are not expected.
- Sea Level Rise: The Site is not located near a coast and is not at risk for flooding related to sea level rise.
- Site Drainage and Stormwater Management: The Site does not have a significant risk of flooding during rainfall or snowfall events, as indicated by the FEMA FIRM maps. On-site stormwater drainage is managed with existing stormwater features including stormwater catch basins.
- Erosion: No evidence of erosion at the Site has been identified.
- High Winds: The Site is located within an area of “Relatively Low” risk for strong winds, according to the FEMA National Risk Index (NRI). The Site institutional

and engineering controls are not anticipated to be susceptible to impacts related to high winds.

- Drought: The Site is located within an area of "Relatively Moderate" risk of drought according to the FEMA NRI. When possible, groundwater monitoring activities will be conducted during the high-water table season.

- Electricity: Currently engineering controls are not required.

- Spill/Contaminant Release: Groundwater monitoring well purge water will be handled, transported and disposed off-site at a permitted facility in accordance with applicable local, State, and Federal regulations.

- Wildfires: The site is located within an area of "Very Low" risk of wildfire according to the FEMA NRI. Wildfire impacts to Site institutional and engineering controls are not expected.

This section will be updated if a remedial system is required in the future.

6.2 Green Remediation Evaluation

NYSDEC's DER-31 Green Remediation requires that green remediation concepts and techniques be considered during all stages of the remedial program including site management, with the goal of improving the sustainability of the cleanup and summarizing the net environmental benefit of any implemented green technology. This section provides an environmental footprint analysis of the remedy, as implemented at the time of this SMP. This section of the SMP also provides a summary of green remediation evaluations to be completed for the site during site management and reported in Periodic Review Reports (PRRs).

An environmental footprint analysis for the Site remedial activities was conducted using the United Stated Environmental Protection Agency (USEPA) Simplified Greenhouse Gas (GHG) Emissions Calculator. The footprint analysis considers the biannual groundwater monitoring and annual site visit as the only components of the remedy. These components generate negligible quantities of materials and waste, do not require on-site water and do not require on-site electricity. Landscape/ecosystem impacts

are limited to the on-site groundwater wells. The results of the footprint analysis are summarized in Table 7 and provided in Appendix J.

Table 7 – Total GHG Emissions Resulting From Site Activities

Direct CO_2 Equivalent Emissions (metric tons) – from fleet/mobile combustion resulting from the annual site inspection[1]	0.0439
Direct CO_2 Equivalent Emissions (metric tons) – from fleet/mobile combustion resulting from the groundwater monitoring (only conducted biannually)[2]	0.0439

Notes:
[1]The GHG calculation uses the EPA GHG Calculator tool with current eGRID NPCC Upstate NY CO_2 emission rates. This calculation assumes one site visit by passenger car totaling 90 miles.
[2]The GHG calculation uses the EPA GHG Calculator tool with current eGRID NPCC Upstate NY CO_2 emission rates. This calculation assumes one site visit by light-duty truck totaling 90 miles.

Best Management Practices (BMP) will be implemented to minimize environmental impacts during site management activities. The following actions will be undertaken:

- Coordinate Site visits to combine Site inspection and groundwater monitoring events during years when both activities are required.

- Minimizing waste generation by reusing materials when feasible. For example, a stainless steel bailer may be used for sample collection rather than a disposable high-density polyethylene (HDPE) bailer at each monitoring location.

- Reduce greenhouse emissions by shutting off vehicles when not in use for periods exceeding five minutes.

6.2.1 Timing of Green Remediation Evaluations

As of the date of this SMP remedial systems do not exist at the Site.

For major remedial system components, green remediation evaluations and corresponding modifications will be undertaken as part of a formal Remedial System Optimization (RSO), or at any time that the NYSDEC project manager feels appropriate,

(e.g. during significant maintenance events or in conjunction with storm recovery activities).

Modifications resulting from green remediation evaluations will be routinely implemented and scheduled to occur during planned/routine operation and maintenance activities after approval from the DER project manager. Reporting of these modifications will be presented in the PRR.

6.2.2 Remedial Systems

As of the date of this SMP remedial systems do not exist at the Site.

Remedial systems will be operated properly considering the current site conditions to conserve materials and resources to the greatest extent possible. Consideration will be given to operating rates and use of reagents and consumables. Spent materials will be sent for recycling, as appropriate.

6.2.3 Building Operations

As of the date of this SMP remedial systems do not exist at the Site.

Structures including buildings and sheds will be operated and maintained to provide for the most efficient operation of the remedy, while minimizing energy, waste generation and water consumption. Building and structures on the Site are not currently utilized for any Site remedial operations.

6.2.4 Frequency of System Checks, Sampling and Other Periodic Activities

As of the date of this SMP remedial systems do not exist at the Site.

Transportation to and from the Site, use of consumables in relation to visiting the Site in order to conduct system checks and/or collect samples, and shipping samples to a

laboratory for analyses have direct and/or inherent energy costs. The schedule and/or means of these periodic activities have been prepared so that these tasks can be accomplished in a manner that does not impact remedy protectiveness but reduces expenditure of energy or resources.

6.2.5 Metrics and Reporting

As of the date of this SMP remedial systems do not exist at the Site.

As discussed in Section 7.0 and as shown in Appendix I – Site Management Forms, information on energy usage, solid waste generation, transportation and shipping, water usage and land use and ecosystems will be recorded to facilitate and document consistent implementation of green remediation during site management and to identify corresponding benefits. A set of metrics has been developed and will be evaluated over time to ensure that green remediation actions are achieving the desired results. These metrics will be evaluated annually. Evaluation results, and the completed inspection form will be provided in the annual PRRs.

6.3 Remedial System Optimization

As of the date of this SMP remedial systems do not exist at the Site.

A Remedial System Optimization (RSO) study will be conducted any time that the NYSDEC project manager or the remedial party requests in writing that an in-depth evaluation of the remedy is needed. An RSO may be appropriate if any of the following occur:

- The remedial actions have not met or are not expected to meet RAOs in the time frame estimated in the Decision Document;
- The management and operation of the remedial system is exceeding the estimated costs;
- The remedial system is not performing as expected or as designed;

- Previously unidentified source material may be suspected;
- Plume shift has potentially occurred;
- Site conditions change due to development, change of use, change in groundwater use, etc.;
- There is an anticipated transfer of the site management to another remedial party or agency; and
- A new and applicable remedial technology becomes available.

An RSO will provide a critique of a site's conceptual model, give a summary of past performance, document current cleanup practices, summarize progress made toward the site's cleanup goals, gather additional performance or media specific data and information and provide recommendations for improvements to enhance the ability of the present system to reach RAOs or to provide a basis for changing the remedial strategy.

A predefined schedule for RSO evaluation and reporting will be established if requested.

The RSO study will focus on overall site cleanup strategy, process optimization and management with the intent of identifying impediments to cleanup and improvements to site operations to increase efficiency, cost effectiveness and remedial time frames. Green remediation technology and principles are to be considered when performing the RSO.

7.0 REPORTING REQUIREMENTS

7.1 Site Management Reports

All site management inspection, maintenance, and monitoring events will be conducted by a qualified environmental professional as defined in 6 NYCRR Part 375, a Professional Engineer (PE) who is licensed and registered in New York State, or a qualified person who directly reports to a PE who is licensed and registered in New York State.

All applicable inspection forms and other records, including media sampling data and system maintenance reports, generated for the site during the reporting period will be provided in electronic format to the NYSDEC in accordance with the requirements of Table 8 and summarized in the Periodic Review Report.

Table 8: Schedule of Interim Monitoring/Inspection Reports

Task/Report	**Reporting Frequency***
Soil Screening for excavations disturbing > one foot bgs	Within 90 days after completion of excavation
Sitewide Inspections	Report results in annual PRRs

* The frequency of events will be conducted as specified until otherwise approved by the NYSDEC project manager.

All interim monitoring/inspections reports will include, at a minimum:

- Date of event or reporting period;
- Name, company, and position of person(s) conducting monitoring/inspection activities;
- Description of the activities performed;

- Where appropriate, color photographs or sketches showing the approximate location of any problems or incidents noted (included either on the checklist/form or on an attached sheet);
- Type of samples collected (e.g., sub-slab vapor, indoor air, outdoor air);
- Copies of all field forms completed (e.g., well sampling logs, chain-of-custody documentation);
- Sampling results in comparison to appropriate standards/criteria;
- A figure illustrating sample type and sampling locations;
- Copies of all laboratory data sheets and the required laboratory data deliverables required for all points sampled (to be submitted electronically in the NYSDEC-identified format);
- Any observations, conclusions, or recommendations; and
- A determination as to whether contaminant conditions have changed since the last reporting event.

Non-routine maintenance event reporting forms will include, at a minimum:

- Date of event;
- Name, company, and position of person(s) conducting non-routine maintenance/repair activities;
- Description of non-routine activities performed;
- Where appropriate, color photographs or sketches showing the approximate location of any problems or incidents (included either on the form or on an attached sheet); and
- Other documentation such as copies of invoices for repair work, receipts for replacement equipment, etc. (attached to the checklist/form).

Data will be reported in digital format as determined by the NYSDEC. Currently, data is to be supplied electronically and submitted to the NYSDEC EQuIS^{TM} database in

accordance with the requirements found at this link http://www.dec.ny.gov/chemical/62440.html.

7.2 Periodic Review Report

A Periodic Review Report (PRR) will be submitted annually to the NYSDEC project manager or less frequently as approved by the NYSDEC project manager. In the event that the site is subdivided into separate parcels with different ownership, a single Periodic Review Report will be prepared that addresses the site described in the Deed Restriction. The report will be prepared in accordance with NYSDEC's DER-10 and submitted within 30 days of the end of each certification period. Media sampling results will also be incorporated into the Periodic Review Report. The report will include:

- Identification, assessment and certification of all ECs/ICs required by the remedy for the site.
- Results of the required annual site inspections, fire inspections and severe condition inspections, if applicable.
- Description of any change of use, import of materials, or excavation that occurred during the certifying period.
- All applicable site management forms and other records generated for the site during the reporting period in the NYSDEC-approved electronic format, if not previously submitted.
- Identification of any wastes generated during the reporting period, along with waste characterization data, manifests, and disposal documentation.
- A summary of any discharge monitoring data and/or information generated during the reporting period, with comments and conclusions.
- Data summary tables and graphical representations of contaminants of concern by media (groundwater), which include a listing of all compounds analyzed, along with the applicable standards, with all exceedances highlighted. These tables and figures will include a presentation of past data as part of an evaluation of contaminant concentration trends, including but not limited to:
  - Trend monitoring graphs that present groundwater contaminant levels from before the start of the remedy implementation to the most current sampling data;

- Trend monitoring graphs depicting system influent analytical data on a per event and cumulative basis;
- A current plume map for sites with remaining groundwater contamination; and
- A groundwater elevation contour map for each gauging event.

- Results of all analyses, copies of all laboratory data sheets, and the required laboratory data deliverables for all samples collected during the reporting period will be submitted in digital format as determined by the NYSDEC. Currently, data is supplied electronically and submitted to the NYSDEC EQuIS™ database in accordance with the requirements found at this link: http://www.dec.ny.gov/chemical/62440.html.

- A site evaluation, which includes the following:
  - The compliance of the remedy with the requirements of the site-specific Remedial Action Work Plan (RAWP), ROD or Decision Document;
  - The operation and the effectiveness of all treatment units, etc., including identification of any needed repairs or modifications;
  - Any new conclusions or observations regarding site contamination based on inspections or data generated by the Monitoring and Sampling Plan for the media being monitored;
  - Recommendations regarding any necessary changes to the remedy and/or Monitoring and Sampling Plan;
  - An update to the climate change vulnerability assessment if site or external conditions have changed since the previous assessment, and recommendations to address vulnerabilities;
  - A summary of the Green Remediation evaluation, including a quantitative and qualitative overview of a site's environmental impacts and recommendations to improve the remedy's environmental footprint. The PRR will include the completed Summary of Green Remediation Metrics form provided in Appendix I;
  - An evaluation of trends in contaminant levels in the affected media to determine if the remedy continues to be effective in achieving remedial goals as specified by the RAWP, ROD or Decision Document; and
  - The overall performance and effectiveness of the remedy.

7.2.1 Certification of Institutional and Engineering Controls

Following the last inspection of the reporting period, a qualified environmental professional as defined in 6 NYCRR Part 375 or Professional Engineer licensed to practice and registered in New York State will prepare, and include in the Periodic Review Report, the following certification as per the requirements of NYSDEC DER-10:

"*For each institutional or engineering control identified for the site, I certify that all of the following statements are true:*

- *The inspection of the site to confirm the effectiveness of the institutional and engineering controls required by the remedial program was performed under my direction;*
- *The institutional control and/or engineering control employed at this site is unchanged from the date the control was put in place, or last approved by the Department;*
- *Nothing has occurred that would impair the ability of the control to protect the public health and environment;*
- *Nothing has occurred that would constitute a violation or failure to comply with any site management plan for this control;*
- *Access to the site will continue to be provided to the Department to evaluate the remedy, including access to evaluate the continued maintenance of this control;*
- *If a financial assurance mechanism is required under the oversight document for the site, the mechanism remains valid and sufficient for the intended purpose under the document;*
- *Use of the site is compliant with the Deed Restriction;*
- *The engineering control systems are performing as designed and are effective;*
- *To the best of my knowledge and belief, the work and conclusions described in this certification are in accordance with the requirements of the site remedial program and generally accepted engineering practices; and*
- *The information presented in this report is accurate and complete.*

I certify that all information and statements in this certification form are true. I understand that a false statement made herein is punishable as a Class "A" misdemeanor, pursuant to Section 210.45 of the Penal Law. I, [name], of [business address], am certifying as [Owner/Remedial Party or Owner's/Remedial Party's Designated Site Representative] and I have been authorized and designated by all site owners/remedial parties to sign this certification] for the site."

"I certify that the New York State Education Department has granted a Certificate of Authorization to provide Professional Engineering services to the firm that prepared this Periodic Review Report."

- *No new information has come to my attention, including groundwater monitoring data from wells located at the site boundary, if any, to indicate that the assumptions made in the qualitative exposure assessment of off-site contamination are no longer valid; and*
- *The assumptions made in the qualitative exposure assessment remain valid.*

The signed certification will be included in the Periodic Review Report.

The Periodic Review Report will be submitted, in electronic format, to the NYSDEC project manager and the NYSDOH project manager. The Periodic Review Report may also need to be submitted in hard-copy format if requested by the NYSDEC project manager.

7.3 Corrective Measures Work Plan

If any component of the remedy is found to have failed, or if the periodic certification cannot be provided due to the failure of an institutional or engineering control or failure to conduct site management activities, a Corrective Measures Work Plan will be submitted to the NYSDEC project manager for approval. This plan will explain the failure and provide the details and schedule for performing work necessary to correct the failure. Unless an emergency condition exists, no work will be performed pursuant to the

Corrective Measures Work Plan until it has been approved by the NYSDEC project manager.

7.4 Remedial System Optimization Report

As of the date of this SMP remedial systems do not exist at the Site.

If an RSO is to be performed (see Section 6.3), upon completion of an RSO, an RSO report must be submitted to the NYSDEC project manager for approval. The RSO report will document the research/ investigation and data gathering that was conducted, evaluate the results and facts obtained, present a revised conceptual site model and present recommendations. RSO recommendations are to be implemented upon approval from the NYSDEC. Additional work plans, design documents, HASPs etc., may still be required to implement the recommendations, based upon the actions that need to be taken. A final engineering report and update to the SMP may also be required.

The RSO report will be submitted, in electronic format, to the NYSDEC project manager and the NYSDOH project manager.

8.0 REFERENCES

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TABLES

TABLE 1
Notifications
(In Text)

Table 2
Groundwater Elevation Data
Former Monarch Chemicals
Geneva, NY

Well	Date	Top of Casing (ft AMSL)	Depth to Water (ft TOC)	Potentiometric Elevation (ft AMSL)
MW-114	10/18/2005	462.03	10.20	451.83
MW-114	1/12/2006	462.03	9.45	452.58
MW-114	11/21/2006	462.03	9.16	452.87
MW-114	9/25/2007	462.03	13.51	448.52
MW-114	4/7/2008	462.03	8.95	453.08
MW-114	8/4/2008	462.03	10.2	451.83
MW-114	8/15/2008	462.03	9.73	452.30
MW-114	10/8/2008	462.03	11.66	450.37
MW-114	11/17/2008	462.03	9.73	452.30
MW-114	4/27/2009	462.03	10.16	451.87
MW-114	8/3/2009	462.03	10.13	451.90
MW-114	4/14/2010	462.03	10.21	451.82
MW-114	8/9/2010	462.03	11.91	450.12
MW-114	11/8/2010	462.03	10.57	451.46
MW-114	11/9/2010	462.03	10.64	451.39
MW-114	11/10/2010	462.03	10.67	451.36
MW-114	11/11/2010	462.03	10.72	451.31
MW-114	11/12/2010	462.03	10.76	451.27
MW-114	11/13/2010	462.03	10.79	451.24
MW-114	11/14/2010	462.03	10.80	451.23
MW-114	11/15/2010	462.03	10.87	451.16
MW-114	11/19/2010	462.03	9.98	452.05
MW-114	11/20/2010	462.03	10.54	451.49
MW-114	4/11/2011	462.03	9.57	452.46
MW-114	4/27/2011	462.03	7.4	454.63
MW-114	6/2/2011	462.03	10.07	451.96
MW-114	12/9/2011	462.03	9.98	452.05
MW-114	3/27/2013	462.03	13.31	448.72
MW-114	12/9/2017	462.03	10.52	451.51
MW-114	12/14/2021	462.03	10.43	451.60
MW-114	3/18/2023	462.03	9.07	452.96
MW-114	7/23/2025	462.03	12.70	449.33

Table 2
Groundwater Elevation Data
Former Monarch Chemicals
Geneva, NY

Well	Date	Top of Casing (ft AMSL)	Depth to Water (ft TOC)	Potentiometric Elevation (ft AMSL)
MW-115	10/18/2005	462.25	9.65	452.60
MW-115	1/12/2006	462.25	7.10	455.15
MW-115	11/21/2006	462.25	7.00	455.25
MW-115	9/25/2007	462.25	12.57	449.68
MW-115	4/7/2008	462.25	6.37	455.88
MW-115	8/4/2008	462.25	9.42	452.83
MW-115	8/15/2008	462.25	8.92	453.33
MW-115	10/8/2008	462.25	10.58	451.67
MW-115	11/17/2008	462.25	8.23	454.02
MW-115	4/27/2009	462.25	7.57	454.68
MW-115	8/3/2009	462.25	9.18	453.07
MW-115	4/14/2010	462.25	8.44	453.81
MW-115	8/9/2010	462.25	11.2	451.05
MW-115	11/8/2010	462.25	9.79	452.46
MW-115	11/9/2010	462.25	9.86	452.39
MW-115	11/10/2010	462.25	9.89	452.36
MW-115	11/12/2010	462.25	9.92	452.33
MW-115	11/13/2010	462.25	9.89	452.36
MW-115	11/14/2010	462.25	9.89	452.36
MW-115	11/15/2010	462.25	9.93	452.32
MW-115	11/19/2010	462.25	9.75	452.50
MW-115	11/20/2010	462.25	9.63	452.62
MW-115	4/11/2011	462.25	7.18	455.07
MW-115	4/27/2011	462.25	5.38	456.87
MW-115	6/2/2011	462.25	7.85	454.40
MW-115	12/9/2011	462.25	8.92	453.33
MW-115	3/27/2013	462.25	5.50	456.75
MW-115	12/9/2017	462.25	8.29	453.96
MW-115	12/14/2021	462.25	7.80	454.45
MW-115	3/20/2023	462.25	6.78	455.47
MW-115	7/23/2025	462.25	12.91	451.31

Table 2
Groundwater Elevation Data
Former Monarch Chemicals
Geneva, NY

Well	Date	Top of Casing (ft AMSL)	Depth to Water (ft TOC)	Potentiometric Elevation (ft AMSL)
MW-115d	11/12/2010	462.06	13.19	448.87
MW-115d	11/13/2010	462.06	11.82	450.24
MW-115d	11/14/2010	462.06	11.81	450.25
MW-115d	11/15/2010	462.06	11.85	450.21
MW-115d	11/20/2010	462.06	11.57	450.49
MW-115d	4/11/2011	462.06	11.33	450.73
MW-115d	4/27/2011	462.06	11.02	451.04
MW-115d	6/2/2011	462.06	11.53	450.53
MW-115d	12/9/2011	462.06	11.62	450.44
MW-115d	3/27/2013	462.06	11.95	450.11
MW-115d	12/9/2017	462.06	11.54	450.52
MW-115d	12/14/2021	462.06	11.35	450.71
MW-115d	3/20/2023	462.06	10.26	451.80
MW-115d	7/23/2025	462.06	12.20	449.86

Table 2
Groundwater Elevation Data
Former Monarch Chemicals
Geneva, NY

Well	Date	Top of Casing (ft AMSL)	Depth to Water (ft TOC)	Potentiometric Elevation (ft AMSL)
MW-116	10/18/2005	462.44	9.4	453.04
MW-116	1/11/2006	462.44	9.5	452.94
MW-116	11/21/2006	462.44	8.16	454.28
MW-116	9/25/2007	462.44	13.29	449.15
MW-116	4/7/2008	462.44	6.64	455.80
MW-116	8/4/2008	462.44	8.78	453.66
MW-116	8/15/2008	462.44	7.49	454.95
MW-116	10/8/2008	462.44	10.37	452.07
MW-116	11/17/2008	462.44	5.42	457.02
MW-116	4/27/2009	462.44	8.85	453.59
MW-116	8/3/2009	462.44	4.8	457.64
MW-116	4/14/2010	462.44	8.62	453.82
MW-116	8/9/2010	462.44	10.7	451.74
MW-116	11/8/2010	462.44	8.43	454.01
MW-116	11/9/2010	462.44	8.74	453.70
MW-116	11/10/2010	462.44	8.92	453.52
MW-116	11/11/2010	462.44	9.09	453.35
MW-116	11/12/2010	462.44	9.20	453.24
MW-116	11/13/2010	462.44	9.30	453.14
MW-116	11/14/2010	462.44	9.34	453.10
MW-116	11/15/2010	462.44	9.47	452.97
MW-116	11/20/2010	462.44	6.52	455.92
MW-116	4/11/2011	462.44	7.4	455.04
MW-116	4/27/2011	462.44	2.77	459.67
MW-116	6/2/2011	462.44	8.16	454.28
MW-116	12/9/2011	462.44	7.04	455.40
MW-116	3/27/2013	462.44	7.81	454.63
MW-116	12/9/2017	462.44	9.21	453.23
MW-116	12/15/2021	462.44	8.50	453.94
MW-116	3/19/2023	462.44	4.31	458.13
MW-116	7/23/2025	462.44	12.75	449.69

Table 2
Groundwater Elevation Data
Former Monarch Chemicals
Geneva, NY

Well	Date	Top of Casing (ft AMSL)	Depth to Water (ft TOC)	Potentiometric Elevation (ft AMSL)
MW-117	10/18/2005	463.44	6.32	457.12
MW-117	1/12/2006	463.44	4.90	458.54
MW-117	11/21/2006	463.44	4.05	459.39
MW-117	9/25/2007	463.44	8.83	454.61
MW-117	4/7/2008	463.44	2.62	460.82
MW-117	8/4/2008	463.44	5.81	457.63
MW-117	8/15/2008	463.44	4.67	458.77
MW-117	10/8/2008	463.44	7.28	456.16
MW-117	11/17/2008	463.44	3.3	460.14
MW-117	4/27/2009	463.44	3.25	460.19
MW-117	8/3/2009	463.44	4.38	459.06
MW-117	4/15/2010	463.44	3.86	459.58
MW-117	8/9/2010	463.44	4.88	458.56
MW-117	11/8/2010	463.44	3.89	459.55
MW-117	11/9/2010	463.44	3.93	459.51
MW-117	11/10/2010	463.44	4.00	459.44
MW-117	11/11/2010	463.44	4.06	459.38
MW-117	11/12/2010	463.44	4.11	459.33
MW-117	11/13/2010	463.44	4.18	459.26
MW-117	11/14/2010	463.44	4.25	459.19
MW-117	11/15/2010	463.44	4.31	459.13
MW-117	11/20/2010	463.44	4.31	459.13
MW-117	4/11/2011	463.44	2.71	460.73
MW-117	4/27/2011	463.44	1.28	462.16
MW-117	6/2/2011	463.44	2.88	460.56
MW-117	12/9/2011	463.44	3.74	459.70
MW-117	3/27/2013	463.44	5.35	458.09
MW-117	12/9/2017	463.44	4.82	458.62
MW-117	12/15/2021	463.44	4.88	458.56
MW-117	3/18/2023	463.44	2.60	460.84
MW-117	7/23/2025	463.44	7.14	456.30

Table 2
Groundwater Elevation Data
Former Monarch Chemicals
Geneva, NY

Well	Date	Top of Casing (ft AMSL)	Depth to Water (ft TOC)	Potentiometric Elevation (ft AMSL)
MW-118	10/18/2005	462.40	6.95	455.45
MW-118	1/12/2006	462.40	5.55	456.85
MW-118	11/21/2006	462.40	5.20	457.20
MW-118	9/25/2007	462.40	9.43	452.97
MW-118	12/20/2007	462.4	6.51	455.89
MW-118	4/7/2008	462.4	4.90	457.50
MW-118	8/4/2008	462.4	6.32	456.08
MW-118	8/15/2008	462.4	5.35	457.05
MW-118	10/8/2008	462.4	7.95	454.45
MW-118	11/17/2008	462.4	5.63	456.77
MW-118	4/27/2009	462.4	6.37	456.03
MW-118	8/3/2009	462.4	5.2	457.20
MW-118	4/15/2010	462.4	6.73	455.67
MW-118	8/9/2010	462.4	7.7	454.70
MW-118	11/8/2010	462.4	6.79	455.61
MW-118	11/9/2010	462.4	6.91	455.49
MW-118	11/10/2010	462.4	7.05	455.35
MW-118	11/11/2010	462.4	7.15	455.25
MW-118	11/12/2010	462.4	7.24	455.16
MW-118	11/13/2010	462.4	7.26	455.14
MW-118	11/14/2010	462.4	7.31	455.09
MW-118	11/15/2010	462.4	7.46	454.94
MW-118	11/20/2010	462.4	6.93	455.47
MW-118	4/11/2011	462.4	5.78	456.62
MW-118	4/27/2011	462.4	4.88	457.52
MW-118	6/2/2011	462.4	5.84	456.56
MW-118	12/9/2011	462.4	6.02	456.38
MW-118	3/27/2013	462.4	6.53	455.87
MW-118	3/25/2015	462.4	6.98	455.42
MW-118	12/9/2017	462.4	6.58	455.82
MW-118	12/15/2021	462.4	5.55	456.85
MW-118	3/18/2023	462.4	4.84	457.56
MW-118	7/23/2025	462.4	8.45	453.95

Table 2
Groundwater Elevation Data
Former Monarch Chemicals
Geneva, NY

Well	Date	Top of Casing (ft AMSL)	Depth to Water (ft TOC)	Potentiometric Elevation (ft AMSL)
MW-121	10/18/2005	461.77	13.65	448.12
MW-121	1/12/2006	461.77	13.25	448.52
MW-121	11/21/2006	461.77	13.00	448.77
MW-121	9/25/2007	461.77	dry	dry
MW-121	12/20/2007	461.77	13.42	448.35
MW-121	4/7/2008	461.77	12.73	449.04
MW-121	8/4/2008	461.77	13.63	448.14
MW-121	8/15/2008	461.77	13.58	448.19
MW-121	10/8/2008	461.77	13.74	448.03
MW-121	11/17/2008	461.77	13.13	448.64
MW-121	4/27/2009	461.77	13.5	448.27
MW-121	8/3/2009	461.77	13.25	448.52
MW-121	4/14/2010	461.77	13.3	448.47
MW-121	8/9/2010	461.77	13.72	448.05
MW-121	11/8/2010	461.77	13.41	448.36
MW-121	11/9/2010	461.77	13.49	448.28
MW-121	11/10/2010	461.77	13.54	448.23
MW-121	11/11/2010	461.77	13.57	448.20
MW-121	11/12/2010	461.77	13.63	448.14
MW-121	11/13/2010	461.77	13.64	448.13
MW-121	11/14/2010	461.77	13.66	448.11
MW-121	11/15/2010	461.77	13.71	448.06
MW-121	11/20/2010	461.77	13.24	448.53
MW-121	4/11/2011	461.77	12.83	448.94
MW-121	4/27/2011	461.77	11.27	450.50
MW-121	6/2/2011	461.77	13.53	448.24
MW-121	12/9/2011	461.77	13.26	448.51
MW-121	3/27/2013	461.77	13.39	448.38
MW-121	12/9/2017	461.77	13.56	448.21
MW-121	12/16/2021	461.77	12.74	449.03
MW-121	3/22/2023	461.77	12.10	449.67
MW-121	7/23/2025	461.77	dry	dry

Table 2
Groundwater Elevation Data
Former Monarch Chemicals
Geneva, NY

Well	Date	Top of Casing (ft AMSL)	Depth to Water (ft TOC)	Potentiometric Elevation (ft AMSL)
MW-122	10/18/2005	461.65	10.30	451.35
MW-122	1/12/2006	461.65	9.25	452.40
MW-122	11/21/2006	461.65	8.79	452.86
MW-122	9/25/2007	461.65	dry	dry
MW-122	12/20/2007	461.65	9.2	452.45
MW-122	4/7/2008	461.65	8.16	453.49
MW-122	8/4/2008	461.65	10.11	451.54
MW-122	8/15/2008	461.65	8.84	452.81
MW-122	10/8/2008	461.65	10.78	450.87
MW-122	11/17/2008	461.65	8.1	453.55
MW-122	4/27/2009	461.65	10.18	451.47
MW-122	8/3/2009	461.65	8.28	453.37
MW-122	4/14/2010	461.65	9.63	452.02
MW-122	8/9/2010	461.65	dry	dry
MW-122	11/8/2010	461.65	9.28	452.37
MW-122	11/9/2010	461.65	9.47	452.18
MW-122	11/10/2010	461.65	9.61	452.04
MW-122	11/11/2010	461.65	9.75	451.90
MW-122	11/12/2010	461.65	9.48	452.17
MW-122	11/13/2010	461.65	9.91	451.74
MW-122	11/14/2010	461.65	9.97	451.68
MW-122	11/15/2010	461.65	10.10	451.55
MW-122	11/20/2010	461.65	8.39	453.26
MW-122	4/11/2011	461.65	8.48	453.17
MW-122	4/27/2011	461.65	6.25	455.40
MW-122	6/2/2011	461.65	9.52	452.13
MW-122	12/9/2011	461.65	8.40	453.25
MW-122	3/27/2013	461.65	9.84	451.81
MW-122	12/9/2017	461.65	10.02	451.63
MW-122	12/16/2021	461.65	8.86	452.79
MW-122	3/22/2023	461.65	3.49	458.68
MW-122	7/23/2025	461.65	dry	dry

Table 2
Groundwater Elevation Data
Former Monarch Chemicals
Geneva, NY

Well	Date	Top of Casing (ft AMSL)	Depth to Water (ft TOC)	Potentiometric Elevation (ft AMSL)
MW-124s	8/15/2008	462.17	6.75	455.42
MW-124s	10/8/2008	462.17	7.44	454.73
MW-124s	11/17/2008	462.17	2.53	459.64
MW-124s	4/27/2009	462.17	3.00	459.17
MW-124s	8/3/2009	462.17	4.02	458.15
MW-124s	4/14/2010	462.17	3.86	458.31
MW-124s	8/9/2010	462.17	6.82	455.35
MW-124s	11/8/2010	462.17	4.84	457.33
MW-124s	11/9/2010	462.17	5.01	457.16
MW-124s	11/10/2010	462.17	5.29	456.88
MW-124s	11/11/2010	462.17	5.70	456.47
MW-124s	11/12/2010	462.17	6.03	456.14
MW-124s	11/13/2010	462.17	6.31	455.86
MW-124s	11/14/2010	462.17	6.50	455.67
MW-124s	11/15/2010	462.17	6.81	455.36
MW-124s	11/20/2010	462.17	3.79	458.38
MW-124s	4/11/2011	462.17	2.29	459.88
MW-124s	4/27/2011	462.17	2.89	459.28
MW-124s	6/2/2011	462.17	2.62	459.55
MW-124s	12/9/2011	462.17	3.82	458.35
MW-124s	3/27/2013	462.17	6.13	456.04
MW-124s	12/10/2017	462.17	6.48	455.69
MW-124s	12/14/2021	462.17	4.98	457.19
MW-124s	3/19/2023	462.17	3.49	458.68
MW-124s	7/23/2025	462.17	9.22	452.95

Table 2
Groundwater Elevation Data
Former Monarch Chemicals
Geneva, NY

Well	Date	Top of Casing (ft AMSL)	Depth to Water (ft TOC)	Potentiometric Elevation (ft AMSL)
MW-124d	8/15/2008	462.24	14.28	447.96
MW-124d	10/8/2008	462.24	13.79	448.45
MW-124d	11/17/2008	462.24	12.83	449.41
MW-124d	4/27/2009	462.24	13.86	448.38
MW-124d	8/3/2009	462.24	15.35	446.89
MW-124d	4/15/2010	462.24	17.62	444.62
MW-124d	8/9/2010	462.24	18.92	443.32
MW-124d	11/8/2010	462.24	18.64	443.60
MW-124d	11/9/2010	462.24	13.10	449.14
MW-124d	11/10/2010	462.24	13.14	449.10
MW-124d	11/11/2010	462.24	13.24	449.00
MW-124d	11/12/2010	462.24	13.26	448.98
MW-124d	11/13/2010	462.24	13.28	448.96
MW-124d	11/14/2010	462.24	13.31	448.93
MW-124d	11/15/2010	462.24	13.78	448.46
MW-124d	11/20/2010	462.24	15.18	447.06
MW-124d	4/11/2011	462.24	18.54	443.70
MW-124d	4/27/2011	462.24	17.35	444.89
MW-124d	6/2/2011	462.24	13.88	448.36
MW-124d	12/9/2011	462.24	12.88	449.36
MW-124d	3/27/2013	462.24	18.23	444.01
MW-124d	12/10/2017	462.24	13.40	448.84
MW-124d	12/14/2021	462.24	14.45	447.79
MW-124d	3/19/2023	462.24	12.59	449.65
MW-124d	7/23/2025	462.24	15.35	446.89

Notes:
AMSL - above mean sea level
TOC - top of casing
Data collected before 2025 by Pinnacle Engineering, Inc.

TABLE 3
SUMMARY OF REMAINING SOIL SAMPLE EXCEEDANCE
FORMER MONARCH CHEMICALS FACILITY
GENEVA, NEW YORK

	Criteria			
Sample Location	Part 375	Part 375	Part 375	GP-02
Sample Depth (feet bgs)	Protection of	Restricted	Restricted	14
Sample Date	Groundwater	Residential	Industrial	10/23/1996
Volatile Organic Compounds (ug/L)				
1,1,1-Trichloroethane	680	100000	1000000	ND (60)
1,1-Dichloroethane	270	19000	240000	**110**
Chloroform (Trichloromethane)	370	4800	180000	ND (60)
cis-1,2-Dichloroethene	190	8700	590000	ND (60)
Ethylbenzene	1000	32000	390000	ND (60)
Tetrachloroethene	1300	15000	81000	**6700**[(A)]
Toluene	700	100000	1000000	ND (60)
Trichloroethene	470	17000	54000	**60**

ABBREVIATIONS AND NOTES:
ug/Kg: micrograms per Kilogram

-: Not Analyzed
ND (2.5): Not detected, number in parentheses is the laboratory reporting limit
*: Not detected data is rejected due to laboratory analysis out of hold time and insufficient presence of preservative.

- Soil analytical results are compared to 6 NYCRR Part 375-6.8 (b) effective December 31, 2025
[(A)]Protection of Groundwater Soil Cleanup Objectives
[(B)]Restricted Residential Soil Cleanup Objectives
[(C)]Restricted Industrial Cleanup Objectives
- Bold indicates concentration detected above the laboratory reporting limit
- Gray shaded values indicate an exceedance of the TOGS criteria.

TABLE 4
SUMMARY OF WATER QUALITY DATA 2020-2025
FORMER MONARCH CHEMICALS FACILITY
GENEVA, NEW YORK

	Criteria	MW-114			MW-115							MW-117			
Location Name		MW-114	MW-114	MW-114	MW-115	MW-115	MW-115	MW-115	MW-115	MW-115	MW-115 *	MW-117	MW-117	MW-117	MW-117
Sample Name	New York TOGS 111 Ambient Water Quality Standards	MW-114-20200304	MW-114-20211214	MW-114-20230318	MW-115d-20200304	MW-115-20200305	MW-115-20211214	MW-115d-20211214	MW-115-20230320	MW-115d-20230320	MW115-073125-1720	MW-117-20200304	MW-117-20211215	MW-117-20230318	MW117-073125-1110
Sample Date		03/04/2020	12/14/2021	03/18/2023	03/04/2020	03/05/2020	12/14/2021	12/14/2021	03/20/2023	03/20/2023	07/31/2025	03/04/2020	12/15/2021	03/18/2023	07/31/2025
Lab Sample ID		20200304	20211214	240-182494-1	20200304	20200305	20211214	20211214	240-182494-2	240-182494-3	L2548555-04	20200304	20211215	240-182494-5	L2548555-01
Volatile Organic Compounds (ug/L)															
1,1,1-Trichloroethane	5	ND (1)	ND (1)	**2.2**	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	2.5 R	ND (1)	ND (1)	ND (1)	ND (2.5)
1,1,2,2-Tetrachloroethane	5	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	0.5 R	ND (1)	ND (1)	ND (1)	ND (0.5)
1,1,2-Trichloroethane	1	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	1.5 R	ND (1)	ND (1)	ND (1)	ND (1.5)
1,1-Dichloroethane	5	ND (1)	**1.8**	ND (1)	**0.74 J**	**3.1 J**	**2.6 J**	**0.79 J**	**3.8 J**	**0.78 J**	**1.9 J**	ND (1)	**0.39 J**	ND (1)	ND (2.5)
1,1-Dichloroethene	5	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	0.5 R	ND (1)	ND (1)	ND (1)	ND (0.5)
1,2,3-Trichlorobenzene	5	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
1,2,4-Trichlorobenzene	5	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
1,2-Dibromo-3-chloropropane (DBCP)	0.04	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
1,2-Dibromoethane (Ethylene Dibromide)	0.0006	-	-	-	-	-	-	-	-	-	2 R	-	-	-	ND (2)
1,2-Dichlorobenzene	3	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
1,2-Dichloroethane	0.6	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	0.5 R	ND (1)	ND (1)	ND (1)	ND (0.5)
1,2-Dichloropropane	1	-	-	-	-	-	-	-	-	-	1 R	-	-	-	ND (1)
1,3-Dichlorobenzene	3	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
1,4-Dichlorobenzene	3	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
1,4-Dioxane	0.35	-	-	-	-	-	-	-	-	-	250 R	-	-	-	250 R
2-Butanone (Methyl Ethyl Ketone)	50	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	5 R	ND (10)	ND (10)	ND (10)	ND (5)
2-Hexanone (Methyl Butyl Ketone)	50	ND (5)	ND (5)	ND (5)	ND (5)	ND (20)	ND (5)	ND (5)	ND (5)	ND (5)	5 R	ND (5)	ND (5)	ND (5)	ND (5)
4-Methyl-2-Pentanone (Methyl Isobutyl Ketone)	NA	ND (5)	ND (5)	ND (10)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	5 R	ND (5)	ND (5)	ND (5)	ND (5)
Acetone	50	ND (10)	ND (10)	ND (10)	ND (10)	ND (40)	ND (10)	ND (10)	ND (10)	ND (10)	13 R	**10**	ND (10)	ND (10)	ND (6.3)
Benzene	1	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	0.5 R	ND (1)	ND (1)	ND (1)	**0.49 J**
Bromodichloromethane	50	-	-	-	-	-	-	-	-	-	0.5 R	-	-	-	ND (0.5)
Bromoform	50	-	-	-	-	-	-	-	-	-	2 R	-	-	-	ND (2)
Bromomethane (Methyl Bromide)	5	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
Carbon disulfide	60	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	5 R	ND (1)	ND (1)	ND (1)	ND (5)
Carbon tetrachloride	5	-	-	-	-	-	-	-	-	-	0.5 R	-	-	-	ND (0.5)
Chlorobenzene	5	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	2.5 R	ND (1)	ND (1)	ND (1)	ND (2.5)
Chlorobromomethane	5	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
Chloroethane	5	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	2.5 R	ND (1)	ND (1)	ND (1)	ND (2.5)
Chloroform (Trichloromethane)	7	ND (1)	ND (1)	ND (1)	ND (1)	ND (1.4)	ND (1)	ND (1)	ND (1)	ND (1)	2.5 R	ND (1)	ND (1)	ND (1)	ND (2.5)
Chloromethane (Methyl Chloride)	5	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	2.5 R	ND (1)	ND (1)	ND (1)	ND (2.5)
cis-1,2-Dichloroethene	5	ND (1)	**4.1**	**0.49 J**	ND (1)	**3.2 J**	**7.9**	ND (1)	**3.4**	ND (1)	**4.3 J**	ND (1)	**1.4**	ND (1)	**4.8**
cis-1,3-Dichloropropene	0.4	-	-	-	-	-	-	-	-	-	0.5 R	-	-	-	ND (0.5)
Cyclohexane	NA	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	10 R	ND (1)	ND (1)	ND (1)	**1.3 J**
Dibromochloromethane	50	-	-	-	-	-	-	-	-	-	0.5 R	-	-	-	ND (0.5)
Dichlorodifluoromethane (CFC-12)	5	-	-	-	-	-	-	-	-	-	5 R	-	-	-	ND (5)
Ethylbenzene	5	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	2.5 R	ND (1)	ND (1)	ND (1)	**5.1**
Isopropylbenzene (Cumene)	5	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	2.5 R	ND (1)	ND (1)	ND (1)	**2.6**
m,p-Xylenes	5	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	**0.71 J**
Methyl acetate	NA	ND (1)	ND (1)	ND (10)	ND (2.5)	ND (10)	ND (2.5)	ND (2.5)	ND (10)	ND (10)	2 R	ND (2.5)	ND (2.5)	ND (10)	ND (2)
Methyl Tert Butyl Ether (MTBE)	10	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
Methylcyclohexane	NA	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	10 R	ND (1)	ND (1)	ND (1)	**1.6 J**
Methylene chloride (Dichloromethane)	5	ND (1)	ND (1)	ND (5)	ND (1)	ND (4)	ND (1)	ND (1)	ND (5)	ND (5)	2.5 R	ND (1)	ND (1)	ND (5)	ND (2.5)
o-Xylene	5	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
Styrene	5	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
Tetrachloroethene	5	**2.1**	ND (1)	**6.5**	ND (1)	**4.4**	**1.2**	ND (1)	**2.8**	ND (1)	**2.4 J**	**0.91 J**	ND (1)	**2**	ND (0.5)
Toluene	5	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	2.5 R	ND (1)	ND (1)	ND (1)	ND (2.5)
trans-1,2-Dichloroethene	5	ND (1)	ND (1)	ND (1)	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	2.5 R	ND (1)	ND (1)	ND (1)	ND (2.5)
trans-1,3-Dichloropropene	0.4	-	-	-	-	-	-	-	-	-	0.5 R	-	-	-	ND (0.5)
Trichloroethene	5	**1.3**	**0.8 J**	**6.5**	ND (1)	ND (4)	**1.2**	ND (1)	**0.6 J**	ND (1)	**0.57 J**	ND (1)	**0.53 J**	ND (1)	ND (0.5)
Trichlorofluoromethane (CFC-11)	5	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
Trifluorotrichloroethane (Freon 113)	5	-	-	-	-	-	-	-	-	-	2.5 R	-	-	-	ND (2.5)
Vinyl chloride	2	ND (1)	ND (1)	ND (1)	ND (1)	ND (3.6)	ND (1)	ND (1)	ND (1)	ND (1)	**1.3 J**	ND (1)	**0.9 J**	ND (1)	**0.62 J**
Xylene (Total)	5	ND (2)	ND (2)	ND (2)	ND (2)	ND (8)	ND (2)	ND (2)	ND (2)	ND (2)	-	ND (2)	ND (2)	ND (2)	-

ABBREVIATIONS AND NOTES:
ug/L: micrograms per liter

-: Not Analyzed
J: Estimated result
R: Result rejected as unusable
NA: Not Applicable
ND (2.5): Not detected, number in parentheses is the laboratory reporting limit
*: Not detected data is rejected due to laboratory analysis out of hold time and 'insufficient presence of preservative.

- For test methods used, see the laboratory data sheets.
- Groundwater analytical results are compared to NY-AWQS: NYSDEC Technical and Operational Guidance Series (TOGS) 1.1.1 Ambient Water Quality Standards and Guidance Values (SGVs) for Class GA Water.
- Bold values indicate an exceedance of the TOGS criteria.
'- Gray shaded values indicate an exceedance of the TOGS criteria.

TABLE 4
SUMMARY OF WATER QUALITY DATA 2020-2025
FORMER MONARCH CHEMICALS FACILITY
GENEVA, NEW YORK

	Criteria	MW-118					MW-122			MW-124d			
Location Name		MW-118	MW-118	MW-118	MW-118	MW-118 (dup)	MW-122	MW-122	MW-122	MW-124d	MW-124d	MW-124d	MW-124d
Sample Name	New York TOGS 111 Ambient Water	MW-118-20200305	MW-118-20211215	MW-118-20230318	MW118-073125-1600	020811-073125-0002	MW-122-20200305	MW-122-20211216	MW-122-20230322	MW-124d-20200306	MW-124d-20211214	MW-124d-20230319	MW124D-073125-1545
Sample Date	Quality Standards	03/05/2020	12/15/2021	03/18/2023	07/31/2025	07/31/2025	03/05/2020	12/16/2021	03/22/2023	03/06/2020	12/14/2021	03/19/2023	07/31/2025
Lab Sample ID		20200305	20211215	240-182494-6	L2548555-03	L2548555-05	20200305	20211216	240-182494-8	20200306	20211214	240-182494-10	L2548555-02
Volatile Organic Compounds (ug/L)													
1,1,1-Trichloroethane	5	ND (5)	ND (5)	ND (2)	ND (2.5)	ND (2.5)	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (2.5)
1,1,2,2-Tetrachloroethane	5	ND (2)	ND (2)	ND (2)	ND (0.5)	ND (0.5)	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (0.5)
1,1,2-Trichloroethane	1	ND (5)	ND (5)	ND (2)	ND (1.5)	ND (1.5)	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (1.5)
1,1-Dichloroethane	5	**140**	**82**	**100**	**48**	**53**	**19**	**19**	**12**	ND (1)	**12**	ND (3.3)	**6.3 J**
1,1-Dichloroethene	5	**2.4 J**	ND (5)	**1.6**	**0.18 J**	**0.2 J**	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (0.5)
1,2,3-Trichlorobenzene	5	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
1,2,4-Trichlorobenzene	5	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
1,2-Dibromo-3-chloropropane (DBCP)	0.04	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
1,2-Dibromoethane (Ethylene Dibromide)	0.0006	-	-	-	ND (2)	ND (2)	-	-	-	-	-	-	ND (2)
1,2-Dichlorobenzene	3	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
1,2-Dichloroethane	0.6	ND (5)	ND (5)	ND (2)	**0.14 J**	**0.14 J**	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (0.5)
1,2-Dichloropropane	1	-	-	-	ND (1)	ND (1)	-	-	-	-	-	-	ND (1)
1,3-Dichlorobenzene	3	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
1,4-Dichlorobenzene	3	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
1,4-Dioxane	0.35	-	-	-	250 R	250 R	-	-	-	-	-	-	250 R
2-Butanone (Methyl Ethyl Ketone)	50	ND (50)	ND (50)	ND (20)	**3.5 J**	**3.8 J**	ND (10)	ND (10)	ND (10)	ND (10)	ND (50)	ND (33)	ND (5)
2-Hexanone (Methyl Butyl Ketone)	50	ND (10)	ND (10)	ND (20)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (25)	ND (33)	ND (5)
4-Methyl-2-Pentanone (Methyl Isobutyl Ketone)	NA	ND (25)	ND (25)	ND (20)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (25)	ND (33)	ND (5)
Acetone	50	ND (50)	ND (50)	ND (20)	ND (24)	**28 J+**	**130**	ND (10)	ND (10)	ND (10)	ND (50)	ND (33)	ND (13)
Benzene	1	ND (5)	ND (5)	ND (2)	**0.23 J**	**0.28 J**	ND (1)	**0.78 J**	**0.52 J**	ND (1)	ND (5)	ND (3.3)	**0.3 J**
Bromodichloromethane	50	-	-	-	ND (0.5)	ND (0.5)	-	-	-	-	-	-	ND (0.5)
Bromoform	50	-	-	-	ND (2)	ND (2)	-	-	-	-	-	-	ND (2)
Bromomethane (Methyl Bromide)	5	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5) J
Carbon disulfide	60	ND (5)	ND (5)	ND (2)	ND (5)	ND (5)	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (5)
Carbon tetrachloride	5	-	-	-	ND (0.5)	ND (0.5)	-	-	-	-	-	-	ND (0.5)
Chlorobenzene	5	ND (5)	ND (5)	ND (2)	ND (2.5)	ND (2.5)	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (2.5)
Chlorobromomethane	5	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
Chloroethane	5	**17**	**19**	**8.7**	**15**	**18**	**12**	**13**	**2.9**	**260**	**150**	**140**	**100 J**
Chloroform (Trichloromethane)	7	ND (5)	ND (5)	ND (2)	ND (2.5)	ND (2.5)	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (2.5)
Chloromethane (Methyl Chloride)	5	ND (5)	ND (5)	ND (2)	ND (2.5)	ND (2.5)	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (2.5)
cis-1,2-Dichloroethene	5	**170**	**100**	**100**	**17**	**19**	**11**	**13**	**7.1**	ND (1)	ND (5)	ND (3.3)	**0.9 J**
cis-1,3-Dichloropropene	0.4	-	-	-	ND (0.5)	ND (0.5)	-	-	-	-	-	-	ND (0.5)
Cyclohexane	NA	ND (5)	ND (5)	ND (2)	**1.3 J**	**1.3 J**	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (10)
Dibromochloromethane	50	-	-	-	ND (0.5)	ND (0.5)	-	-	-	-	-	-	ND (0.5)
Dichlorodifluoromethane (CFC-12)	5	-	-	-	ND (5)	ND (5)	-	-	-	-	-	-	ND (5)
Ethylbenzene	5	ND (5)	ND (5)	ND (2)	ND (2.5)	ND (2.5)	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (2.5)
Isopropylbenzene (Cumene)	5	ND (5)	ND (5)	ND (2)	ND (2.5)	ND (2.5)	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (2.5)
m,p-Xylenes	5	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
Methyl acetate	NA	ND (13)	ND (13)	ND (10)	ND (2)	ND (2)	ND (1)	ND (1)	ND (10)	ND (2.5)	ND (13)	ND (33)	ND (2)
Methyl Tert Butyl Ether (MTBE)	10	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
Methylcyclohexane	NA	ND (5)	ND (5)	ND (2)	**1.4 J**	**1.4 J**	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (10)
Methylene chloride (Dichloromethane)	5	ND (5)	ND (5)	ND (2)	ND (2.5)	ND (2.5)	ND (1)	ND (1)	ND (5)	ND (1)	ND (5)	ND (3.3)	ND (2.5)
o-Xylene	5	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
Styrene	5	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
Tetrachloroethene	5	ND (1.8)	ND (5)	ND (2)	**0.9**	**1**	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (0.5)
Toluene	5	ND (5)	ND (5)	ND (2)	**0.74 J**	**0.88 J**	ND (1)	ND (1)	ND (1)	ND (1)	ND (5)	ND (3.3)	ND (2.5)
trans-1,2-Dichloroethene	5	ND (5)	ND (5)	**1.1 J**	**1.4 J**	**1.7 J**	**0.98 J**	**1.9**	**1**	ND (1)	ND (5)	ND (3.3)	ND (2.5)
trans-1,3-Dichloropropene	0.4	-	-	-	ND (0.5)	ND (0.5)	-	-	-	-	-	-	ND (0.5)
Trichloroethene	5	**23**	ND (5)	**15**	**3**	**3.3**	**0.97 J**	**2.9**	**0.49 J**	ND (1)	ND (5)	ND (3.3)	ND (0.5)
Trichlorofluoromethane (CFC-11)	5	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
Trifluorotrichloroethane (Freon 113)	5	-	-	-	ND (2.5)	ND (2.5)	-	-	-	-	-	-	ND (2.5)
Vinyl chloride	2	**230**	**67**	**72**	**14**	**16**	**1.5**	**1.6**	**1.9**	ND (1)	ND (5)	ND (3.3)	**1.8 J**
Xylene (Total)	5	ND (10)	ND (10)	ND (4)	-	-	ND (2)	ND (2)	ND (2)	ND (2)	ND (10)	ND (6.7)	-

ABBREVIATIONS AND NOTES:
ug/L: micrograms per liter

-: Not Analyzed
J: Estimated result
R: Result rejected as unusable
NA: Not Applicable
ND (2.5): Not detected, number in parentheses is the laboratory reporting limit
*: Not detected data is rejected due to laboratory analysis out of hold time and 'insufficient presence of preservative.

- For test methods used, see the laboratory data sheets.
- Groundwater analytical results are compared to NY-AWQS: NYSDEC Technical and Operational Guidance Series (TOGS) 1.1.1 Ambient Water Quality Standards and Guidance Values (SGVs) for Class GA Water.
- Bold values indicate an exceedance of the TOGS criteria.
'- Gray shaded values indicate an exceedance of the TOGS criteria.

TABLE 5
Post Remediation Sampling Requirements and Schedule
(In Text)

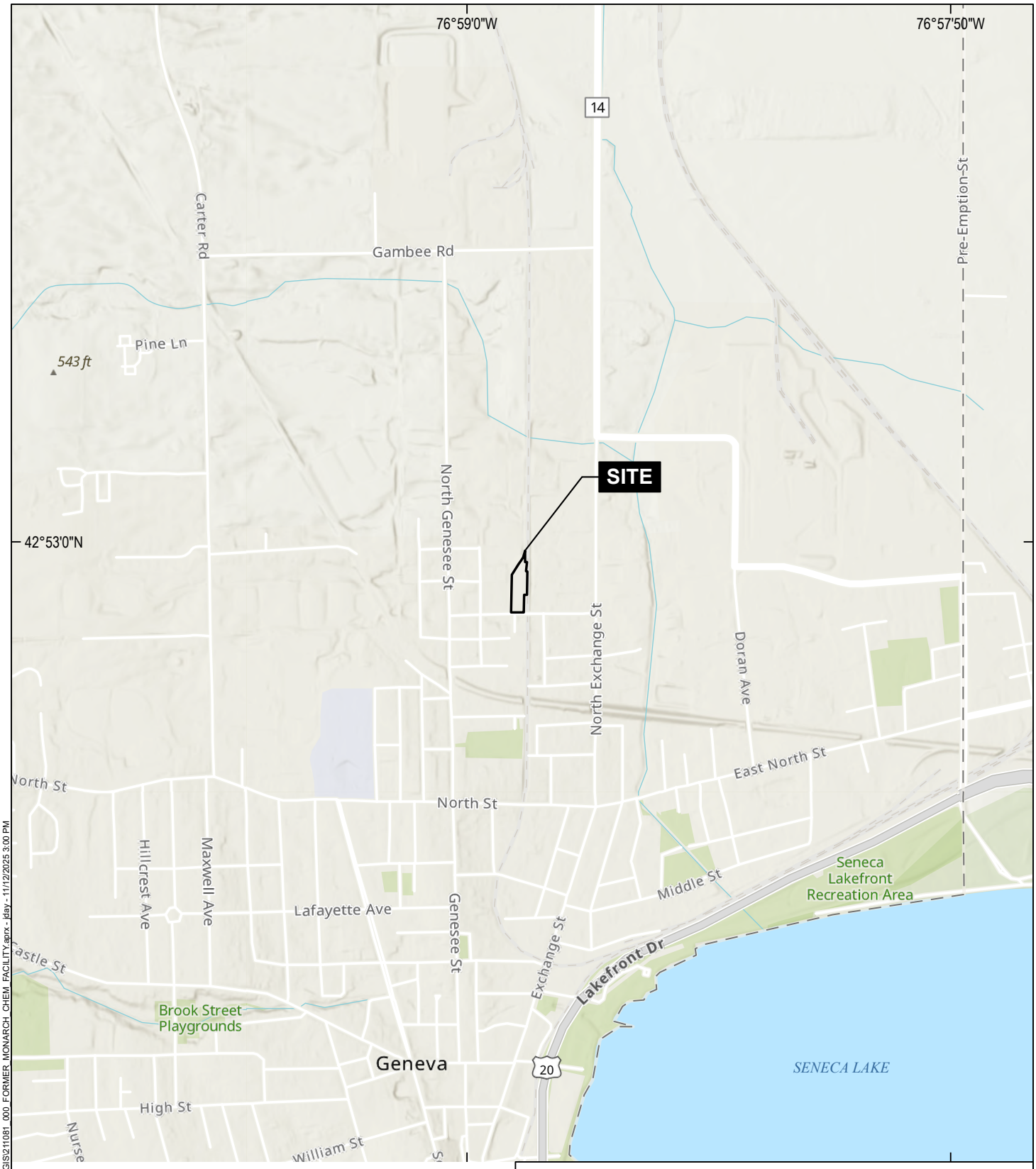
Table 6 - Monitoring Well Construction
Details Former Monarch Chemicals Facility
Geneva, New York

	Ground Surface Elevation (ft AMSL)	Top of Casing Elevation (ft AMSL)	Depth of Borehole (ft below TOC)	Well Diameter (in)	Screen Length (ft)	Total Well Depth (ft below TOC)	Measured Well Depth (ft below TOC)	Depth to Top of Screen (ft below TOC)	Top of Screen Elevation (ft AMSL)	Depth to Bottom of Screen (ft below TOC)	Bottom of Installed Screen Elevation (ft AMSL)	Bottom of Measured Screen Elevation (ft AMSL)	Date Installed
MW--114	462.0	462.03	16	2	10	14.0	13.51	4.0	458.03	14.0	448.03	448.52	14---Jul---05
MW--115	462.3	462.25	16	2	10	14.0	13.11	4.0	458.25	14.0	448.25	449.14	12---Jul---05
MW--115d	462.1	462.06	24	2	5	24.0	24.00	19.0	443.06	24.0	438.06	438.06	12---Nov---10
MW--116	462.4	462.44	16	2	10	13.9	13.48	3.9	458.54	13.9	448.54	448.96	12---Jul---05
MW--117	463.4	463.44	16	2	10	13.7	13.50	3.7	459.74	13.7	449.74	449.94	12---Jul---05
MW--118	462.4	462.40	16	2	10	14.0	13.28	4.0	458.40	14.0	448.40	449.12	12---Jul---05
MW--119	462.7	462.68	16	2	10	14.0	13.56	4.0	458.68	14.0	448.68	449.12	11---Jul---05
MW--120	462.1	462.12	16	2	10	14.0	13.55	4.0	458.12	14.0	448.12	448.57	11---Jul---05
MW--121	461.8	461.77	16	2	10	14.0	13.97	4.0	457.77	14.0	447.77	447.80	13---Jul---05
MW--122	461.7	461.65	16	2	10	14.0	13.55	4.0	457.65	14.0	447.65	448.10	13---Jul---05
MW--124d	462.3	462.33	24	2	5	21.0	21.00	16.0	446.33	21.0	441.33	441.33	14---Aug---08
MW--124s	462.3	462.25	14	2	10	12.5	12.50	2.5	459.75	12.5	449.75	449.75	14---Aug---08

Notes:
TOC - Top of casing
AMSL - Above mean sea level

TABLE 8
Schedule of Interim Monitoring/Inspections Reports
(In Text)

FIGURES



MAP SOURCE: ESRI
SITE COORDINATES: 76°58'53"W, 42°52'55"N

HALEY ALDRICH

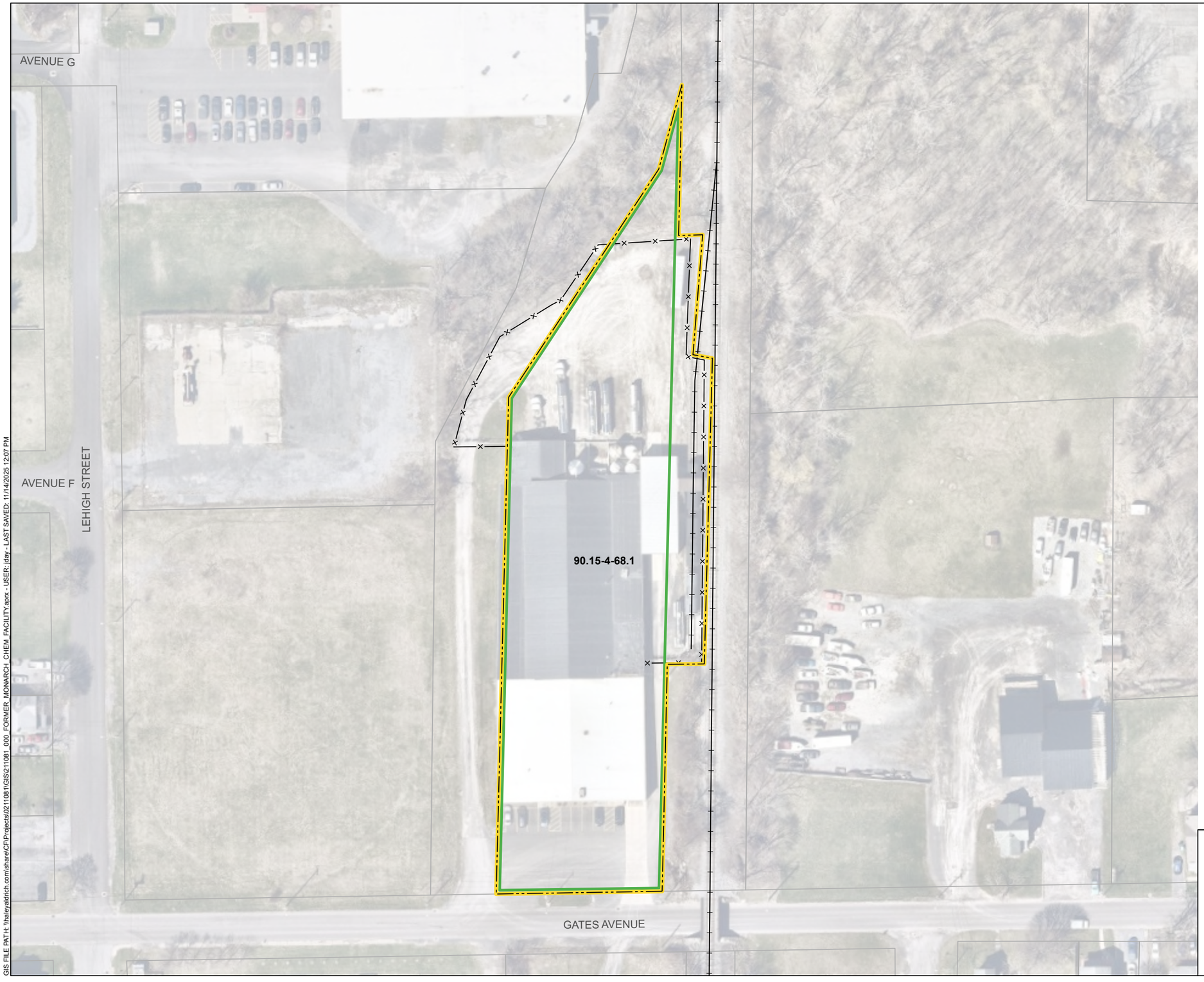
FORMER MONARCH CHEMICALS FACILITY
61 GATES AVENUE
GENEVA, NEW YORK

PROJECT LOCUS

APPROXIMATE SCALE: 1 IN = 2000 FT
NOVEMBER 2025

FIGURE 1

GIS: \\haleyaldrich.com\share\CF\Projects\021108\1\GIS\211081_000_FORMER_MONARCH_CHEM_FACILITY.aprx - jday - 11/12/2025 3:00 PM



LEGEND

- FENCE
- RAILROAD
- VOLUNTARY CLEANUP PROGRAM SITE #V0019-8
- SITE BOUNDARY
- PARCEL BOUNDARY

NOTES

1. ALL LOCATIONS AND DIMENSIONS ARE APPROXIMATE.
2. ASSESSOR PARCEL DATA SOURCE: NEW YORK STATE GIS CLEARNING HOUSE
3. AERIAL IMAGERY SOURCE: NEARMAP, APRIL 17, 2025

N

0 80 160

SCALE IN FEET

HALEY ALDRICH

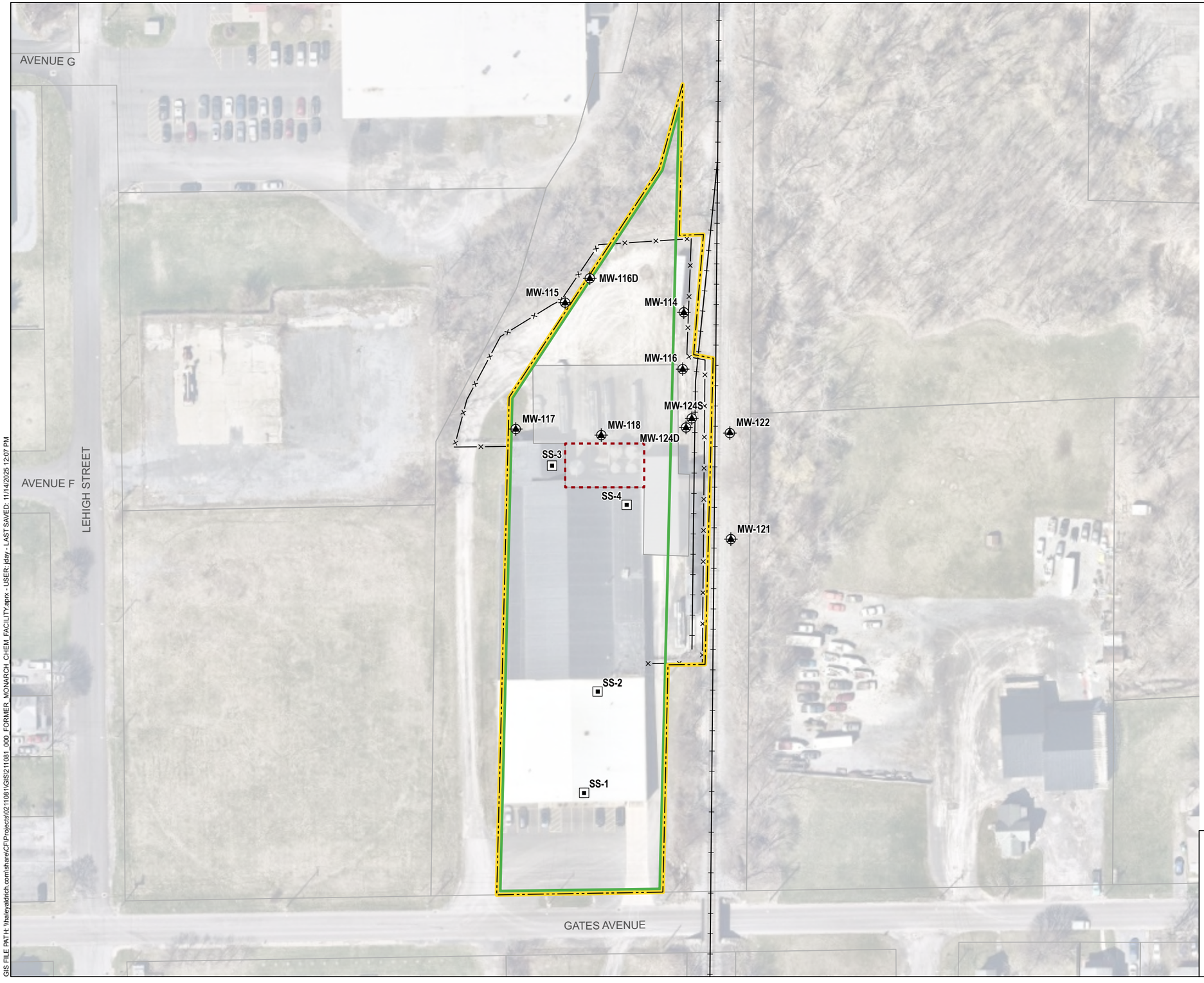
FORMER MONARCH CHEMICALS FACILITY
61 GATES AVENUE
GENEVA, NEW YORK

PROJECT BOUNDARY MAP

NOVEMBER 2025

FIGURE 2

GIS FILE PATH: \\haleyaldrich.com\share\CP\Projects\0211081\GIS\211081_000_FORMER_MONARCH_CHEM_FACILITY.aprx - USER: jday - LAST SAVED: 11/14/2025 12:07 PM



LEGEND

- MONITORING WELL
- SUB-SLAB/INDOOR AIR SVI SAMPLE
- FENCE
- RAILROAD
- CONCRETE PAD
- AST CONTAINMENT AREA
- VOLUNTARY CLEANUP PROGRAM SITE #V0019-8
- SITE BOUNDARY
- PARCEL BOUNDARY

NOTES

1. ALL LOCATIONS AND DIMENSIONS ARE APPROXIMATE.
2. ASSESSOR PARCEL DATA SOURCE: NEW YORK STATE GIS CLEARNING HOUSE
3. DEFINITIONS
SVI = SOIL VAPOR INTRUSION
AST = ABOVEGROUND STORAGE TANK
4. AERIAL IMAGERY SOURCE: NEARMAP, APRIL 17, 2025

N

0 80 160
SCALE IN FEET

HALEY ALDRICH

FORMER MONARCH CHEMICALS FACILITY
61 GATES AVENUE
GENEVA, NEW YORK

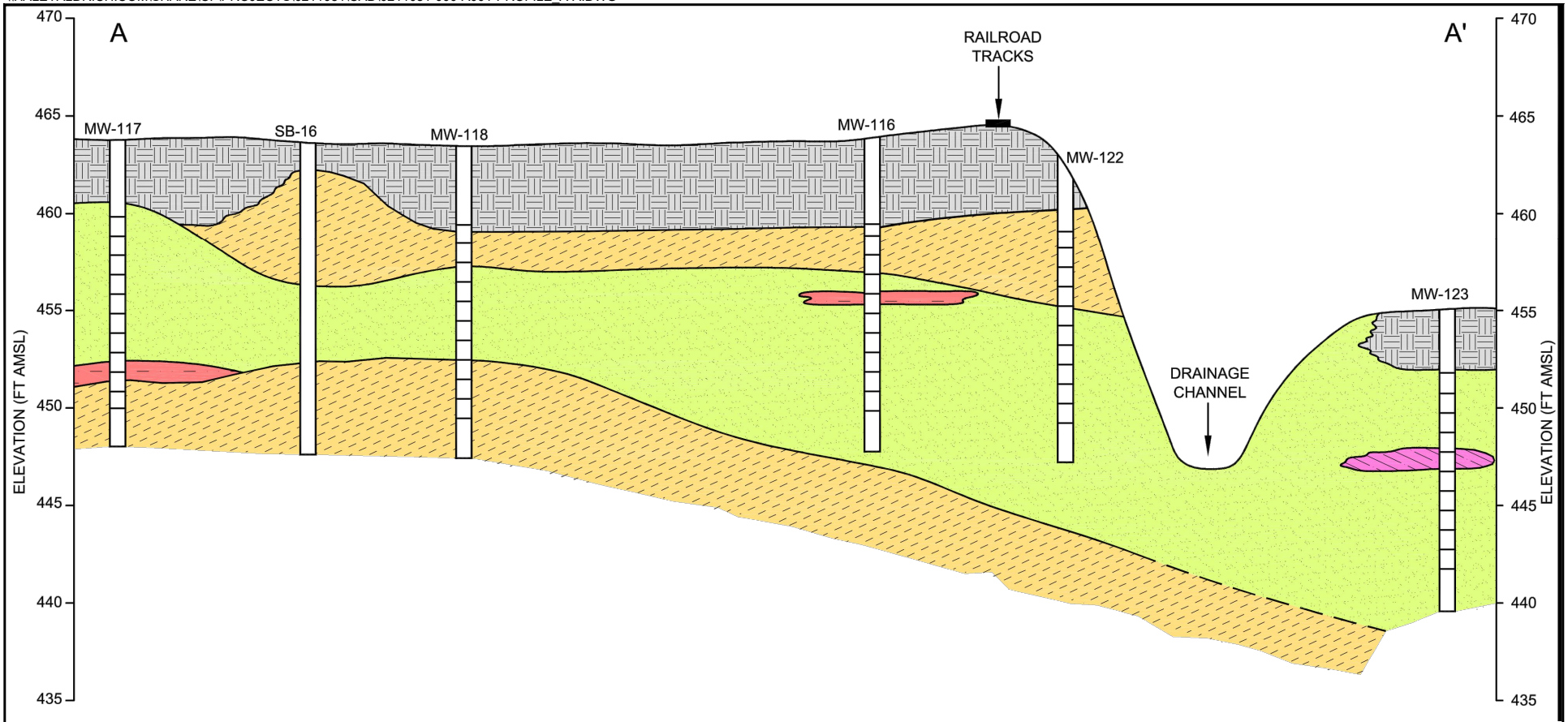
EXPLORATION PLAN

NOVEMBER 2025

FIGURE 3

GIS FILE PATH: \\haleyaldrich.com\share\CFI\Projects\0211081\GIS\211081_000_FORMER_MONARCH_CHEM_FACILITY.aprx - USER: rdaly - LAST SAVED: 11/14/2025 12:07 PM

Saved by: DTE Printed: 2/26/2026 12:47 PM Sheet: FIG 4
\\HALEYALDRICH.COM\SHARE\CF\PROJECTS\0211081\CAD\0211081-000-A001-PROFILE_A-A.DWG



LEGEND

SCREENED INTERVAL

FILL

SILT & CLAY

SAND

CLAY

SILTY SAND

(IN FEET) VERTICAL SCALE 8

VE=4X

20 HORIZONTAL SCALE (IN FEET)

NOTE

1. FIGURE 4 OF THIS SITE MANAGEMENT PLAN HAS BEEN SOURCED DIRECTLY FROM THE "SITE MANAGEMENT PLAN FORMER MONARCH CHEMICAL FACILITY, 61 GATES AVENUE, GENEVA (ONTARIO), NEW YORK 14456" PREPARED BY PINNACLE ENGINEERING, DATED SEPTEMBER 27, 2017.

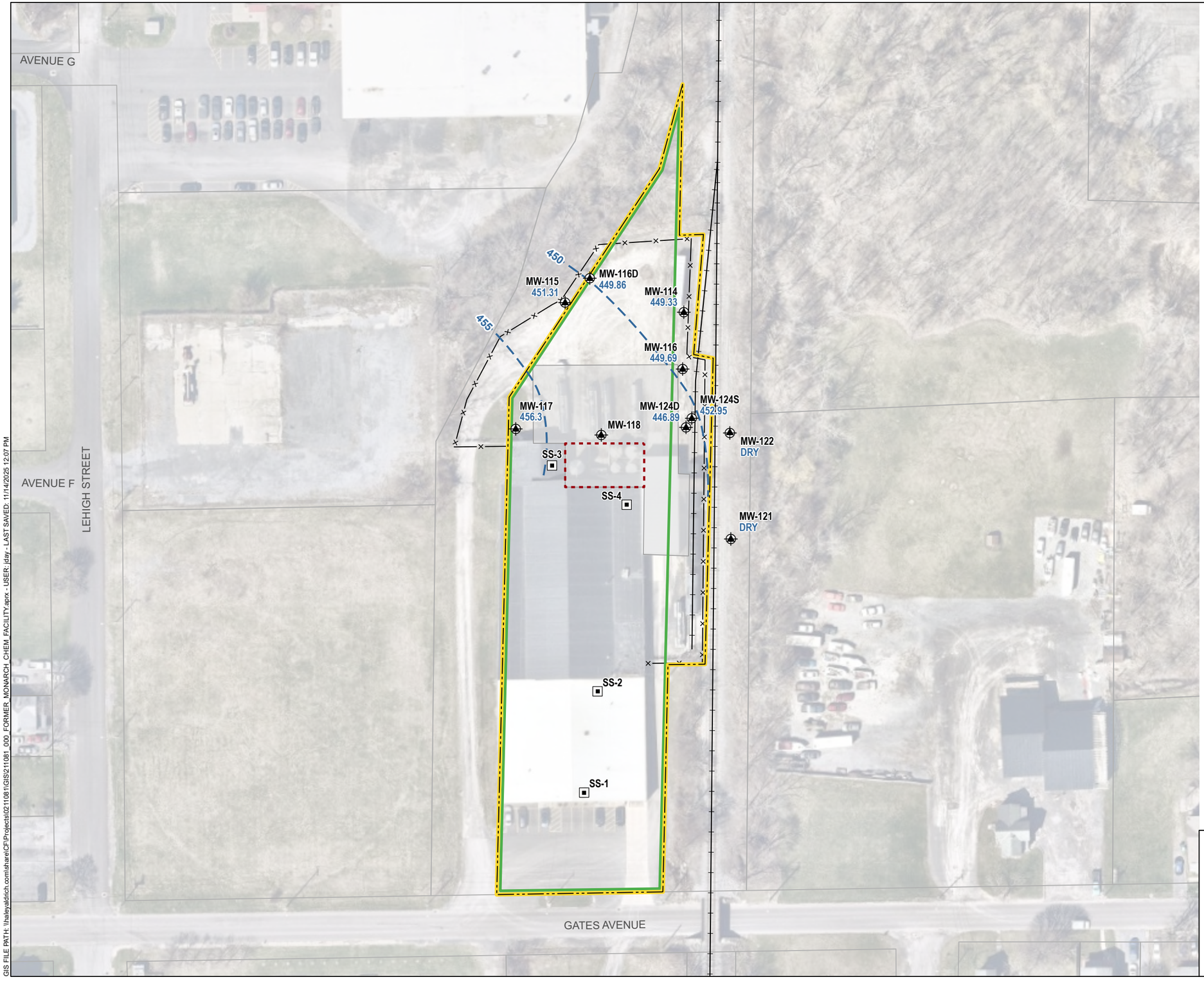
HALEY ALDRICH

FORMER MONARCH CHEMICALS COMPANY
61 GATES AVENUE
GENEVA, NEW YORK

CROSS SECTION A – A' GEOLOGIC PROFILE

SCALE: AS SHOWN
FEBRUARY 2026

FIGURE 4



LEGEND

- MONITORING WELL
- SUB-SLAB/INDOOR AIR SVI SAMPLE
- FENCE
- RAILROAD
- INFERRED GROUNDWATER ELEVATION CONTOUR, 5-FT INTERVAL
- CONCRETE PAD
- AST CONTAINMENT AREA
- VOLUNTARY CLEANUP PROGRAM SITE #V0019-8
- SITE BOUNDARY
- PARCEL BOUNDARY

NOTES

1. ALL LOCATIONS AND DIMENSIONS ARE APPROXIMATE.

2. ASSESSOR PARCEL DATA SOURCE: NEW YORK STATE GIS CLEARNING HOUSE

3. DEFINITIONS
SVI = SOIL VAPOR INTRUSION
AST = ABOVEGROUND STORAGE TANK

4. AERIAL IMAGERY SOURCE: NEARMAP, APRIL 17, 2025

N

0 80 160
SCALE IN FEET

HALEY ALDRICH

FORMER MONARCH CHEMICALS FACILITY
61 GATES AVENUE
GENEVA, NEW YORK

GROUNDWATER CONTOUR MAP JULY 2025

NOVEMBER 2025

FIGURE 5

GIS FILE PATH: \\haleyaldrich.com\share\CFLProjects\0211081\GIS\211081_000_FORMER_MONARCH_CHEM_FACILITY.aprx - USER: rday - LAST SAVED: 11/14/2025 12:07 PM

FIGURE 6
Remaining Soil Exceedances
(In Text)

Location Name	New York TOGS	MW-115						
Sample Name	111 Ambient	MW-115-20200304	MW-115-20200305	MW-115-20211214	MW-115d-20211214	MW-115-20230320	MW-115d-20230320	MW-115-073125-1720
Sample Date	Water Quality	03/04/2020	03/05/2020	12/14/2021	12/14/2021	03/20/2023	03/20/2023	07/31/2025
Lab Sample ID	Standards	20200304	20200305	20211214	20211214	240-182494-2	240-182494-3	L2548555-04
Volatile Organic Compounds (ug/L)								
1,1,1-Trichloroethane	5	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	2.5 R
1,1-Dichloroethane	5	**0.74 J**	**3.1 J**	**2.6 J**	**0.79 J**	**3.8 J**	**0.78 J**	**1.9 J**
Chloroethane	5	ND (1)	ND (4)	ND (1)	ND (1)	ND (1)	ND (1)	2.5 R
cis-1,2-Dichloroethene	5	ND (1)	**3.2 J**	**7.9**	ND (1)	**3.4**	ND (1)	**4.3 J**
Tetrachloroethene	5	ND (1)	**4.4**	**1.2**	ND (1)	**2.8**	ND (1)	**2.4 J**
Trichloroethene	5	ND (1)	ND (4)	**1.2**	ND (1)	**0.6 J**	ND (1)	**0.57 J**
Vinyl chloride	2	ND (1)	ND (3.8)	ND (1)	ND (1)	ND (1)	ND (1)	**1.3 J**

Location Name	New York TOGS	MW-114		
Sample Name	111 Ambient	MW-114-20200304	MW-114-20211214	MW-114-20230318
Sample Date	Water Quality	03/04/2020	12/14/2021	03/18/2023
Lab Sample ID	Standards	20200304	20211214	240-182494-1
Volatile Organic Compounds (ug/L)				
1,1,1-Trichloroethane	5	ND (1)	ND (1)	**2.2**
1,1-Dichloroethane	5	ND (1)	**1.8**	ND (1)
Chloroethane	5	ND (1)	ND (1)	ND (1)
cis-1,2-Dichloroethene	5	ND (1)	**4.1**	**0.49 J**
Tetrachloroethene	5	**2.1**	ND (1)	**6.5**
Trichloroethene	5	**1.3**	**0.8 J**	**6.5**
Vinyl chloride	2	ND (1)	ND (1)	ND (1)

Location Name	New York TOGS	MW-122		
Sample Name	111 Ambient	MW-122-20200305	MW-122-20211216	MW-122-20230322
Sample Date	Water Quality	03/05/2020	12/16/2021	03/22/2023
Lab Sample ID	Standards	20200305	20211216	240-182494-8
Volatile Organic Compounds (ug/L)				
1,1,1-Trichloroethane	5	ND (1)	ND (1)	ND (1)
1,1-Dichloroethane	5	**19**	**19**	**12**
Chloroethane	5	**12**	**13**	**2.9**
cis-1,2-Dichloroethene	5	**11**	**13**	**7.1**
Tetrachloroethene	5	ND (1)	ND (1)	ND (1)
Trichloroethene	5	**0.97 J**	**2.9**	**0.49 J**
Vinyl chloride	2	**1.5**	**1.6**	**1.9**

Location Name	New York TOGS	MW-117			
Sample Name	111 Ambient	MW-117-20200304	MW-117-20211215	MW-117-20230318	MW117-073125-1110
Sample Date	Water Quality	03/04/2020	12/15/2021	03/18/2023	07/31/2025
Lab Sample ID	Standards	20200304	20211215	240-182494-5	L2548555-01
Volatile Organic Compounds (ug/L)					
1,1,1-Trichloroethane	5	ND (1)	ND (1)	ND (1)	ND (2.5)
1,1-Dichloroethane	5	ND (1)	**0.39 J**	ND (1)	ND (2.5)
Chloroethane	5	ND (1)	ND (1)	ND (1)	ND (2.5)
cis-1,2-Dichloroethene	5	ND (1)	**1.4**	ND (1)	**4.8**
Tetrachloroethene	5	**0.91 J**	ND (1)	**2**	ND (0.5)
Trichloroethene	5	ND (1)	**0.53 J**	ND (1)	ND (0.5)
Vinyl chloride	2	ND (1)	**0.9 J**	ND (1)	**0.62 J**

Location Name	New York TOGS	MW-118				
Sample Name	111 Ambient	MW-118-20200305	MW-118-20211215	MW-118-20230318	MW-118-073125-1600	020811-073125-0002
Sample Date	Water Quality	03/05/2020	12/15/2021	03/18/2023	07/31/2025	07/31/2025
Lab Sample ID	Standards	20200305	20211215	240-182494-6	L2548555-03	L2548555-05 (duplicate)
Volatile Organic Compounds (ug/L)						
1,1,1-Trichloroethane	5	ND (5)	ND (5)	ND (2)	ND (2.5)	ND (2.5)
1,1-Dichloroethane	5	**140**	**82**	**100**	**48**	**53**
Chloroethane	5	**17**	**19**	**8.7**	**15**	**18**
cis-1,2-Dichloroethene	5	**170**	**100**	**100**	**17**	**19**
Tetrachloroethene	5	ND (1.8)	ND (5)	ND (2)	**0.9**	**1**
Trichloroethene	5	**23**	ND (5)	**15**	**3**	**3.3**
Vinyl chloride	2	**230**	**67**	**72**	**14**	**16**

Location Name	New York TOGS	MW-124d			
Sample Name	111 Ambient	MW-124d-20200306	MW-124d-20211214	MW-124d-20230319	MW124D-073125-1545
Sample Date	Water Quality	03/06/2020	12/14/2021	03/19/2023	07/31/2025
Lab Sample ID	Standards	20200306	20211214	240-182494-10	L2548555-02
Volatile Organic Compounds (ug/L)					
1,1,1-Trichloroethane	5	ND (1)	ND (5)	ND (3.3)	ND (2.5)
1,1-Dichloroethane	5	ND (1)	**12**	ND (3.3)	**6.3 J**
Chloroethane	5	**260**	**150**	**140**	**100 J**
cis-1,2-Dichloroethene	5	ND (1)	ND (5)	ND (3.3)	**0.9 J**
Tetrachloroethene	5	ND (1)	ND (5)	ND (3.3)	ND (0.5)
Trichloroethene	5	ND (1)	ND (5)	ND (3.3)	ND (0.5)
Vinyl chloride	2	ND (1)	ND (5)	ND (3.3)	**1.8 J**

MW-115
MW-116D
MW-114
MW-116
MW-124S
MW-117
MW-118
MW-124D
MW-122
SS-3
SS-4
MW-121
SS-2
SS-1
AVENUE F
LEHIGH STREET
GATES AVENUE

LEGEND

- MONITORING WELL
- SUB-SLAB/INDOOR AIR SVI SAMPLE
- FENCE
- RAILROAD
- CONCENTRATION EXCEEDENCE
- CONCRETE PAD
- AST CONTAINMENT AREA
- VOLUNTARY CLEANUP PROGRAM SITE #V0019-8
- SITE BOUNDARY
- PARCEL BOUNDARY

NOTES

1. ALL LOCATIONS AND DIMENSIONS ARE APPROXIMATE.

2. ASSESSOR PARCEL DATA SOURCE: NEW YORK STATE GIS CLEARNING HOUSE

3. DEFINITIONS
SVI = SOIL VAPOR INTRUSION
AST = ABOVEGROUND STORAGE TANK
J = VALUE IS ESTIMATED

4. ALL DETECTED ANALYTES SHOWN ON FIGURE.

5. ND (2.5): NOT DETECTED, NUMBER IN PARENTHESES IS THE LABORATORY REPORTIN LIMIT.

6. GROUNDWATER ANALYTICLA RESULTS ARE COMPARED TO NYSDEC TECHNICAL AND OPERATIONAL GUIDANCE SERIES (TOGS) 1.1.1 AMBIENT WATER QUALITY STANDARDS AND GUIDANCE VALUES (SGVS) FOR CLASS GA WATER.

5. SOIL VAPOR ANALYSIS – VOLATILE ORGANIC COMPOUNDS (VOCs)

6. RESULTS ARE DISPLAYED IN MICROGRAMS PER CUBIC METER (µg/m3).

7. AERIAL IMAGERY SOURCE: NEARMAP, APRIL 17, 2025

N

0 80 160
SCALE IN FEET

HALEY ALDRICH

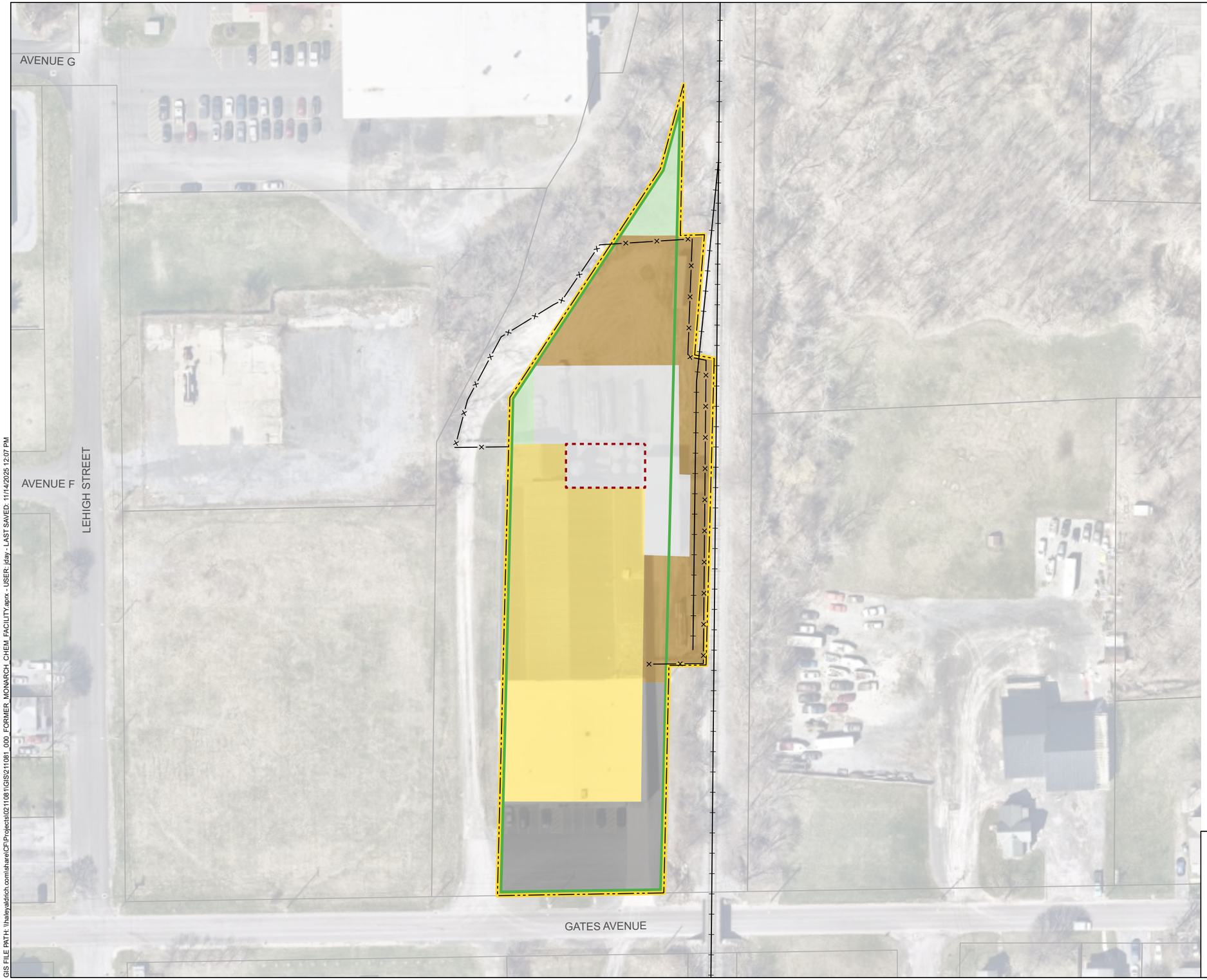
FORMER MONARCH CHEMICALS FACILITY
61 GATES AVENUE
GENEVA, NEW YORK

GROUNDWATER ANALYTICAL RESULTS - 2020 THROUGH 2025

NOVEMBER 2025

FIGURE 7

GIS FILE PATH: \\haleyaldrich.com\share\CF\Projects\021081\GIS\211081_000_FORMER_MONARCH_CHEM_FACILITY.aprx - USER: jday - LAST SAVED: 11/14/2025 12:07 PM



LEGEND

- FENCE
- RAILROAD
- AST CONTAINMENT AREA
- VOLUNTARY CLEANUP PROGRAM SITE #V0019-8
- SITE BOUNDARY
- PARCEL BOUNDARY

SURFACE COVER TYPE

- BUILDING
- BLACKTOP / ASPHALT
- CONCRETE PAD
- GRAVEL / FILL
- VEGETATIVE

NOTES

1. ALL LOCATIONS AND DIMENSIONS ARE APPROXIMATE.

2. ASSESSOR PARCEL DATA SOURCE: NEW YORK STATE GIS CLEARNING HOUSE

3. DEFINITION
AST = ABOVEGROUND STORAGE TANK

4. AERIAL IMAGERY SOURCE: NEARMAP, APRIL 17, 2025

N

0 80 160
SCALE IN FEET

HALEY ALDRICH

FORMER MONARCH CHEMICALS FACILITY
61 GATES AVENUE
GENEVA, NEW YORK

SURFACE COVER TYPE

NOVEMBER 2025

FIGURE 8

GIS FILE PATH: \\haleyaldrich.com\share\CF\Projects\0211081\GIS\211081_000_FORMER_MONARCH_CHEM_FACILITY.aprx - USER: jday - LAST SAVED: 11/14/2025 12:07 PM

APPENDIX A
Declaration of Covenants and Restrictions

DECLARATION of COVENANTS and RESTRICTIONS

THIS COVENANT is made the 18th day of May 2017 by Univar USA, Inc., as successor to Basic Chemical Solutions, LLC, a limited liability company organized and existing under the laws of the State of Washington and having an office for the transaction of business at 61 Gates Avenue, Geneva, NY 14456.

WHEREAS, the former H.B. Fuller – Monarch Chemicals division (Site #V00119-8) is the subject of a Voluntary Cleanup Agreement executed by the H.B. Fuller Company as part of the New York State Department of Environmental Conservation's (the "Department's) Voluntary Cleanup Program, namely that parcel of real property located at the address of 61 Gates Avenue in the City of Geneva, County of Ontario, State of New York, being the same as (or part of) that property conveyed to Basic Chemical Solutions, LLC by Holtrachem, Inc. by deed(s) dated September 28, 1998 and recorded on the September 30, 1998 in Ontario County Clerk's Office in Liber and Page 1002/647, and being more particularly described in Schedule "A," attached to this declaration and made a part hereof, and hereinafter referred to as "the Property"; and

WHEREAS, the Department approved a remedy to eliminate or mitigate all significant threats to the environment presented by the contamination disposed at the Property and such remedy requires that the Property be subject to restrictive covenants (the "Remedy".)

NOW, THEREFORE, Univar USA, Inc., for itself and its successors and/or assigns, covenants that:

First, the Property subject to this Declaration of Covenants and Restrictions is as shown on a map attached to this declaration as Schedule "B" and made a part hereof.

Second, unless prior written approval by the Department or, if the Department shall no longer exist, any New York State agency or agencies subsequently created to protect the environment of the State and the health of the State's citizens, hereinafter referred to as "the Relevant Agency," is first obtained, where contamination remains at the Property subject to the provisions of the Site Management Plan ("SMP"), there shall be no construction, use or occupancy of the Property that results in the disturbance or excavation of the Property which threatens the integrity of the engineering controls or which results in unacceptable human exposure to contaminated soils. The SMP may be obtained from the New York State Department of Environmental Conservation, Division of Environmental Remediation, Site Control Section, 625 Broadway, Albany, NY 12233.

Third, the owner of the Property shall not disturb, remove, or otherwise interfere with the installation, use, operation, and maintenance of engineering controls required for the Remedy,

which are described in the SMP, unless in each instance the owner first obtains a written waiver of such prohibition from the Department or Relevant Agency.

Fourth, the owner of the Property shall prohibit the Property from ever being used for purposes other than for **Commercial as described in 6 NYCRR Part 375-1.8(g)(2)(iii) and Industrial as described in 6 NYCRR Part 375-1.8(g)(2)(iv)**
without the express written waiver of such prohibition by the Department or Relevant Agency.

Fifth, the use of groundwater underlying the property is prohibited without necessary water quality treatment as determined by the NYSDOH or the Ontario County Department of Health to render it safe for use as drinking water or for industrial purposes, and the user must first notify and obtain written approval to do so from the Department.

Sixth, the owner of the Property shall provide a periodic certification, prepared and submitted by a professional engineer or environmental professional acceptable to the Department or Relevant Agency, which will certify that the institutional and engineering controls put in place are unchanged from the previous certification, comply with the SMP, and have not been impaired.

Seventh, the owner of the Property shall continue in full force and effect any institutional and engineering controls required for the Remedy and maintain such controls, unless the owner first obtains permission to discontinue such controls from the Department or Relevant Agency, in compliance with the approved SMP, which is incorporated and made enforceable hereto, subject to modifications as approved by the Department or Relevant Agency.

Eighth, this Declaration is and shall be deemed a covenant that shall run with the land and shall be binding upon all future owners of the Property, and shall provide that the owner and its successors and assigns consent to enforcement by the Department or Relevant Agency of the prohibitions and restrictions that the Voluntary Cleanup Agreement requires to be recorded, and hereby covenant not to contest the authority of the Department or Relevant Agency to seek enforcement.

Ninth, any deed of conveyance of the Property, or any portion thereof, shall recite, unless the Department or Relevant Agency has consented to the termination of such covenants and restrictions, that said conveyance is subject to this Declaration of Covenants and Restrictions.

IN WITNESS WHEREOF, the undersigned has executed this instrument the day written below.

By: _____

Print Name: MARK METCALF

Title: Director - Random Date: 5/18/2017

Grantor's Acknowledgment

STATE OF NEW YORK)

) s.s.:

COUNTY OF)

On the 18th day of May, in the year 201_, before me, the undersigned, personally appeared mark metcalf, personally known to me or proved to me on the basis of satisfactory evidence to be the individual(s) whose name is (are) subscribed to the within instrument and acknowledged to me that he/she/they executed the same in his/her/their capacity(ies), and that by his/her/their signature(s) on the instrument, the individual(s), or the person upon behalf of which the individual(s) acted, executed the instrument.

_____

Notary Public State of ~~New York~~ Ohio

NOTARIAL SEAL STATE OF OHIO

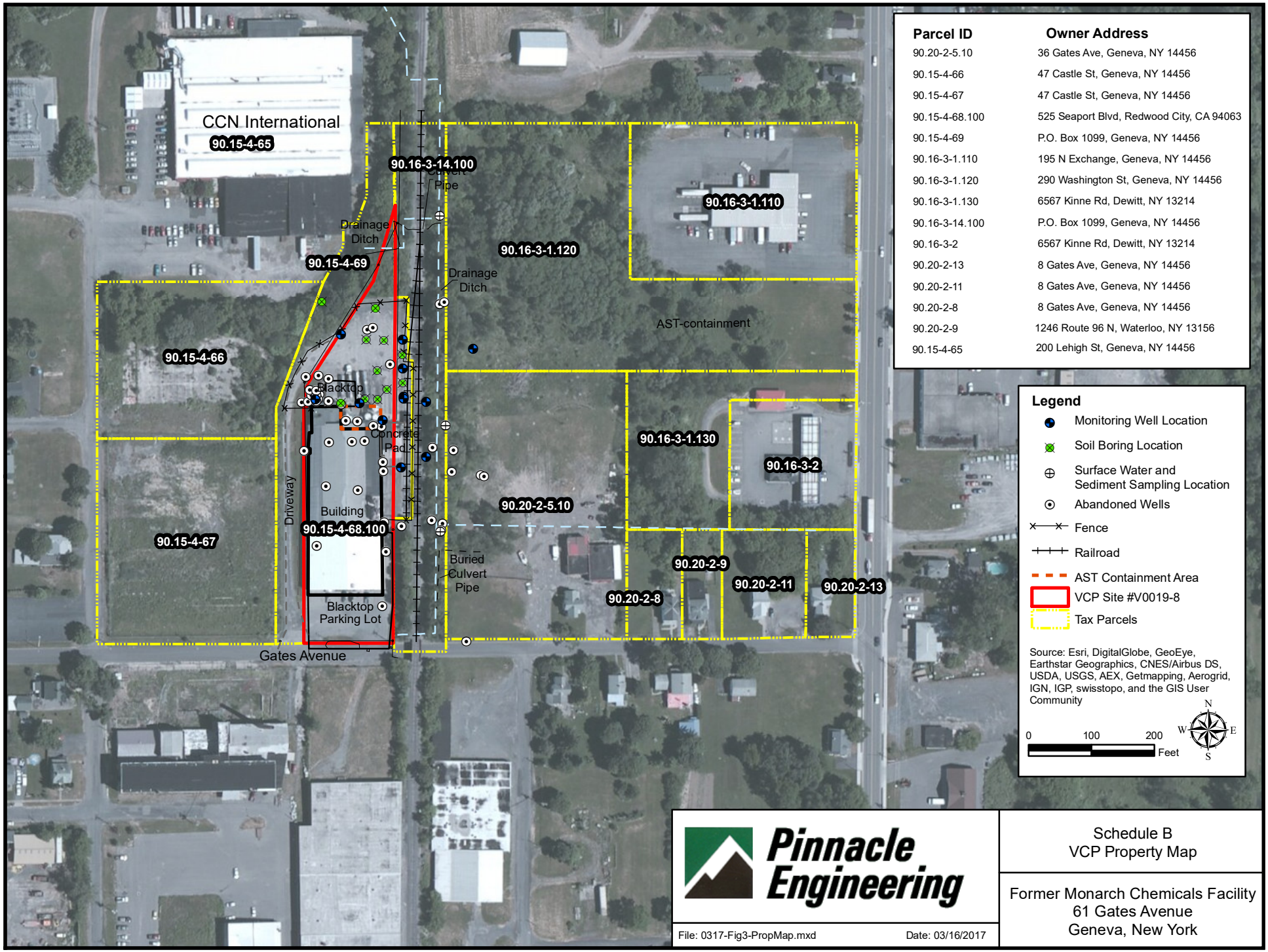
RENA J. ZAMSKI
NOTARY PUBLIC, STATE OF OHIO
MY COMMISSION EXPIRES 1/9/22

[06/14]

SCHEDULE "A"

ALL THAT TRACT OF LAND situate in the City of Geneva, County of Ontario and State of New York, bounded and described as follows: Commencing at a point on the north line of Gates Avenue, distant 310.0 feet easterly from the intersection of the north line of Gates Avenue, with the easterly line of Lehigh Street, as measured along the north line of Gates Avenue; thence (1) N 10° 15' 00" E a distance of 417.21 feet to a point; (2) N 42° 57' 00" E a distance of 225.14 feet to a point; thence (3) N 35° 02' 00" E, a distance of 72.00 feet to a point; thence (4) S 11° 42' 00 W, a distance of 672.09 feet to a point on the north line of Gates Avenue; thence (5) N 79° 49' 00" W, along the north line of Gates Avenue, a distance of 134.80 feet to a point and place of beginning; all as shown on a map dated November 1, 1979, made by Roland M. Phillips, L.S. No. 049149 from a Survey dated October 31, 1979 and from information shown on a map prepared by Ronald M. Phillips and dated November 17, 1977, said maps being entitled "Map of a Survey of a Parcel owned by K&L Realty Company, City of Geneva, Ontario County, New York" and "Map of a Parcel owned by Lake City Developers, Inc., City of Geneva, Ontario County, New York", respectively, being filed in the Ontario County Clerk's Office on November 30, 1979, being Map No. 9003 and on February 28, 1978 Map No. 8215, respectively.

SCHEDULE "B"



Parcel ID	Owner Address
90.20-2-5.10	36 Gates Ave, Geneva, NY 14456
90.15-4-66	47 Castle St, Geneva, NY 14456
90.15-4-67	47 Castle St, Geneva, NY 14456
90.15-4-68.100	525 Seaport Blvd, Redwood City, CA 94063
90.15-4-69	P.O. Box 1099, Geneva, NY 14456
90.16-3-1.110	195 N Exchange, Geneva, NY 14456
90.16-3-1.120	290 Washington St, Geneva, NY 14456
90.16-3-1.130	6567 Kinne Rd, Dewitt, NY 13214
90.16-3-14.100	P.O. Box 1099, Geneva, NY 14456
90.16-3-2	6567 Kinne Rd, Dewitt, NY 13214
90.20-2-13	8 Gates Ave, Geneva, NY 14456
90.20-2-11	8 Gates Ave, Geneva, NY 14456
90.20-2-8	8 Gates Ave, Geneva, NY 14456
90.20-2-9	1246 Route 96 N, Waterloo, NY 13156
90.15-4-65	200 Lehigh St, Geneva, NY 14456

Legend

- Monitoring Well Location
- Soil Boring Location
- Surface Water and Sediment Sampling Location
- Abandoned Wells
- Fence
- Railroad
- AST Containment Area
- VCP Site #V0019-8
- Tax Parcels

Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AEX, Getmapping, Aerogrid, IGN, IGP, swisstopo, and the GIS User Community

0 100 200 Feet

N W E S

Pinnacle Engineering

File: 0317-Fig3-PropMap.mxd Date: 03/16/2017

Schedule B
VCP Property Map

Former Monarch Chemicals Facility
61 Gates Avenue
Geneva, New York

APPENDIX B
Site Contact List

APPENDIX B – LIST OF SITE CONTACTS

Name	Phone/Email Address
Site Owner – FNLR Compounds Matter Too LLC William Turner Chase Romney	wturner@fortress.com cromney@fortress.com
Remedial Party - H.B. Fuller Company/Jonathan Blaine	(651) 236-5900 jonathan.blaine@hbfuller.com
Qualified Environmental Professional – H & A of New York Engineering and Geology / Santa McKenna	(585) 321-4242 smckenna@haleyaldrich.com
NYSDEC DER Project Manager – Kathryn Lovell, GIT	(585) 438-5280 kathryn.lovell@dec.ny.gov
NYSDEC DER Project Manager's Supervisor – David Pratt, P.E.	(585) 226-5449 david.pratt@dec.ny.gov
NYSDEC Site Control - Kelly A. Lewandowski, P.E.	(518) 402-9547 kelly.lewandowski@dec.ny.gov
NYSDOH Project Manager - Julia Kenney, P.G.	(518) 402-7873 julia.kenney@health.ny.gov

APPENDIX C
Excavation Workplan

APPENDIX C – EXCAVATION WORK PLAN (EWP)

C-1 NOTIFICATION

At least 15 days prior to the start of any activity that is anticipated to encounter remaining contamination or breach or alter the site's cover system, the site owner or their representative will notify the NYSDEC contacts listed in the table below. Table 1 includes contact information for the above notification. The information on this table will be updated as necessary to provide accurate contact information. A full listing of site-related contact information is provided in Appendix B.

Table 1: Notifications*

NYSDEC DER Project Manager – Kathryn Lovell, GIT	(585)438-5280 kathryn.lovell@dec.ny.gov
NYSDEC DER Project Manager's Supervisor – David Pratt, P.E.	585-226-5449 david.pratt@dec.ny.gov
NYSDEC Site Control - Kelly A. Lewandowski, P.E.	518-402-9547 kelly.lewandowski@dec.ny.gov

* Note: Notifications are subject to change and will be updated as necessary.

This notification will include:

- A detailed description of the work to be performed, including the location and areal extent of excavation, plans/drawings for site re-grading, intrusive elements or utilities to be installed below the soil cover, estimated volumes of contaminated soil to be excavated, any modifications of truck routes, and any work that may impact an engineering control;
- A summary of environmental conditions anticipated to be encountered in the work areas, including the nature and concentration levels of contaminants of concern, potential presence of grossly contaminated media, and plans for any pre-construction sampling;

- A schedule for the work, detailing the start and completion of all intrusive work, and submittals (e.g., reports) to the NYSDEC documenting the completed intrusive work;
- A summary of the applicable components of this EWP;
- A statement that the work will be performed in compliance with this EWP, 29 CFR 1910.120 and 29 CFR 1926 Subpart P;
- A copy of the contractor's health and safety plan (SHSP), in electronic format, if it differs from the SHSP provided in Appendix C of this SMP;
- Identification of disposal facilities for potential waste streams; and
- Identification of sources of any anticipated backfill, along with the required request to import form and all supporting documentation including, but not limited to, chemical testing results.

The NYSDEC project manager will review the notification and may impose additional requirements for the excavation that are not listed in this EWP. The alteration, restoration and modification of engineering controls must conform with Article 145 Section 7209 of the Education Law regarding the application professional seals and alterations.

C-2 SOIL SCREENING METHODS

Visual, olfactory and instrument-based (e.g. photoionization detector) soil screening will be performed during all excavations into known or potentially contaminated material (remaining contamination) or a breach of the cover system. A qualified environmental professional as defined in 6 NYCRR Part 375, a PE who is licensed and registered in New York State, or a qualified person who directly reports to a PE who is licensed and registered in New York State will perform the screening. Soil screening will be performed when invasive work is done and will include all excavation and invasive work performed during development, such as excavations for foundations and utility work, after issuance of the COC.

Soils will be segregated based on field screening into material that requires off-Site disposal and material that requires testing to determine if the material can be reused on Site. Soil sampling and screening will be competed in accordance with the Sampling and Analysis Plan (Appendix E) of the SMP. Further discussion of possible off-Site disposal of materials and on-Site reuse will be made depending on Soil Profile Results.

C-3 SOIL STAGING METHODS

Soil stockpiles will be continuously encircled with a berm and/or silt fence. Hay bales will be used as needed near catch basins, surface waters and other discharge points.

Stockpiles will be kept covered at all times with appropriately anchored tarps. Stockpiles will be routinely inspected and damaged tarp covers will be promptly replaced.

Stockpiles will be inspected at a minimum once each week and after every storm event. Results of inspections will be recorded in a logbook and maintained at the site and available for inspection by the NYSDEC.

C-4 MATERIALS EXCAVATION AND LOAD-OUT

A qualified environmental professional as defined in 6 NYCRR Part 375, a PE who is licensed and registered in New York State, or a qualified person who directly reports to a PE who is licensed and registered in New York State will oversee all invasive work and the excavation and load-out of all excavated material.

The owner of the property and remedial party (if applicable) and its contractors are responsible for safe execution of all invasive and other work performed under this Plan.

The presence of utilities and easements on the site will be investigated by the qualified environmental professional. It will be determined whether a risk or impediment to the planned work under this SMP is posed by utilities or easements on the site. A site

utility stakeout will be completed for all utilities prior to any ground intrusive activities at the site.

Loaded vehicles leaving the site will be appropriately lined, tarped, securely covered, manifested, and placarded in accordance with appropriate Federal, State, local, and NYSDOT requirements (and all other applicable transportation requirements). Trucks transporting contaminated soil must have either tight-fitting opaque covers that are secured on the sides and/or back, or opaque covers that are locked on all sides.

A truck wash will be operated on-site, as appropriate. The qualified environmental professional will be responsible for ensuring that all outbound trucks will be washed at the truck wash before leaving the site until the activities performed under this section are complete. Truck wash waters will be collected and disposed of off-site in an appropriate manner.

Locations where vehicles enter or exit the site shall be inspected daily for evidence of off-site soil tracking.

The qualified environmental professional will be responsible for ensuring that all egress points for truck and equipment transport from the site are clean of dirt and other materials derived from the site during intrusive excavation activities. Cleaning of the adjacent streets will be performed as needed to maintain a clean condition with respect to site-derived materials. Material accumulated from the street cleaning and egress cleaning activities will be disposed off-site at a permitted landfill facility in accordance with all applicable local, State, and Federal regulations.

C-5 MATERIALS TRANSPORT OFF-SITE

All transport of materials will be performed by licensed haulers in accordance with appropriate local, State, and Federal regulations, including 6 NYCRR Part 364. Haulers will be appropriately licensed and trucks properly placarded.

Material transported by trucks exiting the site will be secured with either tight-fitting opaque covers that are secured on the sides and/or back, or opaque covers that are locked on all sides. Loose-fitting canvas-type truck covers will be prohibited. If loads contain wet material capable of producing free liquid, truck liners will be used.

Trucks will be prohibited from stopping and idling in the neighborhood outside the project site.

Egress points for truck and equipment transport from the site will be kept clean of dirt and other materials during site remediation and development.

Queuing of trucks will be performed on-site in order to minimize off-site disturbance. Off-site queuing will be prohibited.

C-6 MATERIALS DISPOSAL OFF-SITE

All material excavated and removed from the site will be treated as contaminated and regulated material and will be transported and disposed off-site in a permitted facility in accordance with all local, State and Federal regulations. If disposal of material from this site is proposed for unregulated off-site disposal (i.e. clean soil removed for development purposes), a formal request with an associated plan will be made to the NYSDEC project manager. Unregulated off-site management of materials from this site will not occur without formal NYSDEC project manager approval.

Off-site disposal locations for excavated soils will be identified in the pre-excavation notification. This will include estimated quantities and a breakdown by class of disposal facility if appropriate, (e.g. hazardous waste disposal facility, solid waste landfill, petroleum treatment facility, C&D debris recovery facility). Actual disposal quantities and associated documentation will be reported to the NYSDEC in the Periodic Review Report. This documentation will include, but will not be limited to: waste profiles, test results, facility acceptance letters, manifests, bills of lading and facility receipts.

Non-hazardous historic fill and contaminated soils taken off-site will be handled consistent with 6 NYCRR Parts 360, 361, 362, 363, 364 and 365. Material that does not meet Unrestricted SCOs is prohibited from being taken to a New York State C&D debris recovery facility (6 NYCRR Subpart 361-5 registered or permitted facility).

C-7 MATERIALS REUSE ON-SITE

The qualified environmental professional, as defined in 6 NYCRR Part 375, will ensure that procedures defined for materials reuse in this SMP are followed and that unacceptable material (i.e. contaminated) does not remain on-site. Contaminated on-site material, including historic fill and contaminated soil, that is acceptable for reuse on-site will be placed below the demarcation layer or impervious surface, and will not be reused within the cover system or within landscaping berms. Contaminated on-site material may only be used beneath the site cover as backfill for subsurface utility lines with prior approval from the DEC project manager.

Proposed materials for reuse on-site must be sampled for full suite analytical parameters including per- and polyfluoroalkyl substances (PFAS) and 1,4-dioxane. The sampling frequency will be in accordance with DER-10 Table 5.4(e)10 unless prior approval is obtained from the NYSDEC project manager for modification of the sampling frequency. The analytical results of soil/fill material testing must meet the site use criteria presented in NYSDEC DER-10 Appendix 5 – Allowable Constituent Levels for Imported Fill or Soil for all constituents listed, and the NYSDEC Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances March 2023 or date of current version, whichever is later guidance values. Approvals for modifications to the analytical parameters must be obtained from the NYSDEC project manager prior to the sampling event.

Soil/fill material for reuse on-site will be segregated and staged as described in Sections C-2 and C-3 of this EWP. The anticipated size and location of stockpiles will be provided in the 15-day notification to the NYSDEC project manager. Stockpile locations will be based on the location of site excavation activities and proximity to nearby site features. Material reuse on-site will comply with requirements of NYSDEC DER-10 Section 5.4(e)4. Any modifications to the requirements of DER-10 Section 5.4(e)4 must be approved by the NYSDEC project manager.

Any demolition material proposed for reuse on-site will be sampled for asbestos and the results will be reported to the NYSDEC for acceptance. Concrete crushing or processing on-site will not be performed without prior NYSDEC approval. Organic matter (wood, roots, stumps, etc.) or other solid waste derived from clearing and grubbing of the site may only be reused on-site with written approval from the NYSDEC project manager.

C-8 FLUIDS MANAGEMENT

All liquids to be removed from the site, including but not limited to, excavation dewatering, decontamination waters and groundwater monitoring well purge and development waters, will be handled, transported and disposed off-site at a permitted facility in accordance with applicable local, State, and Federal regulations. Dewatering, purge and development fluids will not be recharged back to the land surface or subsurface of the site, and will be managed off-site, unless prior approval is obtained from NYSDEC.

Discharge of water generated during large-scale construction activities to surface waters (i.e. a local pond, stream or river) will be performed under a SPDES permit.

C-9 COVER SYSTEM RESTORATION

After the completion of soil removal and any other invasive activities the cover system will be restored in a manner that complies with the Decision Document. The existing cover system is comprised of a combination of gravel fill and native soils

extending to depths of approximately 14 to 24 feet below ground surface, asphalt pavement, concrete-covered sidewalks, and concrete building slabs.. If the type of cover system changes from that which exists prior to the excavation (i.e., a soil cover is replaced by asphalt), this will constitute a modification of the cover element of the remedy and the upper surface of the remaining contamination. A figure showing the modified surface will be included in the subsequent Periodic Review Report and in an updated SMP. The alteration, restoration and modification of engineering controls must conform with Article 145 Section 7209 of the Education Law regarding the application professional seals and alterations.

C-10 BACKFILL FROM OFF-SITE SOURCES

All materials proposed for import onto the site will be approved by the qualified environmental professional, as defined in 6 NYCRR Part 375, and will be in compliance with provisions in this SMP prior to receipt at the site. A Request to Import/Reuse Fill or Soil form, which can be found at http://www.dec.ny.gov/regulations/67386.html, will be prepared and submitted to the NYSDEC project manager allowing a minimum of 5 business days for review. A copy of the form is presented in Appendix K.

Material from industrial sites, spill sites, other environmental remediation sites, or potentially contaminated sites will not be imported to the site.

All imported soils will meet the backfill and cover soil quality standards established in 6 NYCRR 375-6.7(d) and DER-10 Appendix 5 for industrial use. Based on an evaluation of the land use, protection of groundwater and protection of ecological resources criteria, the resulting soil quality standards are DER-10. Soils that meet ‘general’ fill requirements under 6 NYCRR Part 360.13, but do not meet backfill or cover soil objectives for this site, will not be imported onto the site without prior approval by NYSDEC project manager. Soil material will be sampled for the full suite of analytical parameters, including PFAS and 1, 4-dioxane. Solid waste will not be imported onto the site.

Trucks entering the site with imported soils will be securely covered with tight fitting covers. Imported soils will be stockpiled separately from excavated materials and covered to prevent dust releases.

C-11 STORMWATER POLLUTION PREVENTION

Barriers and hay bale checks will be installed and inspected once a week and after every storm event. Results of inspections will be recorded in a logbook and maintained at the site and available for inspection by the NYSDEC. All necessary repairs shall be made immediately.

Accumulated sediments will be removed as required to keep the barrier and hay bale check functional.

All undercutting or erosion of the silt fence toe anchor shall be repaired immediately with appropriate backfill materials.

Manufacturer's recommendations will be followed for replacing silt fencing damaged due to weathering.

Erosion and sediment control measures identified in the SMP shall be observed to ensure that they are operating correctly. Where discharge locations or points are accessible, they shall be inspected to ascertain whether erosion control measures are effective in preventing significant impacts to receiving waters.

Silt fencing or hay bales will be installed around the entire perimeter of the construction area.

C-12 EXCAVATION CONTINGENCY PLAN

If underground tanks or other previously unidentified contaminant sources are found during post-remedial subsurface excavations or development related construction, excavation activities will be suspended until sufficient equipment is mobilized to address the condition. The NYSDEC project manager will be promptly notified of the discovery.

Sampling will be performed on product, sediment and surrounding soils, etc. as necessary to determine the nature of the material and proper disposal method. Chemical analysis will be performed for a full list of analytes [TAL metals, TCL volatiles and semi-volatiles (including 1,4-dioxane), TCL pesticides and PCBs, and PFAS], unless the site history and previous sampling results provide sufficient justification to limit the list of analytes. In this case, a reduced list of analytes will be proposed to the NYSDEC project manager for approval prior to sampling. Any tanks will be closed as per NYSDEC regulations and guidance.

Identification of unknown or unexpected contaminated media identified by screening during invasive site work will be promptly communicated by phone within two hours to NYSDEC's Project Manager. Reportable quantities of petroleum product will also be reported to the NYSDEC spills hotline. These findings will be also included in the Periodic Review Report.

C-13 COMMUNITY AIR MONITORING PLAN

A figure showing the location of air sampling stations each day of the soil excavation will be prepared with sampling station names and dates. These locations will be adjusted on a daily or more frequent basis based on actual wind directions to provide an upwind and at least two downwind monitoring stations.

Air monitoring will be completed in accordance with the CAMP provided in Appendix E of the SMP. Exceedances of action levels listed in the CAMP will be reported to NYSDEC and NYSDOH Project Managers.

C-13A: Special Requirements for Work Within 20 Feet of Potentially Exposed Individuals or Structures

When work areas are within 20 feet of potentially exposed populations or occupied structures, the continuous monitoring locations for VOCs and particulates must reflect the nearest potentially exposed individuals and the location of ventilation system intakes for nearby structures. The use of engineering controls such as vapor/dust barriers, temporary negative-pressure enclosures, or special ventilation devices should be considered to prevent exposures related to the work activities and to control dust and odors. Consideration should be given to implementing the planned activities when potentially exposed populations are at a minimum, such as during weekends or evening hours in non-residential settings.

- If total VOC concentrations opposite the walls of occupied structures or next to intake vents exceed 1 part-per-million, monitoring should occur within the occupied structure(s). Depending upon the nature of contamination, chemical-specific colorimetric tubes of sufficient sensitivity may be necessary for comparing the exposure point concentrations with appropriate pre-determined response levels (response actions should also be pre-determined). Background readings in the occupied spaces must be taken prior to commencement of the planned work. Any unusual background readings should be discussed with NYSDOH prior to commencement of the work.

- If total particulate concentrations opposite the walls of occupied structures or next to intake vents exceed 150 micrograms per cubic meter, work activities should be suspended until controls are implemented and are successful in reducing the total particulate concentration to 150 micrograms per cubic meter or less at the monitoring point.

- Depending upon the nature of contamination and remedial activities, other parameters (e.g., explosivity, oxygen, hydrogen sulfide, carbon monoxide) may also need to be monitored. Response levels and actions should be pre-determined, as necessary, for each site.

C-14 ODOR CONTROL PLAN

As of the date of this SMP remedial systems do not exist at the Site. This section is not applicable.

This odor control plan is capable of controlling emissions of nuisance odors off-site. Specific odor control methods to be used on a routine basis will include. If nuisance odors are identified at the site boundary, or if odor complaints are received, work will be halted and the source of odors will be identified and corrected. Work will not resume until all nuisance odors have been abated. NYSDEC and NYSDOH will be notified of all odor events and of any other complaints about the project. Implementation of all odor controls, including the halt of work, is the responsibility of the remedial party's Remediation Engineer, and any measures that are implemented will be discussed in the Periodic Review Report.

All necessary means will be employed to prevent on- and off-site nuisances. At a minimum, these measures will include: (a) limiting the area of open excavations and size of soil stockpiles; (b) shrouding open excavations with tarps and other covers; and (c) using foams to cover exposed odorous soils; If odors develop and cannot be otherwise controlled, additional means to eliminate odor nuisances will include: (d) direct load-out of soils to trucks for off-site disposal; (e) use of chemical odorants in spray or misting systems; and, (f) use of staff to monitor odors in surrounding neighborhoods.

If nuisance odors develop during intrusive work that cannot be corrected, or where the control of nuisance odors cannot otherwise be achieved due to on-site conditions or close proximity to sensitive receptors, odor control will be achieved by sheltering the excavation and handling areas in a temporary containment structure equipped with appropriate air venting/filtering systems.

C-15 DUST CONTROL PLAN

Particulate monitoring must be conducted according to the Community Air Monitoring Plan (CAMP) provided in Section C-12. If particulate levels at the site exceed the thresholds listed in the CAMP or if airborne dust is observed on the site or leaving the site, the dust suppression techniques listed below will be employed. The remedial party will also take measures listed below to prevent dust production on the site.

A dust suppression plan that addresses dust management during invasive on-site work will include, at a minimum, the items listed below:

- Dust suppression will be achieved using a dedicated on-site water truck for road wetting. The truck will be equipped with a water cannon capable of spraying water directly onto off-road areas including excavations and stockpiles.
- Clearing and grubbing of larger sites will be done in stages to limit the area of exposed, unvegetated soils vulnerable to dust production.
- Gravel will be used on roadways to provide a clean and dust-free road surface.
- On-site roads will be limited in total area to minimize the area required for water truck sprinkling.

C-16 OTHER NUISANCES

As of the date of this SMP remedial systems do not exist at the Site. This section is not applicable.

A plan for rodent control will be developed and utilized by the contractor prior to and during site clearing and site grubbing, and during all remedial work.

A plan will be developed and utilized by the contractor for all remedial work to ensure compliance with local noise control ordinances.

APPENDIX D
Site Health and Safety Plan

Company HASP

Revision Number	Revision Date	Summary of Changes	Approved By
0	8/1/2025	Initial	Brian Fitzpatrick
1	11/3/2025	Revise WorkCare to Acuity	Maggie Cahoon

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1. PURPOSE

The purpose of this Company Health and Safety Plan (HASP) is to address site-specific hazards for work activities that Haley & Aldrich undertakes on Hazardous Waste Operations and Emergency Response (HAZWOPER) sites. This document identifies appropriate mitigations and control measures to help eliminate or reduce identified risks or directs staff to complete project Job Hazard Analysis (JHA) to further document the risks and mitigations specific to that site. Project JHAs and task-specific JHAs are used to augment this document to provide more detailed information on risk and mitigations. Project JHAs provide directions on site-specific risks such as, but not limited to, simultaneous operations, security, and emergency response. JHAs task pages are used to identify the tasks associated with the project work, and the risks and mitigations specific to that task.

Included in this document are references to Haley & Aldrich Operating Procedures (OPs) and policies related to the work performed at project sites.

2. EXPECTATIONS

2.1 FOUNDATIONAL SAFETY BEHAVIORS

The Foundational Safety Behaviors are designed to advance and streamline safety across the organization. These behaviors were developed through extensive feedback, collaboration, and engagement across the company.

These behaviors are not just guidelines, but must be integrated into daily operations of all staff members to ensure consistency across the organization. By taking ownership of these behaviors, every staff member contributes to a safer work environment, ultimately advancing the overall safety culture at Haley & Aldrich.

2.2 STOP WORK AUTHORITY

In accordance with Haley & Aldrich OP1035 - Stop Work Authority, any individual has the right to refuse to perform work that they believe to be unsafe without fear of retaliation. They also have the authority, obligation, and responsibility to stop others from working in an unsafe manner. Stop Work does not always mean to stop all

work on a project, but it can also mean that you are stopping a task or activity to ensure that everyone is working safely.

- **Stop** working immediately when perceiving a dangerous situation.
- **Notify** coworkers, supervisors, and any other relevant individuals of the stop-work action.
- **Investigate** the situation and come to an agreement on whether work should resume or be suspended until the risk is mitigated.
- **Necessary corrections** shall be made and inspected by qualified experts to verify that the issue has been resolved and that work can proceed safely.
- **Resume work** once the relevant authority has approved of the situation and issued a notice that corrective actions have been implemented.
- **Follow up** from management in the form of investigations, improvements, or relevant reports.

STOP Work Authority is the stop work policy for all personnel and subcontractors on the site. When work has been stopped due to an unsafe condition, Haley & Aldrich site management (e.g., Project Manager [PM], Site Safety Officer [SSO], Regional Safety Manager [RSM], etc.) will be notified immediately.

Reasons for issuing a Stop Work order include, but are not limited to:

- The belief/perception that injury to personnel or accident-causing significant damage to property or equipment is imminent.
- A Haley & Aldrich subcontractor is in breach of site safety requirements and/or their own site HASP.
- Identifying a substandard condition (e.g., severe weather) or activity that creates an unacceptable safety risk as determined by a qualified person.

Work will not resume until the unsafe act has been stopped OR sufficient safety precautions have been taken to remove or mitigate the risk to a degree of which work can resume safely. Any instances of stopping work will be documented through Health & Safety's digital reporting tool. Once work has been stopped, only the Haley & Aldrich PM or SSO can give the order to resume work. Haley & Aldrich senior management is committed to support anyone who exercises their "Stop Work" authority.

2.3 HEALTH AND SAFETY REPORTING

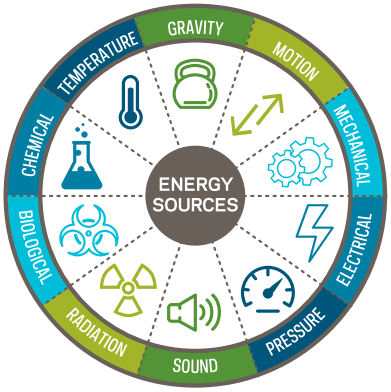
All staff members are required to use Health & Safety's digital reporting tool to document any safety events occurring at work, or related to work. Forms are available for various purposes, including events, injuries, and sensitive information. Staff must report safety events promptly, and the Health & Safety team is responsible for collaborating with both onsite and supporting staff to address safety concerns and feedback in a timely manner.

3. ISSUANCE AND COMPLIANCE

The Company HASP was developed in accordance with Occupational Safety and Health Administration (OSHA) regulations 29 CFR 1910.120 and 1926.65. This document outlines general requirements and information related to our project work. Project-specific documents, such as the JHAs, are developed by those most familiar with the

work and are reviewed and approved prior to starting work by the Haley & Aldrich RSM and PM. These documents must be reviewed and modified regularly as site conditions change.

- All Haley & Aldrich JHAs must be signed by Haley & Aldrich personnel involved in the implementation of the Scope of Work (SOW).
- Haley & Aldrich's subcontractors must have their own project health and safety documents which address hazards specific to their trade. All subcontractor JHAs must be signed by subcontractor personnel involved in the implementation of their respective SOW.
- The Company HASP, electronic or printed, must be accessible at all times when Haley & Aldrich staff are present.
- If changes are necessary based on the presence of high energy hazards, the RSM and PM must approve updates to the JHA. Any revision to the JHA requires staff members and subcontractors to be informed of the changes so that they understand the requirements of the change. Staff members and subcontractors must sign the applicable JHAs associated with their work following any revisions.



- Deviations from the JHA are permitted with approval from the Haley & Aldrich RSM and PM. Unauthorized deviations may constitute a violation of Haley & Aldrich company procedures/policies and may result in disciplinary action.
- Project JHAs will be relied upon by Haley & Aldrich staff, subcontractors, and visitors to the site. This document will also be available for review but is not intended to be the sole source of project safety. This HASP and all project-specific JHAs will be made available for review to Haley & Aldrich's subcontractors and other interested parties (e.g. facility personnel and regulatory agencies) prior to commencing work, to ensure that Haley & Aldrich has properly informed our subcontractors and others of the potential hazards associated with the implementation of the SOW to the extent that Haley & Aldrich is aware.

This Company HASP provides required information in alignment with OSHA regulations. General safety and health compliance programs in support of this HASP and project documents (e.g., injury reporting, medical surveillance, personal protective equipment [PPE] selection, etc.) are described in the Haley & Aldrich Corporate Health and Safety Program Manual and within Haley & Aldrich's Operating Procedures (OPs). Copies of project documents must be readily available on site and reviewed daily. Both the manual and SOPs can be located on the Haley & Aldrich's Company Intranet. Users of this HASP and other project documents should always refer to these resources and incorporate to the extent possible. The manual and SOPs are available to clients and regulators upon request.

4. ROLES AND RESPONSIBILITIES

4.1 PROJECT STAFF ROLES AND RESPONSIBILITIES

Project teams consist of multiple staff members working together to provide support to those executing work in the field. Below are general responsibilities of team members that contribute to project support.

4.1.1 Haley & Aldrich Project Manager (PM)

The Haley & Aldrich PM has the overall responsibility for the health and safety of Haley & Aldrich project personnel. The PM is responsible for ensuring the following to properly implement this HASP:

- Sufficient resources and materials are available.
- Day-to-day field investigation activities are planned and executed in a manner consistent with project-specific JHAs.
- Only staff who are properly trained and experienced are placed on a project site.
- Sufficient resources and materials are allocated to implement this HASP and are properly used.
- Site staff understand how to access a copy of the project-specific JHA through printed resources and project files, and this HASP electronically.
- All relevant company policies, procedures, and expectations are followed.

4.1.2 Haley & Aldrich Site Safety Officer (SSO)

The Haley & Aldrich SSO, or their designee, will be present during the execution of work. The SSO may perform additional roles on-site but will be responsible for Haley & Aldrich's health and safety and that of contracted subcontractors. The SSO will be responsible for administration of the site safety program and will ensure the elements of this HASP are properly implemented and understood by site personnel. The senior person for Haley & Aldrich on-site is the SSO, unless otherwise designated. The SSO will be named in the project-specific JHA.

4.1.3 Haley & Aldrich Regional Safety Manager (RSM)

The RSM, or their designee, is the safety lead for all Company projects within their designated region. The RSM is a full-time Haley & Aldrich staff member, trained as a safety and health professional. They will review and approve the site-specific safety documents and any required amendments, and lead the project teams in their safety efforts. The RSM is active in identifying and mitigating any potential safety and health issues. RSMs are available for site visits and play an active role as a resource for field staff members with any questions or concerns regarding site safety.

4.1.4 Haley & Aldrich Health & Safety Team

When the "Health & Safety Team" is referenced in this document it means, the Chief H&S Officer, Corporate H&S staff, RSMs, and/or their designees.

4.1.5 Haley & Aldrich Subcontractors

When Haley & Aldrich is the controlling employer, we expect each subcontractor will provide a qualified representative who will act as their Safety Lead, unless otherwise denoted in the contract. This person will be responsible for the planning, coordination, and safe execution of subcontractor tasks. This includes the preparation of a site-specific HASP, JHAs, performing daily safety planning, and coordinating directly with the Haley & Aldrich SSO for other site safety activities. This person will play a lead role in safety planning for subcontractor tasks, and in ensuring all their staff members and lower-tier subcontractors are in adherence with

applicable local, state, and/or federal regulations, and/or industry- and project-specific safety standards or best management practices.

The subcontractors' safety documentation will be at least as stringent as the health and safety requirements of the Haley & Aldrich HASP and JHAs. This will be verified by Haley & Aldrich prior to the start of work.

If any field staff working for the subcontractors do not speak English, their supervisor/foreman must be bilingual and be present in all safety meetings for translations. Appropriate multi-language signs must also be utilized in accordance with each worker's native language.

5. SCOPE OF WORK (SOW)

5.1 GENERAL AND PROJECT TASKS

The SOW for each project can be found on the project's JHA cover page. Attached task-specific JHA pages will detail tasks that are to be performed by all Haley & Aldrich staff members and will allow supporting Health & Safety team members to guide the project team through the process of planning for and mitigating possible hazards. These files are in safety folders under each project's Teams page.

When Haley & Aldrich is the controlling employer, we expect that project-specific tasks performed by subcontractors will be identified and evaluated by Haley & Aldrich through the review of their JHAs, unless otherwise denoted in the contract. General hazards associated with work performed on site can be identified through Section 7 Hazard Assessment.

6. SITE OVERVIEW

6.1 SITE CHARACTERIZATION AND ANALYSIS

An evaluation of work activities is performed for each project including a hazard assessment for each task or activity to identify associated hazardous conditions, appropriate staff member protection methods, and PPE. The evaluation of potential site conditions and hazards is ongoing and continues throughout the duration of the project. Site characterization information can be found in the project-specific JHA with additional information regarding the SOW. All hazard assessment and mitigations are selected and documented through project-specific JHAs.

6.1.1 Preliminary Evaluation

A preliminary evaluation of a site's characteristics shall be approved by a qualified person familiar with site conditions to aid in the selection of appropriate protection methods prior to site entry. The qualified person is an individual who possesses extensive knowledge, training, and experience in relation to the possible characteristics and hazards of the site, and has demonstrated their ability to resolve problems related to the subject matter, work, or project.

A more detailed evaluation of the site's specific characteristics shall be performed by the RSM and PM to further identify existing site hazards and to further aid in the selection of the appropriate engineering controls and PPE for the tasks to be performed.

6.2 SITE DESCRIPTION AND WORK AREAS

A site description and identified work areas will be outlined in the scope of associated JHAs. Project teams are to provide this information on project documents to assist the RSM reviewing the documents with identifying hazards and appropriate mitigations.

6.3 REQUIRED INFORMATION

The information listed below, to the extent of which is available, shall be obtained by Haley & Aldrich project teams prior to completing the JHA and allowing any staff members to enter the site:

- Location and approximate size of the site.
- Description of the activities or job tasks being performed.
- Duration of planned staff member activity.
- Site topography and accessibility by air and roads.
- Safey and health hazards expected at the site.
- Pathways for hazardous substance dispersion.
- Present status and capabilities of emergency response teams that would aid hazardous waste clean-up site at the time of an emergency.
- Hazardous substances and health hazards involved or expected at the site, and their chemical and physical properties.

7. HAZARD ASSESSMENT

7.1 HAZARD ASSESSMENT

Hazard assessments are performed as part of project preparation for all projects that Haley & Aldrich performs. A hazard assessment is intended to identify and describe safety and health hazards associated with site work. All project teams are expected to identify hazards associated with the site, the tasks, and simultaneous operations. Assessments are performed through a JHA. All JHAs are to be included in project safety folders, provided to all on-site personnel for their applicable tasks, and reviewed by field staff prior to mobilization. Modifications to JHAs are to be communicated with the entire project team as soon as they occur, and JHAs are to be reviewed daily during the execution of the work associated with the JHA. With this information, risks are then eliminated or effectively controlled.

All project-specific and task-specific JHAs are to be modified when:

- The SOW is changed by adding, eliminating, or modifying tasks.
- New methods of performing site tasks are selected.

- Observation of the performance of site tasks results in a revised characterization of the hazards.
- New hazards are identified.
- Exposure data indicate changes in the concentration and/or likelihood of exposure.
- Change in personnel or subcontractors.
- When a near miss or incident occurs.

7.2 CHEMICAL HAZARDS

Haley & Aldrich staff members have the potential to interact with a variety of chemical hazards while working in the field. Chemical hazards must be identified and addressed on a project-by-project basis. Project-specific personal air monitoring plans shall be developed and described in the project-specific JHA. Additionally, other routes of exposure to chemical hazards (e.g., absorption, injection, ingestion) and associated controls will be addressed in either project-specific or task-specific JHAs. Personal air monitoring plans and JHAs must be reviewed and approved by a RSM. All project-specific or task-specific JHAs are to be modified when new chemical hazards are identified.

For more information on chemical hazards and exposure limits, see Appendix A – Chemical Hazards or appropriate Safety Data Sheets.

7.3 PHYSICAL HAZARDS

Haley & Aldrich staff members have the potential to interact with a variety of physical hazards while working in the field. Physical hazards will be identified and addressed in either project-specific or task-specific JHAs. JHAs must be reviewed and approved by a RSM and PM. All project-specific or task-specific JHAs are to be modified when new physical hazards are identified.

For more information on physical hazards, see Appendix B – Physical Hazards.

7.4 WEATHER HAZARDS

Haley & Aldrich staff members have the potential to interact with a variety of weather-related hazards while working in the field. Weather-related hazards will be identified and addressed in either project-specific or task-specific JHAs. JHAs must be reviewed and approved by a RSM and PM. All project-specific or task-specific JHAs are to be modified when new weather-related hazards are identified.

For more information on weather-related hazards, see Appendix C – Weather-Related Hazards.

7.5 BIOLOGICAL HAZARDS

Haley & Aldrich staff members have the potential to interact with a variety of biological hazards while working in the field. Biological hazards will be identified and addressed in either project-specific or task-specific JHAs. JHAs must be reviewed and approved by a RSM and PM. All project-specific or task-specific JHAs are to be modified when new biological hazards are identified.

For more information on biological hazards, see Appendix D – Biological Hazards.

8. PROTECTIVE MEASURES

8.1 IMPLEMENTING CONTROLS

The hierarchy of controls should always be used to evaluate how hazards can be mitigated. When possible, staff members should look to eliminate and substitute hazards or design engineering controls before implementing administrative controls and PPE. To effectively assess hazards and best select mitigations, field staff must be engaged in discussion, as they have the best understanding of the hazardous conditions and insights into how they can be controlled.

Staff members are to review these controls and use the JHAs to document hazards and their associated controls.

8.2 FIT FOR DUTY

It is important for those planning the work to support staff members to be fit for duty and complete their work in a safe manner. See OP1063 – Fit for Duty for more information. Below are guidelines to maintain a safe environment and encourage staff to be fit for duty:

- Scheduling enough staff on sites so they are able to rotate work.
- Allowing staff who have worked multiple shifts in a row to take time off.
- Giving staff members the weekend, when possible, to rest and prepare for the following work week.
- Ensuring staff members are physically capable of completing work tasks assigned to them.
- Being mindful of total worker health, ensuring that stress, emotional state, and conditions outside of work are considered.

Staff members are responsible to show up to work:

- Not under the influence of drugs or alcohol, including prescription medicines that may interfere with their ability to perform work safely.
- Symptom free for 24 hours from any contagious illnesses.
- Adhering to work restrictions prescribed by a medical professional.
- Fit for duty to perform job tasks assigned.

When there is a risk of fatigue, it is the responsibility of the PM and RSM to develop controls for staff to be fit for duty, and to build those mitigations into the project-specific JHA.

8.3 PERSONAL PROTECTIVE EQUIPMENT (PPE)

PPE is used to provide adequate personnel protection only after feasible engineering and administrative control options have been exhausted. See OP1024 – Personal Protective Equipment for more information. All PPE is provided by Haley & Aldrich to users and is selected to ensure that it is constructed and designed to protect staff members against known or anticipated hazards. Selected PPE should properly and appropriately fit the staff member. These provisions follow the requirements of all applicable regulations (29 CFR 1910.120(c)(5), 29 CFR 1910.132 to 1910.140, and 29 CFR 1926.28). It is the responsibility of the user to ensure that PPE is used and maintained in a sanitary and reliable condition.

All staff members at Haley & Aldrich are required to wear standard PPE listed below, unless a hazard assessment has determined the hazard related to some of the items listed below is not present. This includes but is not limited to:

- Hard hat;
- High visibility vest;
- Safety glasses;
- Cut-resistant gloves (A2);
- Hearing protection;
- Safety-toed boots; and
- Work clothing consisting of, at a minimum, long pants and a short-sleeve shirt.

The selection of additional PPE and other controls is based on an evaluation of the hazards present and job activities being performed. This selection is outlined in the PPE Hazard Assessment as part of the project-specific JHAs. PPE may vary from task to task, and all personnel engaged in the project work activities must use the appropriate level of protection as required by the activity to be performed. If an office does not have the necessary PPE for the work being conducted, contact the Local Health & Safety Coordinator.

If during the planned activities unique conditions are observed, an updated hazard assessment will be performed and reviewed by the PM and RSM. No changes in PPE will be conducted without a hazard assessment, review and approval by RSM and PM, and documentation of the proper training, if applicable.

8.4 LEVELS OF PERSONAL PROTECTIVE EQUIPMENT

8.4.1 Level D Protection

Level D protection should be used when:

- The atmosphere contains no known hazard; and
- Work functions preclude splashes, immersion, or the potential for unexpected inhalation of, or contact with, hazardous levels of any chemicals.

The following constitute Level D equipment which may be used when appropriate and selected by a RSM or PM:

- Coveralls.
- Gloves.
- Boots/shoes that meet ASTM International (ASTM) F2413.
- Boots, outer, chemical-resistant (disposable).
- Safety glasses or chemical splash goggles.
- Hard hat.
- Face shield.

8.4.2 Level C Protection

Level C protection should be used when:

- The atmospheric contaminants, liquid splashes, or other direct contact will not adversely affect or be absorbed through any exposed skin;
- The types of air contaminants have been identified, concentrations measured, and an air-purifying respirator (APR) is available that can remove the contaminants; and
- All criteria for the use of an APR are met.

APRs can be used only when the contaminant(s) is known, cartridges/canisters exist, and concentrations are within the substance-specific standard guidelines or within the maximum use concentration (MUC) for the APR used. An appropriate cartridge/canister selection process and change schedule are outlined in our Respiratory Protection Program, according to the respiratory protection regulation (29 CFR 1910.134(d)(3)(iii)(B)(2)). See Respiratory Protection below for more information.

If warning properties (chemical odors, tastes, or physical irritation) are detected, staff members must immediately leave the work area and notify their PM and RSM.

The following constitute Level C equipment and may be used when appropriate and selected by a RSM:

- Full-face or half-mask, air purifying respirators (National Institute for Occupational Safety and Health [NIOSH] approved).
- Hooded chemical-resistant clothing (overalls; two-piece chemical-splash suit; disposable chemical-resistant overalls).
- Coveralls.
- Gloves, outer, chemical-resistant.
- Gloves, inner, chemical-resistant.
- Boots/shoes that meet ASTM F2413.
- Boot-covers, outer, chemical-resistant (disposable).
- Hard hat.
- Escape mask.
- Face shield.

8.4.3 Level B Protection

This level of protection is appropriate when the highest level of respiratory protection is necessary, but a lesser level of skin protection is needed. Level B protection should be used when:

- The type and atmospheric concentration of substances have been identified and require a high level of respiratory protection, but less skin protection;
- The atmosphere contains less than 19.5 percent oxygen; or

- The presence of incompletely identified vapors or gases is indicated by a direct-reading organic vapor detection instrument, but vapors and gases are not suspected of containing high levels of chemicals harmful to skin or capable of being absorbed through the skin.

The following constitute Level B equipment and may be used when appropriate and selected by a RSM:

- Positive pressure, full-facepiece, self-contained breathing apparatus (SCBA), or positive pressure supplied air respirator with escape SCBA (NIOSH approved).
- Hooded chemical-resistant clothing (overalls and long-sleeved jacket; coveralls; one- or two-piece chemical splash suit; disposable chemical-resistant overalls).
- Coveralls.
- Gloves, outer, chemical-resistant.
- Gloves, inner, chemical-resistant.
- Boots/shoes that meet ASTM F2413.
- Boot-covers, outer, chemical-resistant (disposable).
- Hard hat.
- Face shield.

8.4.4 Level A Protection

This level of protection should be selected when the greatest level of skin, respiratory, and eye protection is required. Level A protection should be used when:

- The hazardous substance has been identified and requires the highest level of protection for skin, eyes, and the respiratory system based on either the measured (or potential for) high concentration of atmospheric vapors, gases, or particulates; or the site operations and work functions involve a high potential for splash, immersion, or exposure to unexpected vapors, gases, or particulates of materials that are harmful to skin or capable of being absorbed through the skin;
- Substances with a high degree of hazard to the skin are known or suspected to be present, and skin contact is possible; or
- Operations are being conducted in confined, poorly ventilated areas, and the absence of conditions requiring Level A have not yet been determined.

The following constitute Level A equipment and may be used when appropriate and selected by an RSM:

- Positive pressure, full face-piece SCBA, or positive pressure supplied air respirator with escape SCBA, approved by NIOSH.
- Totally encapsulating chemical-protective suit.
- Coveralls.
- Long underwear.
- Gloves, outer, chemical-resistant.
- Gloves, inner, chemical-resistant.

- Boots/shoes that meet ASTM F2413.
- Hard hat (under suit).
- Disposable protective suit, gloves, and boots (depending on suit construction, may be worn over totally encapsulating suit).

8.5 INSPECTION, CLEANING, MAINTENANCE, AND STORAGE

PPE must be inspected prior to use and must be cleaned and maintained at regular intervals, as regulatory required, and as required by the manufacturer so that the PPE provides the requisite protection. PPE should be cleaned as needed, after each use, and before storage. PPE must be stored in a manner to avoid conditions that could result in damage, such as exposure to heat, light, moisture, or contamination (e.g., dirt, dust, or impacted media). PPE that is either defective or damaged must not be used and must be discarded or removed from service until repaired by a qualified person or the manufacturer. It is also important to ensure that contaminated PPE that cannot be decontaminated is not reused and is disposed of in a manner that protects staff members from exposure to hazards. For more information on decontamination, see Section 14 Decontamination.

PPE that is not grossly contaminated can be bagged and disposed in regular trash receptacles.

8.6 TRAINING AND PROPER FITTING

Staff members are trained on when PPE is necessary, what PPE is necessary for certain work, how to properly don, doff, adjust, and wear PPE, the limitations of the PPE, what to do if the PPE fails, the proper care, maintenance, useful life, and disposal of the PPE, and at the end of any training, should be able to demonstrate how to use PPE properly. Training is included as part of the Annual 8-Hour HAZWOPER Refresher for those that maintain a certificate. Refresher training must be performed annually for all staff members who use PPE, and more frequently, as necessary, in the event of any of the following:

- Changes in the workplace or types of PPE used which would make the previous training obsolete;
- Inadequacies in the staff's knowledge or use of PPE indicate that the staff has not retained the requisite understanding or skill; and
- Any other situation that arises in which retraining appears necessary to ensure safe use of PPE.

Staff members have the responsibility to communicate any issues with the fit or use of PPE to a member of the Health & Safety Team to ensure proper PPE is provided for hazards that are present.

8.7 DONNING AND DOFFING

Proper donning and doffing procedures are dependent on the type of PPE being worn by the staff member. Donning and doffing procedures are shown in Annual 8-Hour HAZWOPER Refresher courses and are to be demonstrated by local health and safety leads when PPE is given to a staff member for the first time.

8.8 EVALUATION OF EFFECTIVENESS

Staff members and RSMs are responsible for communicating the effectiveness of PPE with the Health & Safety Team. RSMs or their designees routinely visit projects sites to evaluate hazards present, and compare them to project documents developed, to ensure that the best mitigations are in place to protect staff members.

8.9 LIMITATIONS

Some PPE may create limitations when working in extreme environments, including extreme heat. Staff members are to review OP1015 – Heat Stress* as part of the hazard assessment with their project team ahead of work starting to identify appropriate PPE when there is concern for working conditions.

- *Note - OP1015 Heat Stress is currently in review to align with Federal OSHA's proposed heat stress ruling, while still meetings requirements of State OSHA heat safety plans. Staff must use the Heat Injury and Illness Prevention Plan (linked as OP1015 - Heat Stress) above to reduce risk when working in the heat.

8.10 RESPIRATORY PROTECTION

Before beginning any work that generates airborne contaminants, chemical, or dust, respiratory protection must be assessed. For more information on medical evaluation and clearance, fit testing, respirator maintenance and repair, storage, cartridge selection, and training, see OP1023 – Respiratory Protection.

Staff members that wish to wear respirators for voluntary use must complete a Voluntary Use Respirator Form. Contact the Health & Safety Team for more information.

8.11 HEARING CONSERVATION

Staff members exposed to, or with the potential to be exposed to, an 8-hour time-weighted average (TWA) sound level equal to or greater than 85 dBA must participate in a hearing conservation program. See OP1031 – Hearing Conservation for more information. All exposed personnel receive awareness training about the hazards of noise, the procedures to properly use and maintain hearing protection, and the importance of annual testing.

Hearing protection is made available when noise exposures equal or exceed an 8-hour TWA sound level of 85 dBA. Hearing protection is required when the 8-hour TWA sound level is greater than 90 dBA. Where noise exposure meets or exceeds this level, noise is listed as a physical hazard in the JHA for the tasks or operation, and hearing protection is included as one of the control measures.

Hearing protection is also required for any staff members who have not yet had a baseline audiogram or who have experienced a standard threshold shift and are exposed to an 8-hour TWA sound level greater than 85 dBA.

9. TRAINING REQUIREMENTS

9.1 HEALTH AND SAFETY TRAINING REQUIREMENTS

All Haley & Aldrich Staff members must be trained to a level required by their job function and responsibility prior to working on non-HAZWOPER and HAZWOPER sites. Haley & Aldrich staff members, contractors, subcontractors, and consultants who have the potential to be exposed to contaminated materials or physical hazards must complete the training described in the following sections.

Corporate Health & Safety maintains documentation of training records. PMs must review staff member training records to ensure they are trained and compliant with OSHA requirements for the tasks being performed. PMs may also access training records at the request of clients and/or OSHA through Corporate Health & Safety.

9.2 40-HOUR HAZWOPER

The 40-Hour HAZWOPER course provides instruction on the nature of hazardous waste work, protective measures, proper use of PPE, recognition of signs and symptoms which might indicate exposure to hazardous substances, and decontamination procedures. All staff members who are required to participate in work on hazardous waste sites- including equipment operators, general laborers, and supervisors- will be required to complete an initial 40 hours of training as specified by 29 CFR 1910.120 (e)(3).

9.3 8-HOUR ANNUAL REFRESHER

Personnel who complete the 40-hour health and safety training and are required to maintain its validity beyond one calendar year are required to attend an annual 8-hour refresher training course as per 29 CFR 1910.120 (e)(8). Haley & Aldrich maintains records of all staff members 40-hour HAZWOPER training status, and when required can provide proof of this status with a certificate of completion.

9.4 8-HOUR SUPERVISOR

The Haley & Aldrich SSO directly responsible for hazardous waste operations, or who supervise staff members engaged in these operations, must have eight additional hours of Supervisor training in accordance with 29 CFR 1910.120 (e)(4) Supervisor Training. This includes, but is not limited to, accident reporting/investigation, regulatory compliance, work practice observations, emergency response procedures, and maintenance of an internal Safety & Health program.

9.5 ADDITIONAL TRAINING FOR SPECIFIC PROJECTS

Haley & Aldrich PMs must ensure field staff are trained in any additional OSHA requirements for tasks they are required to perform, including information on specific instruments, equipment, and construction hazards, and can demonstrate competence. Specialized training will be provided at the awareness level, and competent person level where necessary, to staff members before engaging in the specific work activities. Examples of trainings offered include but are not limited to, Hearing Conservation, Respiratory Protection, Trenching & Excavations, Confined Space Entry (entrant, supervisor, attendant), Heavy Equipment (aerial lifts, forklifts, etc.), First Aid/CPR, Fall Protection, Nuclear Density Gauges, and Hazardous Substances specified in 1926 Subpart Z.

10. MEDICAL SURVEILLANCE

10.1 MEDICAL SURVEILLANCE

A medical surveillance program is present when staff members have the potential to be exposed to contaminants or hazards that cause adverse health effects. This program ensures that staff are protected from exposure to hazardous materials or health hazards that have been identified and when a potential exposure has occurred. This section follows the requirements as presented by OSHA in 29 CFR 1910.120(f), with additional guidelines for

substance-specific requirements in 29 CFR 1910 1001-1052 if they are identified as present on-site. Additional information can be referenced in OP1009 – Workplace Medical, as listed below:

- Staff members covered
- Exam Frequency
- Exam Content
- Subpart Z Hazards and Additional Testing
- Physical Exam, Review, Written Opinion
- Recordkeeping Requirements
- Hearing Conservation
- Bloodborne Pathogens

11. AIR MONITORING PLANS AND EQUIPMENT

To determine the types and quantities of airborne contaminants that staff members may be exposed to on site, a project-specific evaluation of chemical hazards should be performed. Chemical hazards will be evaluated to develop a personal air monitoring plan with controls in accordance with applicable OSHA regulations. Personal air monitoring plans must be developed per project to incorporate project-specific details such as scope, chemical hazards, expected concentrations, site conditions, and available monitoring equipment, and must be documented in project-specific JHAs. All personal air monitoring plans must be reviewed and approved by the RSM and PM prior to starting work. Additional information about air monitoring equipment can be found in operating procedures:

- OP1004 – Operation and Calibration of PID
- OP1005 – Operation and Calibration of FID
- OP1006 – Operation of Draeger Gas Detector Pump
- OP1007 – Operation and Calibration of Combustible Gas Indicators

For information on chemical hazards and exposure limits from commonly encountered chemicals on site, see Appendix A – Chemical Hazards or appropriate Safety Data Sheets.

11.1 INITIAL ENTRY AND BACKGROUND MONITORING

Initial exposure monitoring and/or background monitoring should be conducted to measure staff member exposure to hazardous chemicals or atmospheres where the potential for the following conditions may exist:

- Possible Immediately Dangerous to Life or Health (IDLH) conditions;
- Potential for exposure near or over permissible exposure limits and/or action levels;
- Potential for exposure to any radioactive source;
- Oxygen rich or deficient environments;

- Flammable atmospheres;
- Or any other dangerous conditions on site.

Initial exposure monitoring should be conducted in accordance with applicable OSHA regulations for monitoring frequency, methodology, and notification procedures. Generally, an initial determination may include a representative sample of the staff member's exposure who is exposed to the greatest concentrations of the chemical hazard of concern. Contact your regional H&S representative to perform this process.

When conducting initial or background monitoring, staff must be protected from potential exposure by using appropriate engineering controls or PPE.

11.2 PERIODIC MONITORING

Periodic monitoring shall be established in accordance with applicable OSHA standards for each chemical hazard. The frequency of monitoring shall be further established by the results of initial exposure determinations, changes to work processes, controls, personnel changes which may result in new or additional exposure, or when there is any other reason to suspect a change in exposure.

The following examples are changes to site conditions that may impact and increase potential exposure to air contaminants or health Hazards:

- Work in a new area of a site.
- Work with new materials that may present a health hazard.
- The start of new tasks or work operations (i.e., soil vapor intrusion sampling vs. confined space entry).
- When staff are handling leaking drums or containers in working areas with liquid contamination.

11.3 AIR MONITORING EQUIPMENT

The air monitoring equipment below may be used in personal air monitoring plans for the associated contaminant or health hazard. See JHA for site-specific equipment and usage.

Equipment	Detectable Contaminants / Hazard
PID 10.2eV or 11.7eV	Volatile Organic Compounds (VOCs)
OV Monitor	Volatile Organic Compounds (VOCs)
Gas Chromatograph	Volatile Organic Compounds (VOCs)
Colorimetric Tubes	Volatile Organic Compounds (VOCs)
Combustible Gas Indicator	LEL
Multiple Gas Detector	LEL / O_2 / H_2S / CO
RAM (Real-time Air Monitor)	Particulates
Air Sampling Pump	Chemical specific

11.3.1 Calibration, Functionality Tests, and Use of Air Monitoring Equipment

The calibration of all monitoring equipment is required in accordance with manufacturers requirements and Haley & Aldrich Operating Procedures listed above in Section 10 and site-specific requirements (e.g., at the

beginning and end of each workday). Calibration must be done by a qualified laboratory- at least annually- unless manufacturer recommendations differ, or you encounter problems. Rental companies will perform calibration services.

Daily functionality tests, also known as bump tests, of equipment shall be documented in the field notes or Daily Field Report. Documentation should include:

- Date/time,
- Equipment ID number,
- Zero reading, if applicable, and
- Reading obtained with calibration gas or gases.

The required equipment listed above must be in good working condition and be available on-site at all times during applicable work activities, as outlined in the JHA. Work shall not commence unless the equipment is present, calibrated, and operable.

12. SITE CONTROL

The goal of a site control plan is to establish procedures that minimize potential worker exposure to hazardous substances, protect the public from the site's hazards, and provide site security. Understanding the site control program is crucial in the event of an emergency. The degree of site control necessary depends on site characteristics, site size, and the surrounding community. Haley & Aldrich incorporates the elements required for a site control plan in the JHA. This includes information to access a site map that identifies work zones, site communications, applicable Haley & Aldrich SOPs, and the location of the nearest medical facility. Please reference the project-specific JHA for site control information.

All operating procedures, such as OP1025 - Signs, Signals, and Barricades, OP1043 - Site Traffic Control, and OP1046 - Site Control, are available to staff members at any time through our internal site. Operating procedures can be provided to any external party at any time upon request.

12.1 GENERAL SITE CONTROL INFORMATION

One of the main goals of a site control plan is to establish procedures that minimize potential exposure to hazardous substances that workers may encounter before the initiation of a project as described by 29 CFR 1910.120 (d)(1). Understanding the site control program is crucial in the event of an emergency. The degree of site control required depends on site characteristics, site size, and the surrounding community. The information provided below identifies all elements used to control the activities and movements of people and equipment at the project site.

12.2 SITE MAP WITH DESIGNATED WORK ZONES

See project-specific JHAs for site map with identified work zones and exit locations. Language to support JHA development related to site control can be found in Appendix B – Physical Hazards.

12.3 SITE COMMUNICATIONS

Haley & Aldrich encourages all staff members to communicate face to face with one another. If face-to-face communication is not feasible, conversation over cellular phones is acceptable. Haley & Aldrich does provide other forms of communication to staff members, such as satellite phones, radios, or walkie-talkies, when this is necessary. If staff are entering areas where hearing is of concern due to noise, PPE, or other reasons, staff will develop agreed upon hand signals to communicate.

12.4 LOCATION OF MEDICAL ASSISTANCE

See project-specific JHA for information about, and route to, the nearest medical facility. Additional emergency contact information can also be accessed on the same JHA.

13. EMERGENCY RESPONSE PLAN

13.1 EMERGENCY RESPONSE PLANS

Emergency Response Plans are developed prior to the beginning of work for all uncontrolled hazardous waste sites. Emergency response plans help facilitate anticipated emergencies on hazardous waste sites by providing clear direction for a course of action when each anticipated emergency occurs. Additionally, emergency response plans inform staff members of safe practices surrounding other hazards on the project, such as safe distances from machinery, and site security.

The major categories of emergencies that could occur during project work are:

- Fire(s)/Combustion
- Hazardous Materials Event
- Medical Emergency
- Natural Disaster

A detailed list of emergency types and response actions are summarized in Appendix E – Emergency Recognition And Prevention. Emergency response plans are included in the project-specific JHA.

13.2 HALEY & ALDRICH AS THE CONTROLLING EMPLOYER

Haley & Aldrich has developed the following Emergency Response plan to adhere to the requirements specified in 20 CFR 1910.120(l). This plan is applicable to all project sites where Haley & Aldrich is designated as the controlling employer of a project.

13.2.1 Pre-Emergency Planning

Before the start of field activities, the PM must prepare for emergencies to include the following:

- Meet with the controlling employer, subcontractor, or client to discuss emergency procedures in the event a person is injured.

- Develop, review, and communicate actions for specific scenarios, which will be incorporated into the project-specific JHA.
- The SSO will establish evacuation routes and assembly areas for the sites.
- All personnel entering the site, including subcontractors and visitors, will be informed of evacuation routes and assembly areas. Emergency response documents will be available for all to access on site. Additional copies can be provided upon request.
- Haley & Aldrich staff members should perform an emergency drill during the initial stages of the project. This can either be a physical drill performed by the field staff members while on the project site, or a step-by-step walkthrough of where to go and what to do with the entire project team on a call prior to beginning the work.

13.2.2 Personnel, Roles, Lines of Authority, and Communication

Site personnel must be trained on the use of the site emergency communication plan during site orientation. Emergency telephone numbers are accessible in the project-specific JHA. The SSO is responsible for establishing the communication network and discussing it with all staff prior to the start of work. This includes signals agreed upon to identify emergency situations which will be documented in the project-specific JHA. It is the responsibility of the SSO to regularly remind staff of the lines of communication, and ensure subcontractors are aware and understand the meaning of each form of communication.

In the event of an emergency, work must be stopped, and site personnel must be notified of the situation immediately. The scene should be surveyed to determine next steps. The Haley & Aldrich SSO will follow the procedure listed below:

- Evaluate the incident and assess the need for assistance and/or evacuation.
- Take appropriate measures to stabilize the incident scene.
- Assign roles and responsibilities for emergency response, as needed.
- Contact applicable Emergency Services if necessary.
- Notify other members of the project team (PM, RSM, Field Service Manager).

13.2.3 Safe Distances and Places of Refuge

Safe distances and places of refuge will be identified in the project-specific JHA.

13.2.4 Site Security and Control

Site security and control will be identified in the project-specific JHA.

13.2.5 Evacuation Routes and Procedures

Evacuation routes will be highlighted on the site map included in the project-specific JHA and discussed regularly during work kickoff meetings. Emergency procedures will be developed as part of the project documents.

13.2.6 Emergency Medical Treatment and First Aid

Haley & Aldrich field staff are trained in First Aid/CPR/AED use. Emergency services will be contacted immediately if an injury greater than first aid is required. Minor injuries to Haley & Aldrich staff that only require first aid should be addressed on site, reported to the SSO and other members of the project team, and then managed by Acuity. Minor injuries to contractors should be reported to the SSO, addressed on site, and then be managed by the contractor themselves.

13.2.7 Emergency Alerting and Response Procedures

In the event of an emergency, Haley & Aldrich will communicate with everyone on site. Emergency Response procedures will regularly be reviewed during morning tailgate meetings, and other scheduled & unscheduled project communication. In instances where emergency services are required, they will be contacted immediately and supported from the time they arrive on site until the emergency has been addressed.

13.2.8 PPE and Emergency Equipment

Haley & Aldrich provides all staff members with OSHA-approved first aid kits. Additional emergency response PPE and equipment is identified in project-specific or task-specific JHAs.

13.2.9 Critiques of Response and Follow-Up

Haley & Aldrich will exercise the use of reporting, incident investigation, and root cause analysis to identify learning opportunities related to emergency response. Any findings from the incident investigation or root cause analysis process will be used to improve emergency response plans for the project where the incident occurred, if still active, and for future emergency response plans on all other projects.

13.3 MULTI-EMPLOYER PROJECT SITES

Haley & Aldrich is often not the controlling employer on project sites. When a project requires a staff member to enter or work on a site where there are multiple employers, the project team must review the controlling employer's emergency response plan and incorporate the required information into our own emergency response plan.

The following information should be identified in the controlling employer's emergency response plan, and be incorporated into our own plan and procedures:

- Evacuation Alarm;
- Evacuation Routes;
- Evacuation Response Drills;
- Contractors written emergency and contingency plans;
- Discuss exit routes and fire extinguisher locations; and
- Confirm presence and access to emergency response supplies.

13.3.1 Procedures for Reporting a Fire or Other Emergency

- Haley & Aldrich requires staff members to discuss reporting procedures on the JHA Cover Page.
- In the event of a fire or other emergency, staff must immediately evacuate or remove themselves from the dangerous situation.
- Once in a safe location, staff should contact 911 if a life-threatening situation is present. If not:
- The staff member should immediately contact the safety coordinator of the controlling employer and follow their direction.

13.3.2 Procedures for Emergency Evacuation

Haley & Aldrich requires staff members to incorporate a site map into the project-specific JHA documents that designates work zones, and highlights emergency exit routes.

13.3.3 Procedures to Account for All Staff After an Evacuation

Staff members are responsible for knowing what other Haley & Aldrich staff are on the same project site at a time. The staff should first contact the other staff on site, and then contact the PM to confirm all staff are accounted for.

13.3.4 Procedures for Staff Performing Rescue or Medical Duties

If extensive rescue or medical duties are needed, staff are to call Acuity for non-emergency situations (1-888-397-8099), or 911 for emergency situations. Haley & Aldrich staff members are not to perform extensive rescue or medical duties.

13.3.5 Name or Title of Who is Responsible for the Plan

The PM is the responsible party for all aspects of the project. If a staff member does not think the project documents are sufficient, they can reach out to the PM or Health & Safety.

14. DECONTAMINATION AND DISPOSAL METHODS

14.1 GENERAL DECONTAMINATION AND DISPOSAL INFORMATION

All possible and necessary steps shall be taken to reduce or minimize contact with chemicals and contaminated/ impacted materials while performing field activities. Decontamination procedures are designed to achieve an orderly, controlled removal or neutralization of contaminants that may accumulate on personnel or equipment. These procedures minimize worker contact with contaminants, and protect against the transfer of contaminants outside designated work zones. Any procedures developed will be communicated to staff members and implemented before any staff members or equipment may enter areas on site where potential for exposure to hazardous substances exists.

All staff members leaving a contaminated area shall be appropriately decontaminated, and all contaminated clothing and equipment leaving a contaminated area shall be appropriately disposed of or decontaminated.

Decontamination areas and procedures will change from site to site, and it is the responsibility of the field staff, the PM, and Health & Safety to communicate with the Controlling Employer to understand appropriate procedures for each site. If procedures are changed as a result of inspection and monitoring, all affected staff are to be notified of these changes.

14.1.1 Required Information for Projects

Decontamination will be specific to each project. PMs and their teams are expected to incorporate the following information into the project-specific JHA.

- Location of decontamination areas
  - Geographical areas designated for decontamination that will minimize exposure of uncontaminated staff members and equipment.
- Equipment and solvents to be used for decontamination, such as, but not limited to:
  - Acetone
  - Alconox soap
  - Brushes
  - Disposal bags
  - 5-gallon buckets
  - Distilled water
  - Drums
  - Hexane
  - Methanol
  - Paper towels
  - Polyethylene sheeting
  - Pressure/steam cleaner
  - Tap water
  - Wash tubs
- Showers and change rooms
  - If temperature conditions prevent the effective use of water, then other effective means for cleansing shall be provided and used.
  - Establishing showers and changing rooms are the responsibility of the Controlling Employer.
- Disposal methods
  - Procedures for disposal of contaminated materials, decontamination waste, and single use PPE shall meet applicable client, local, state, and federal requirements.
  - PPE that is not grossly contaminated can be bagged and disposed in regular trash receptacles.
  - PPE that is grossly contaminated must be bagged and sealed, and field personnel should communicate with the PM to determine proper disposal.

14.1.1.1 Personal Hygiene Safeguards

PMs must also make staff members aware of the following minimum personal hygiene safeguards that shall be adhered to:

- No smoking or tobacco products in any project work areas.
- No eating or drinking in the exclusion zone.
- It is required that personnel present on site wash hands before eating, smoking, taking medication, chewing gum/tobacco, using the restroom, applying cosmetics, and before leaving the site for the day.

It is recommended that personnel present on site shower or bathe at home at the end of each day of working on the site.

14.1.1.2 Unauthorized Staff Members

PMs must make staff members aware of who is performing work that requires the staff member to follow decontamination processes. All other staff members are considered unauthorized staff and shall not perform HAZWOPER work. Unauthorized staff are not to remove protective clothing or equipment from change rooms or enter any decontamination areas.

14.1.2 Personal Protective Equipment and Clothing

Protective clothing and equipment shall be decontaminated, cleaned, laundered, maintained, or replaced as needed to maintain their effectiveness. Staff members whose non-impermeable clothing becomes wetted with hazardous substances shall immediately remove that clothing and proceed to shower. The clothing shall be disposed of or decontaminated before it is removed from the work zone. See Section 14.1.1 Equipment and solvents to be used for decontamination, and Standard Personal Decontamination Procedures below for more information on general decontamination processes. All information must be communicated to staff members before the start of the project, and as any changes are made to the decontamination process.

Commercial laundries or cleaning establishments that decontaminate protective clothing or equipment shall be informed of the potentially harmful effects of exposures to hazardous substances.

14.2 STANDARD PERSONAL DECONTAMINATION PROCEDURES

Outer gloves and boots should be decontaminated periodically as necessary and at the end of the day. Brush off solids with a hard brush and clean with soap and water or another appropriate cleaner whenever possible. Remove inner gloves carefully by turning them inside out during removal. Wash hands and forearms frequently. It is good practice to wear work-designated clothing while on-site which can be removed as soon as possible. Non-disposable overalls and outer work clothing should be bagged onsite prior to laundering. If gross contamination is encountered on-site, contact the PM and RSM to discuss proper decontamination procedures.

The steps required for decontamination will depend upon the degree and type of contamination but will generally follow the sequence below.

1. Rinse boots and gloves of gross contamination.
2. Scrub boots and gloves clean.
3. Rinse boots and gloves.

4. Remove outer boots (if applicable).
5. Remove outer gloves (if applicable).
6. Remove and wipe clean hard hat.
7. Remove Tyvek coverall (if applicable).
8. Remove respirator, wipe clean and store (if applicable).
9. Remove inner gloves (if outer gloves were used).

PPE that is not grossly contaminated can be bagged and disposed in regular trash receptacles.

14.3 SMALL EQUIPMENT DECONTAMINATION

Pretreatment of heavily contaminated equipment may be conducted as necessary:

1. Remove gross contamination using a brush or wiping with a paper towel.
2. Soak in a solution of Alconox and water (if possible).
3. Wipe off excess contamination with a paper towel.

Standard decontamination procedure:

1. Wash using a solution of Alconox and water.
2. Rinse with potable water.
3. Rinse with methanol (or equivalent).
4. Rinse with distilled/deionized water.

Inspect the equipment for any remaining contamination and repeat as necessary.

14.4 MONITORING THE EFFECTIVENESS OF DECONTAMINATION PROCEDURES

Project teams and Health & Safety are responsible for routinely auditing the effectiveness of decontamination procedures through regular site visits and project audits. Visual examination is used to ensure that procedures are implemented as described, and that they appear to control the spread of contaminants under changing conditions.

Air samples can be taken in the clean zone to ensure that airborne contaminants have not spread to clean areas of the facility. Surface samples can be taken from the inside surfaces of PPE, from decontaminated equipment, and from surfaces within clean areas of the facility to ensure that decontamination and control procedures are performing as anticipated.

Results of the inspections of decontamination procedures and documentation of any action taken to correct deficiencies are recorded and stored in Health & Safety's reporting tool.

15. PERMIT-REQUIRED CONFINED SPACE ENTRY

15.1 GENERAL CONFINED SPACE INFORMATION

OSHA defines a confined space as a space that is large enough for an staff member to enter, limited means of entry and exit, and not designed for continuous staff member occupancy. Examples of these spaces include manholes, stacks, pipes, storage tanks, pits, sumps, hoppers, and bins. Before any staff are allowed to enter or

even break the plane of a confined space, OSHA requires that a competent person is responsible for determining whether the confined space is a Permit-Required Confined Space (PRCS), using the OSHA decision flow chart found in 29 CFR 1910.46 Appendix A. An RSM must be contacted if you will be breaking the plane (putting any body part inside the entrance of a confined space) or entering a confined space.

15.2 PERMIT-REQUIRED CONFINED SPACE INFORMATION

PRCS are confined spaces where potential hazards like hazardous atmospheres, engulfment, or oxygen deficiency are present, requiring a formal permit to be issued before anyone can enter, ensuring proper safety procedures are followed and monitored by designated personnel. Haley & Aldrich maintains a PRCS program to identify and inform the involved staff members of the specific hazards of the PRCS. Before Haley & Aldrich staff are allowed to break the plane of a PRCS, the RSM must be engaged to oversee the permitting process. For more information on confined space entry, see OP1060 – Confined Space Entry.

15.2.1 Hazardous Atmosphere Permit-Required Confined Spaces

Haley & Aldrich develops and implements a monitoring and inspection plan for every confined space where Hazardous Atmosphere is a concern. If the confined space has an actual or potential hazardous atmosphere, including oxygen deficiency, it is a PRCS and staff must implement a monitoring and inspection plan, along with other requirements specified in OP1060 – Confined Space Entry. This includes the following steps.

- All conditions that make it unsafe to remove the entrance cover to the confined space will be eliminated before the cover is removed.
- After removal of the cover, the entrance to the confined space will be marked and guarded to prevent accidental fall through of individuals or other foreign objects.
- Before a staff member enters the space, the atmosphere will be tested for oxygen content, flammable gases and vapors, and potential toxic air contaminants, in that order.
- Any entrants will observe this testing process.
- Staff will not enter spaces that contain a hazardous atmosphere, nor remain in the space if a hazardous atmosphere is subsequently detected.
- Continuous forced air ventilation will be utilized according to the requirements specified in 29 CFR 1910.146 (c)(5)(ii)(E).
- The confined space will be tested for hazardous atmosphere continuously to ensure the forced air ventilation is functioning properly.
- Staff members will leave the confined space immediately if hazardous atmosphere is detected.
- An attendant must always be present when staff are in a confined space.

In the event of a change to the PRCS, whether it be reason for entry or presence of hazards, Haley & Aldrich staff members will immediately contact the RSM, who may reclassify the confined space, or stop work until it is safe to continue.

15.2.2 Permit-Required Confined Space Hazards Additional to Atmosphere

All PRCS entries with hazards other than or additional to hazardous atmosphere will also require the development of a PRCS entry permit. Other hazards include, but are not limited to engulfment, entrapment, drowning, and electrical hazards. Each PRCS entry is project-specific and is governed by a permit that must be complete prior to entry. All PRCS programs will adhere to the OSHA requirements provided in 29 CFR 1910.146 (d).

15.3 WHEN HALEY & ALDRICH IS NOT THE CONTROLLING EMPLOYER ON SITE

When Haley & Aldrich is not the controlling employer, and staff members are required to enter a confined space, the following protocol will be followed as detailed in 29 CFR 1910.146 (c)(8).

- Haley & Aldrich staff will be sure to complete the necessary permits required by the controlling employer and adhere to the requirements specified on the permit.
- Ensure that all of the hazards present in the confined space are clearly communicated with the Haley & Aldrich Staff Member.
- Communicate an entry, work, and exit plan with the contractor for the planned work inside the confined space.
- Debrief with the controlling employer following exit from the confined space.

15.4 CONFINED SPACE TRAINING

All Haley & Aldrich staff members will receive the proper training prior to being assigned the work that is specified in 29 CFR 1910.146 (g). All staff member training certificates can be provided upon request.

16. SPILL CONTAINMENT PROGRAM

16.1 POTENTIAL SOURCES

This section describes the potential for hazardous substance spills at this site, and procedures for controlling and containing such spills. Haley & Aldrich will ensure that spill containment planning is conducted, and appropriate control measures are established, consistent with regulatory requirements.

16.1.1 Handling Drums

Only employees who have completed U.S. Department of Transportation (DOT) certification training can handle drums. If you do not maintain the appropriate training, or you training has expired, please complete a training request ticket to H&S to be enrolled in a new DOT training course.

Hazardous substances and contaminated soils, liquids, and other residues shall be handled, transported, labeled, and disposed of in accordance with the following procedures:

- Drums and containers used during the clean-up shall meet the appropriate DOT, OSHA, and EPA regulations for the wastes that they contain.

- When practical, drums and containers shall be inspected, and their integrity shall be assured prior to being moved. Drums or containers that cannot be inspected before being moved because of storage conditions (i.e., buried beneath the earth, stacked behind other drums, stacked several tiers high in a pile, etc.) shall be moved to an accessible location and inspected prior to further handling.
- Unlabeled drums and containers shall be considered to contain hazardous substances and handled accordingly until the contents are positively identified and labeled.
- Site operations shall be organized to minimize the number of drums or containers moved.
- Prior to movement of drums or containers, all staff members exposed to the transfer operation shall be warned of the potential hazards associated with the contents of the drums or containers.
- U.S. Department of Transportation specified salvage drums or containers, and suitable quantities of proper absorbent shall be kept available and used in areas where spills, leaks, or ruptures may occur.
- Where major spills may occur, a spill containment program shall be implemented to contain and isolate the entire volume of the hazardous substance being transferred.
- Drums and containers that cannot be moved without rupture, leakage, or spillage shall be emptied into a sound container using a device classified for the material being transferred.
- A ground-penetrating system or other type of detection system or device shall be used to estimate the location and depth of buried drums or containers.
- Soil or covering material shall be removed with caution to prevent drum or container rupture.
- Fire extinguishing equipment shall be on hand and ready for use to control incipient fires.

16.1.2 Opening Drums and Containers

The following procedures shall be followed in areas where drums or containers are being opened:

- Where an airline respirator system is used, connections to the source of air supply shall be protected from contamination and the entire system shall be protected from physical damage.
- Staff members not actually involved in opening drums or containers shall be kept a safe distance from the drums or containers being opened.
- If staff members must work near or adjacent to drums or containers being opened, a suitable shield that does not interfere with the work operation shall be placed between the staff member and the drums or containers being opened to protect the staff member in case of accidental explosion.
- Controls for drum or container opening equipment, monitoring equipment, and fire suppression equipment shall be located behind the explosion-resistant barrier.
- When there is a reasonable possibility of flammable atmospheres being present, material handling equipment and hand tools shall be of the type to prevent sources of ignition.
- Drums and containers shall be opened in such a manner that excess interior pressure will be safely relieved. If pressure cannot be relieved from a remote location, appropriate shielding shall be placed between the staff member and the drums or containers to reduce the risk of staff member injury.
- Staff members shall not stand upon or work from drums or containers.

16.1.2.1 Material Handling Equipment

Material handling equipment used to transfer drums and containers shall be selected, positioned, and operated to minimize sources of ignition related to the equipment from igniting vapors released from ruptured drums or containers. Any material handling equipment necessary must be selected and identified in project documents, communicated with staff members performing the work, and readily available for work.

16.1.3 Radioactive Wastes

- Drums and containers containing radioactive wastes shall not be handled until their hazard to staff members is properly assessed.
- If handling radioactive wastes is necessary, staff members must coordinate with Health & Safety to ensure that proper controls are in place to mitigate as much exposure as possible.

16.1.4 Shock-Sensitive Wastes

- Drums and containers containing shock sensitive wastes shall not be handled until their hazard to staff members is properly assessed.
- If handling shock sensitive wastes is necessary, staff members must coordinate with Health & Safety to ensure that proper controls are in place to mitigate as much exposure as possible.

16.1.5 Laboratory Waste Packs

The following precautions shall be taken, as a minimum, when handling laboratory waste packs (lab packs):

- Lab packs shall be opened only when necessary and then only by an individual knowledgeable in the inspection, classification, and segregation of the containers within the pack according to the hazards of the wastes.
- If crystalline material is noted on any container, the contents shall be handled as a shock-sensitive waste until the contents are identified.

16.1.6 Sampling of Drum and Container Contents

Sampling of containers and drums shall be done in accordance with a sampling procedure which is part of the project-specific JHA.

16.1.7 Shipping and Contents

Drums and containers shall be identified and classified prior to packaging for shipment. Identifying, classifying, and signing manifests for drums and containers requires training and an agency agreement to be in place. Before assigning staff members to perform this work, PMs must ensure staff have the proper training. Drum or container staging areas shall be kept to the minimum number necessary to identify and classify materials safely and prepare them for transport. Staging areas shall be provided with adequate access and egress routes. Bulking of hazardous wastes shall be permitted only after a thorough characterization of the materials has been completed.

16.1.8 Tank and Vault Procedures

Appropriate tank or vault entry procedures shall be identified and followed as outlined in project-specific JHAs, and must be developed in compliance with confined space entry requirements outlined in Section 15 Confined Space Entry.

16.2 SITE EVALUATION

All spills on the site are considered hazardous before they are evaluated. If an evaluation of the site and hazards present has been conducted, and the project team has determined that there is little if any potential for a hazardous substance spill of a sufficient quantity to require containment planning, equipment, and procedures, then no spill containment procedures will be required at the site. Spill containment kits should still be made available on site in the event an unforeseen incident occurs or there is a release from a piece of operation equipment. Staff members must always be trained on how to respond if there has been a potential spill, including any Controlling Employer requirements.

If following an evaluation it is determined there is the potential for a hazardous spill on site, then the spill containment procedures must be followed. The general outline below is to be followed and used to develop site-specific spill containment through JHAs. JHAs for spill containment should include but will not be limited to:

- Potential hazardous substances, their locations, and available controls
- Available spill containment equipment
- Initial notification and response
- Spill evaluation and response
- Post spill evaluation.

16.3 POTENTIAL SPILLS AND AVAILABLE CONTROLS

If there is potential for a hazardous spill on site, all site personnel are trained in the contents of their project-specific spill containment program and their roles and responsibilities during spill response operations. This information is to be outlined in the project-specific JHA and communicated with all staff at initial assignment and whenever there are changes in the process, hazards, or responsibilities. Project teams must identify the location and type of potential hazardous substance spills at this site. The project-specific JHA must also describe the activities or situations in which an accidental spill could occur and whether an emergency response is likely to be needed.

Where spills, leaks, or ruptures can occur, this site must have suitable quantities of proper absorbent and DOT-specified salvage drums and containers. In addition, all areas subject to potential spills are diked or a means to adequately dike these areas in the event of a spill is available so that the entire volume of the spilled hazardous substance can be contained and isolated. The type and location of spill containment equipment must also be outlined in the project-specific JHA.

16.4 INITIAL SPILL NOTIFICATION AND RESPONSE

Any staff member who discovers a hazardous substance spill must immediately notify the designated site contact, their PM, and RSM. The staff member, to their ability, shall report the following information:

- Hazardous substance involved.
- Location of the spill.
- Estimated quantity of substance spilled, the direction/flow of the spill material, related fire/explosion incidents, and any associated injuries.

16.5 SPILL EVALUATION AND RESPONSE

A member of the Health & Safety Team is responsible for working with project teams to evaluate hazardous substance spills and determine the appropriate response. When the hazardous substance spill evaluation is being made, the spill area is isolated to the greatest extent possible.

When an incidental release occurs, cleanup personnel receive instructions in a pre-cleanup meeting as to spill conditions, PPE, response activities, decontamination, and waste handling. The procedures of the Emergency Response section of this HASP are immediately implemented when the spill is determined to require emergency precautions and action. If necessary to protect those outside the cleanup area, notification of the appropriate authorities is made.

The following are general measures that must be considered for response/cleanup personnel to take when responding to a spill:

- To minimize the potential for a hazardous spill, hazardous substances, control/absorbent media, drums and containers, and other contaminated materials must be properly stored and labeled.
- When a spill occurs, only those persons involved in overseeing or performing spill containment operations will be allowed within the designated hazard areas. If necessary, the area will be roped or otherwise blocked off. Unauthorized personnel are kept clear of the spill area.
- Appropriate PPE is donned before entering the spill area.
- Appropriate spill control measures are applied during spill response.
- Whenever possible without endangerment to personnel, the spill is stopped at the source or as close to the source as possible.
- Ignition points are removed if fire or explosion hazards exist.
- Surrounding reactive materials are removed.
- Drains or drainage in the spill area is blocked or surrounded by berms to exclude the spilled waste and any materials applied to it.
- Provisions are made to contain and recover a neutralizing solution, if used.
- Small spills or leaks from a drum, tank, or pipe will result in the establishment of an evacuation zone to allow cleanup and to prevent staff member exposure. For small spills, sorbent materials are placed directly on the spill to prevent further spreading and aid in recovery.

- The spill area is sprayed with appropriate foam where the possibility of volatile emissions exists.
- If the spill results in the formation of a toxic vapor cloud from vaporization, reaction with surrounding materials, or the outbreak of fire, further evacuation may be required.
- To dispose of spill waste, all contaminated sorbents, liquid waste, or other spill cleanup will be placed in small quantities in approved drums for proper storage or disposal as hazardous waste.
- For more information on specific spill containment plans, please refer to project documents.

16.6 POST SPILL EVALUATION

At the conclusion of cleanup operations, documentation of spill response report must be prepared through our appropriate forms on our Forms, Audits, and Checklists page on the Health & Safety Intranet. The report includes, at a minimum, the following information:

- Date of spill incident.
- The hazardous substance(s) released.
- The quantity released.
- The location of the release.
- The effect of the release.
- Cause of incident.
- Spill response actions.
- Who responded.
- Spill equipment used.
- How the spill material was discarded or contained.
- Any outside agencies involved, including their incident reports.
- Lessons learned or suggested improvements.

The spill area is inspected for residual contaminants to determine whether safety and environmental requirements are met. The use of surface and air sampling is utilized in this determination as necessary. The root cause of the spill is examined, and corrective steps must be taken to ensure the engineering and control measures in place have performed as required. If alternative precautions or measures are needed, they are made available and implemented.

All durable equipment placed into use during cleanup activities must be decontaminated, as specified in this section, for future utilization. All spill response equipment and supplies are restocked as required.

APPENDIX A – CHEMICAL HAZARDS

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Metals			
Antimony	50 mg/m^3	0.5 mg/m^3 0.5 mg/m^3 0.5 mg/m^3	***Skin:*** Irritation ***Eye:*** Irritation ***Respiratory Tract:*** Nose and throat irritation, cough, loss of smell ***Gastrointestinal:*** Nausea, vomiting, diarrhea, stomach cramps
Arsenic	5 mg/m^3	0.01 mg/m^3 0.01 mg/m^3 0.002 mg/m^3 Known human carcinogen.	***Skin:*** Irritation, burning, itching and rash. Contact dermatitis is possible. ***Eye:*** Red, watery eyes and irritation. Conjunctivitis may occur in some people. ***Lung:*** Nose and throat irritation, and possible nasal perforation. ***CNS:*** Can cause nausea, poor appetite, vomiting, and muscle cramps, cognitive impairments and peripheral neuropathy.
Barium (and soluble compounds, as Ba, except barium sulfate)	50 mg/m^3 (as Ba)	0.5 mg/m^3 (as Ba) 0.5 mg/m^3 (as Ba) 0.5 mg/m^3 (as Ba).	***Skin:*** Irritation. Skin burns may be possible. ***Eye:*** Irritation, itching, and burning on contact ***Lung:*** Upper respiratory irritation. **Other:** Gastroenteritis; muscle spasms; slow pulse; extrasystoles; hypokalemia.
Beryllium	4.0 mg/m^3 (as Be)	0.002 mg/m^3 (as Be) 0.002 mg/m^3 (as Be) 0.0002mg/m^3 proposed TLV 0.0005 mg/m^3 (as Be, ceiling) Probable human carcinogen.	***Skin:*** Severe irritation. Skin ulcers may be possible. ***Eye:*** Severe irritation, itching, and burning on contact. Sometimes an allergic eye problem develops, breaking out again with future exposures. ***Lung:*** Over-exposure can severely irritate the airways and lungs, causing nasal discharge, tightness in the chest, cough, shortness of breath, and/or fever. Future exposures can cause further attacks. Death can occur in severe cases. High level acute exposures can lead to pneumonitis.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Cadmium	9.0 mg/m^3	0.005 mg/m^3 (Cd dust) 0.01 mg/m^3 (Cd dust) Reduce to lowest possible level. Known human carcinogen.	***Skin:*** Possible irritation may occur. ***Eye:*** Irritation and possible damage on contact. ***Lung:*** High exposures can cause rapid and severe lung damage, with shortness of breath, chest pain, cough, and a possible buildup of fluids in the lungs (pulmonary edema), which is a medical emergency. In serious cases, death or permanent lung damage can occur. Illness can be delayed for 48 hours, allowing over-exposures to occur without warning signs. ***Other:*** High exposure to cadmium may cause nausea, cramps, salivation, vomiting and diarrhea.
Chromium	250 mg/m^3 (as Cr)	1.0 mg/m^3 (as Cr) 0.5 mg/m^3 (as CrIII) 0.5 mg/m^3 (as CrIII) Hexavalent chromium is a known human carcinogen via inhalation.	***Skin:*** Irritation, rash and possible dermatitis. ***Eye:*** Chromium particles can irritate the eyes. ***Lung:*** Inhalation of chromium may cause a condition known as “metal fume fever” (flu-like symptoms).
Cobalt	20 mg/m^3 (as Co)	0.10 mg/m^3 0.02 mg/m^3 0.05 mg/m^3	***Skin***: Dermatitis. ***Lung***: Cough, shortness of breath, wheezing, respiratory hypersensitivity, asthma
Copper (dusts and mists, as Cu, except copper fumes)	100 mg/m^3 (as Cu)	1.0 mg/m^3 (as Cu) 1.0 mg/m^3 (as Cu) 1.0 mg/m^3 (as Cu)	***Skin:*** Irritation, rash and possible dermatitis. ***Eye:*** Red, watery eyes and irritation ***Lung:*** Irritation of nose, pharynx; possible perforation of the nasal septum
Lead	100 mg/m^3 (as Pb)	0.05 mg/m^3 (as Pb) 0.05 mg/m^3 (as Pb) 0.05 mg/m^3 (as Pb)	***Skin:*** Irritation/scratching on contact with particles. ***Eye:*** Irritation/scratching on contact with particles. ***Lung:*** Possible irritation of the nose, throat, and lungs following exposure to fibers and dusts.
Manganese	500 mg/m^3	5 mg/m^3 0.2 mg/m^3 1 mg/m^3	***Lung:*** Cough, dry throat, breathing difficulty, chest tightness, flu-like fever. ***Gastrointestinal (ingestion route):*** Abdominal pain, nausea, vomiting.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Mercury	10 mg/m^3 (as Hg)	0.1 mg/m^3 (skin) 0.025 mg/m^3 (skin) 0.1 mg/m^3 (skin)	***Skin:*** Irritation, rash, dermatitis, and possible redness and the appearance of small blisters. Dermal absorption will cause toxic systemic effects. ***Eye:*** Irritation, inflammation, watering, swelling, conjunctivitis. ***Lung:*** Inhalation of mercury vapors may cause irritation of the mucous membranes in the respiratory tract causing cough, phlegm, and lung tissue irritation leading to bronchitis, pneumonitis, and even delayed pulmonary edema.
Nickel	10 mg/ m^3	1.0 mg/m^3 1.5 mg/m^3 0.015 mg/m^3	***Skin:*** Irritation/scratching on contact with dusts. May cause "nickel itch" or nickel dermatitis. ***Eye:*** Irritation and scratching with possible progression to conjunctivitis. ***Lung:*** Irritation of the nose, throat and lungs on exposure to powders and dusts.
Selenium	1 mg/m^3	0.2 mg/m^3 0.2 mg/m^3 0.2 mg/m^3	***Skin:*** Irritation, burns, dermatitis ***Eye:*** Irritation and burns ***Respiratory Tract:*** Nose and throat irritation, shortness of breath, bronchitis ***Other:*** Metallic taste, garlic breath, GI disturbance
Silica	25-50 mg/m^3 (suspected human carcinogen)	<u>10 mg/m^3</u> % SiO_2 +2 (respirable dust) 0.05 mg/m^3 (respirable dust) 0.05 mg/m^3	***Eye:*** Irritation. ***Lung:*** Cough, dyspnea (breathing difficulty), wheezing.
Silicon	ND	15 mg/m^3 (total dust) 10 mg/m^3 (total dust) 10 mg/m^3 (total dust)	***Skin:*** Irritation and possible rash upon contact with skin (contact dermatitis). ***Eye:*** Severe irritation and possible inflammation. ***Lung:*** Irritation of the nose, throat, and lungs following exposure. Can cause coughing, dyspnea, nausea and headache.
Silver	10 mg/m^3 (as Ag)	0.01 mg/m^3 0.01 mg/m^3 0.01 mg/m^3	***Skin:*** Irritation and ulceration. ***Eye:*** Whites of eyes turn blue/gray eyes. ***Respiratory:*** Nose and throat irritation.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Thallium (soluble compounds as Tl)	15 mg/m^3 (as Tl)	0.01 mg/m^3 (as Tl) 0.01 mg/m^3 (as Tl) 0.01 mg/m^3 (as Tl)	***Eye:*** Irritation ***GI Tract***: Nausea, diarrhea, vomiting, metallic taste in mouth ***Respiratory***: Pulmonary edema ***CNS****:* Perineuritis, tremor, convulsions, psychosis. ***Other***: Ptosis, strabismus, chest tightness, chest pain, chorea, potential liver and kidney damage; alopecia
Zinc (as zinc oxide dust)	500 mg/m^3 (as ZnO dust)	15 mg/m^3 (total dust) 5 mg/m^3 (respirable dust) 5 mg/m^3 (TWA) 15 mg/m^3 (Ceiling) 2 mg/m^3 (TWA) 10 mg/m^3 (STEL)	***Respiratory***: Dry throat, cough, chest tightness, decreased pulmonary function. ***GI Tract*****:** Nausea, metallic taste in mouth, vomiting ***CNS****:* Blurred vision, malaise. ***Other***: Fever, chills, muscle aches, headaches, lower back pain, difficulty breathing,
BTEX			
Benzene	500 ppm	1.0 ppm 0.5 ppm ppm Known human carcinogen.	***Skin:*** Irritation with potential for redness, blistering and burning. ***Eye:*** Severe irritation; pain and permanent damage possible. ***Lung:*** Nose, throat and respiratory tract irritation causing difficulty in breathing, possible pulmonary edema (fluid in lungs) on high exposure that can be fatal. ***CNS:*** Confusion, dizziness, tightening of the leg muscles, cranial pressure (headaches), possible coma.
Chlorobenzene	1,000 ppm	75 ppm 10 ppm none	***Skin:*** Irritation with potential for redness and burning. ***Eye:*** Irritation and redness. ***Lung:*** Nose, throat and respiratory tract irritation ***CNS:*** CNS depressant, drowsiness

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Ethylbenzene	800 ppm	100 ppm 100 ppm 100 ppm	***Skin:*** Irritation with potential for redness, blistering, and serious burning. Chemical will pass through intact skin to cause toxic systemic effects, including narcosis. ***Eye:*** Severe irritation, burning, pain, and permanent damage possible. ***Lung:*** Nose, throat and respiratory tract irritation causing difficulty in breathing, possible pulmonary edema (fluid in lungs) on high exposure. ***CNS:*** Narcosis, headaches, light-headedness, dizziness, vertigo, coma, and death due to respiratory paralysis.
Toluene	500 ppm	200 ppm 50 ppm (skin) 100 ppm	***Skin:*** Severe irritation and possible burns. Will pass through unbroken skin to cause toxic systemic effects (weakness, nausea, collapse, and death). ***Eye:*** Severe irritation with damage and possible loss of vision likely. ***Lung:*** Severe irritation to the respiratory tract and associated mucosa. Can cause weakness, nausea, confusion, collapse, coma, and death (respiratory failure). ***CNS:*** A CNS depressant. Weakness, confusion, mood changes and other personality changes, nausea, collapse. ***Other:*** Ingestion will cause irritation to the gastrointestinal system with nausea, vomiting, convulsions, dyspnea, jaundice, tremor, confusion, and nervousness. Toluene is toxic to the liver and kidneys.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Xylene (mixed monomers)	900 ppm	100 ppm 100 ppm 100 ppm	***Skin:*** Irritation with potential for redness, blistering and serious burning. Chemical will pass through intact skin to cause systemic effects, including narcosis. ***Eye:*** Severe irritation, burning, pain, and permanent damage possible. ***Lung:*** Nose, throat, and respiratory tract irritation causing difficulty in breathing, possible pulmonary edema (fluid in lungs) on high exposure. ***CNS:*** Narcosis, headaches, light-headedness, dizziness, vertigo, drowsiness, loss of consciousness, coma, and death due to respiratory paralysis.
Diesel Fuel	ND	-- 100 mg/m^3 --	***Skin:*** Irritation, dermatitis ***Eye:*** Irritation ***Lung:*** Cough, difficulty breathing, nausea, chest tightness ***CNS:*** Lightheadedness, loss of consciousness. ***Other:*** Danger of cutaneous absorption, absorption through the skin may be significant contribution to overall exposure.
Gasoline	--	-- 300 ppm Reduce to lowest level	***Skin:*** Irritation with potential for redness, blistering, burning, and absorption. ***Eye:*** Severe irritation, conjunctivitis, redness, possible pain, and visual disturbance. ***Lung:*** Nose, throat, and respiratory tract irritation causing difficulty in breathing, possible pulmonary edema on high exposure (a medical emergency) that can be fatal. ***CNS:*** Inebriation, dizziness, stupor, hallucinations, slurred speech, mental confusion, loss of consciousness and possible convulsions. ***Other:*** Acute exposure can also lead to acute hemorrhage of the pancreas, fatty degeneration of the liver and kidneys, and congestion of the spleen.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Kerosene	ND	Not determined 200 mg/m^3 100 mg/m^3	***Skin:*** Irritation, dermatitis ***Eye:*** Irritation ***Lung:*** Burning sensation in chest, irritation of nose and throat ***CNS:*** Headache, nausea, lassitude, restlessness, incoherence, confusion, drowsiness.
Volatiles			
1,1-Dichloroethane	3,000 ppm	100 ppm 100 ppm 100 ppm	***Skin:*** Irritation. ***CNS:*** Depression. **Other**: Liver, kidney and lung damage.
1,2-Dichloroethane (Ethylene dichloride)	50 ppm (Ca)	50 ppm 10 ppm 1 ppm	***Skin:*** Irritation with potential for redness, pain, burns, and tissue damage. ***Eye:*** Irritation and smarting of the conjunctiva. Can cause clouding of the cornea (corneal opacity). ***Lung:*** Irritation of the mucous membranes of the nose, throat, and respiratory tract causing possible ulceration, coughing, chest pains, difficulty breathing, and pulmonary edema. ***CNS:*** Depression with headaches, light-headedness, dizziness, nausea, vomiting, diarrhea, narcotic and anesthetic effects.
1,1-Dichloroethene (vinylidene chloride)	ND	-- 5 ppm Not determined	***Skin:*** Irritation ***Eye:*** Irritation ***Respiratory system:*** Irritated throat ***CNS:*** Depressant, with dizziness, headache, nausea.
1,2-Dichloroethene	1,000 ppm	200 ppm 200 ppm 200 ppm	***Eye:*** Irritation ***Lung:*** Irritation ***CNS:*** Depressant
1,4 Dioxane	500 ppm (Ca)	100 ppm 20 ppm 1 ppm	***Skin:*** Irritation ***Eyes:*** Irritation ***Lung:*** Nose and throat irritation ***GI:*** Nausea, vomiting, liver failure ***CNS:*** Drowsiness, headache
2-butanone (MEK)	3,000 ppm	200 ppm 200 ppm 200 ppm	***Skin:*** Irritation, dermatitis ***Eye:*** Irritation ***Resp. System:*** Irritation of upper respiratory tract ***CNS:*** Headache, dizziness, vomiting

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Acetone	2,500 ppm	1,000 ppm 500 ppm 250 ppm	***Skin:*** Dermatitis ***Eye:*** Irritation ***Respiratory tract:*** Upper respiratory tract irritation ***CNS:*** Headache, dizziness CNS depression
Carbon Disulfide	500 ppm	20 ppm 1 ppm (skin) 1 ppm (skin)	***Skin*****:** Skin burns, dermatitis ***Eye*****:** Ocular changes, burns ***CNS*****:** Dizziness, headache, lassitude, anxiety, Parkinson-like syndrome ***Other*****:** Gastritis ***Target Organs:*** central nervous system, peripheral nervous system, cardiovascular system, kidneys, liver
Carbon Tetrachloride	200 ppm	10 ppm 5 ppm 2 ppm (60 minute STEL)	***Skin:*** Irritation with potential for redness, drying and cracking. ***Eye:*** Irritation with tearing and burning. ***Lung:*** Irritation of the mucous membranes of the nose, throat, and respiratory tract. ***CNS:*** Depression of the CNS with headache, dizziness, confusion. ***GI***: Gastric distress, nausea, vomiting, loss of appetite.
Chloroethane (ethyl chloride)	3,800 ppm	1,000 ppm 100 ppm Handle with caution	***CNS:*** Loss of coordination, inebriation, **GI:** Abdominal cramps **Other:** Cardiac arrhythmia, cardiac arrest, liver and kidney damage
Chloroform	500 ppm	50 ppm 10 ppm Reduce to lowest level	**Skin:** Irritation ***Eyes:*** Irritation ***Central Nervous System:*** Dizziness, nausea, confusion, headache, weakness, exhaustion and anesthetic effects.
Chloromethane (methyl chloride)	2,000 ppm	100 ppm (C: 200 ppm) 50 ppm As low as possible –Ca	***Skin:*** Frostbite ***CNS:*** Dizziness, stagger, slurred speech, convulsions, coma **GI:** Nausea, vomiting
Cyclohexane	2,000 ppm	300 ppm 100 ppm 300 ppm	***Skin:*** Redness, dryness, irritation, dermatitis ***Eye:*** Redness, irritation ***Lung:*** Nose and throat irritation, cough, ***CNS:*** Headache, dizziness, weakness, drowsiness, narcosis, coma ***Gastrointestinal:*** Abdominal pain, nausea, vomiting

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Methyl Isobutyl Ketone (MIBK or Hexone)	500 ppm	100 ppm 50 ppm 50 ppm	***Skin:*** Irritation and dermatitis with possible localized rash and redness. Absorption is likely. ***Eye:*** Irritation with pain and inflammation that can lead to corneal damage and possible visual disturbances. ***Lung:*** Irritation causing coughing and/or shortness of breath. Higher exposures can cause dizziness, headache and, possibly, coma and death. ***CNS:*** A mild to moderate narcotic compound producing neurotoxicity as it is partially metabolized to methanol.
Methyl Tertiary Butyl Ether (MTBE)	ND	TLV- 50 ppm	***Lung:*** Nose and throat irritation. ***CNS:*** Headache, nausea, dizziness, mental confusion.
Methylcyclohexane	1,200 ppm	500 ppm 400 ppm 400 ppm	***Skin:*** Irritation, drying and cracking of exposed areas upon prolonged contact ***Eye:*** Irritation ***Lung:*** Upper respiratory tract irritation ***CNS:*** Dizziness, lightheadedness, drowsiness
Methylene chloride (Dichloromethane)	2,300 ppm	25 ppm 50 ppm Reduce to lowest level	***Skin:*** Irritation with potential for redness, pain, burns and tissue damage. ***Eye:*** Irritation and smarting of the conjunctiva. Can cause tearing, redness and pain. ***Lung:*** Irritation of the mucous membranes of the nose, throat, and respiratory tract causing possible ulceration, coughing, chest pains, difficulty breathing and pulmonary edema. ***CNS:*** Depression with headaches, light-headedness, dizziness, nausea, vomiting, diarrhea, narcotic and anesthetic effects.
Pentachlorophenol	2.5 mg/m^3	0.5 mg/m^3(skin) 0.5 mg/m^3 0.5 mg/m^3	***Skin:*** Dermatitis ***Eye:*** Irritation ***Respiratory Tract:*** Irritation of nose and throat, coughing, sneezing ***CNS:*** Headache, dizziness, lassitude ***GI:*** Nausea, vomiting ***Cardiac:*** chest pain

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Stoddard Solvent (Mineral Spirits)	20,000 mg/m^3	500 mg/m^3 100 ppm 350 mg/m^3	***Skin:*** Irritation, dermatitis ***Eye*:** Irritation ***Lung:*** Nose and throat irritation. May cause chemical pneumonia if liquid is aspirated. ***CNS:*** Depressive effects. Dizziness. ***Other:*** Kidney damage
Tert-Butyl Alcohol (TBA, 2-Methyl-2-propanol)	1,600 ppm	100 ppm --- 100 ppm	***Skin:*** Irritation ***Eye:*** Irritation ***Respiratory:*** Nose and throat irritation ***CNS:*** Drowsiness, narcosis
Tetrachloroethylene (Perchloroethylene)	150 ppm	100 ppm 25 ppm Lowest possible level	***Skin:*** Moderate irritation with redness and possibly painful inflammation. It will pass through unbroken skin easily to cause toxic systemic effects. ***Eye:*** Severe irritation and burning but rarely causes permanent damage. ***Lung:*** Nose and throat irritation. Produces intoxicating effects and may cause lung effects as well. ***CNS:*** Unconfirmed effects by all routes of exposure. There may be numbness around the lips and mouth, dizziness, loss of coordination, headache, somnolence, slurred speech, flushed face, irregular heartbeat, and other unspecified effects on nervous system functions.
1,1,2,2-Tetrachloroethane	100 ppm	5 ppm 1 ppm 1 ppm	***Skin:*** Moderate irritation with redness and possibly painful inflammation. It will pass through unbroken skin easily to cause toxic systemic effects. Dermatitis is also possible. ***Eye:*** Severe irritation and burning, but rarely causes permanent damage. ***Lung:*** Nose and throat irritation. Produces intoxicating effects and may cause lung effects as well. ***CNS:*** Adverse effects (depression) by all routes of exposure. There may be numbness around the lips and mouth, dizziness, loss of coordination, headache, somnolence, slurred speech, flushed face, irregular heartbeat, and other unspecified effects on nervous system functions.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
1,1,1-Trichloroethane (Methyl chloroform)	700 ppm	350 ppm 350 ppm 350 ppm	***Skin:*** Irritation with potential for redness, pain, dryness, scaling, cracking, and infection. Dermatitis may occur in some individuals. ***Eye:*** Severe irritation, burning, pain and smarting of the conjunctiva. Permanent damage to visual acuity is possible. ***Lung:*** Nose, throat, and respiratory tract irritation. High concentrations can cause severe irritation. ***CNS:*** Headaches, light-headedness, dizziness, hallucinations, loss of coordination, aggressive behavior, motor changes. Death can occur due to cardiac arrhythmia and possible cardiac arrest.
1,1,2-Trichloroethane	100 ppm	10 ppm 10 ppm 10 ppm	***Skin:*** Irritation ***Eye:*** Irritation ***Respiratory:*** Nose irritation ***CNS:*** Depressant
Trichloroethylene	1,000 ppm	100 ppm 10 ppm 25 ppm	***Skin:*** Moderate irritation with redness and possibly painful inflammation with blisters and burns. It will pass through unbroken skin easily to cause toxic systemic effects. ***Eye:*** Severe irritation and burning with a possibility for injury to the corneal epithelium. ***Lung:*** Nose and throat irritation. Produces intoxicating effects and may cause lung effects such as pulmonary edema (may be fatal). ***CNS:*** Depressive effects by all routes of exposure. There may be numbness around the lips and mouth, dizziness, loss of coordination, headache, somnolence, slurred speech, blurred vision, flushed face, irregular heartbeat, and other effects on nervous system functions. Death can result from cardiac arrest.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Vinyl Chloride	--	1 ppm 1 ppm Reduce to lowest level Known human carcinogen.	***Skin:*** Irritation with potential for redness, pain, inflammation and tissue damage (frostbite). ***Eye:*** Severe irritation, burning, pain, and permanent damage possible. ***Lung:*** Nose, throat, and respiratory tract irritation. Symptoms can include dyspnea, asthma, and pneumoconiosis. ***CNS:*** Narcosis, headaches, light-headedness, dizziness, drowsiness, nausea, vomiting, confusion, decreased mental status, possible loss of consciousness, and death (respiratory arrest).
Pesticides			
2,4 Dichlorophenoxy-cetic acid	100 mg/m^3	10 mg/m^3 10 mg/m^3 10 mg/m^3	***Skin***: Dermatitis ***Inhalation***: Irritation of nose and throat; irritation of lungs causing coughing and/or shortness of breath; fluid buildup in lungs; headache; fatigue; muscle weakness and twitching; poor coordination in arms and legs. ***Ingestion***: Nausea, vomiting, sweating, fever, diarrhea, stomach pain, and loss of appetite and weight.
Carbaryl (Sevin)	100 mg/m^3	5 mg/m^3 5 mg/m^3 5 mg/m^3	***Skin***: May cause severe irritation, rash or a burning feeling. Will pass through unbroken skin and enter the bloodstream. ***Eyes***: May cause severe irritation. ***Inhalation***: Can cause blurred vision, sweating, nausea, vomiting and abdominal pain. Higher exposures can cause pulmonary edema and death. ***Ingestion***: Symptoms include nausea, vomiting, diarrhea, abdominal cramps, miosis, lachrymation, and excessive salivation.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Chlordane	100 mg/m^3	0.5 mg/m^3 (skin) 0.5 mg/m^3 (skin) 0.5 mg/m^3 (skin)	***Skin:*** Liquid can pass through unbroken skin. Contact may cause irritation and rash. Symptoms of exposure include headache, nausea, abdominal pain, and vomiting. ***Eye:*** Severe irritation and possible damage on contact. ***Lung:*** Irritation of the nose and throat. Breathing vapors can cause cough, convulsions, unconsciousness, and death. ***CNS:*** A central nervous system stimulant. Symptoms include ataxia, excitement, tremors, convulsions, blurred vision, confusion, delirium, and irritability.
Copper sulfate	100 mg/m^3 as Cu	Dust/mist – 1 mg (Cu)/m^3 1 mg (Cu)/m^3 1 mg (Cu)/m^3	**Skin**: Contact may cause smarting and first degree burns, second degree burns on prolonged contact. Can pass through skin and cause toxic systemic effects. **Inhalation**: Irritation of the upper respiratory system, nose and throat. May cause nosebleeds and “metal fume fever” (flu-like illness with chills, aches and cough.) **Ingestion**: Will cause intense pain and local corrosion with hemorrhages, prostration, anuria, hematuria, anemia, coma, and death.
DDT (Dichlorodiphenyl-trichloroethane)	500 mg/m^3	1.0 mg/m^3 (skin) 1.0 mg/m^3 0.5 mg/m^3 (skin)	***Skin:*** Dermatitis associated with DDT is unusual. Most effects from skin contact are internal due to adsorption of liquid solutions containing DDT. Symptoms include vomiting, nausea, malaise, and muscular twitching at high doses (above the PEL). ***Eye:*** Possible irritation. ***Lung:*** Irritation causing coughing and/or shortness of breath. Higher exposures can lead to respiratory problems. ***CNS:*** Dizziness, headaches, nausea, vomiting, muscle jerks, severe seizures, and death.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Dieldrin	50 mg/m^3	0.25 mg/m^3 (skin) 0.25 mg/m^3 (skin) 0.25 mg/m^3 (skin)	***Skin:*** Liquid can pass through unbroken skin. Contact may cause irritation and rash. Symptoms of exposure include headache, nausea, abdominal pain, vomiting and CNS stimulation. ***Eye:*** Mild to severe irritation. Absorption through direct eye contact is possible. ***Lung:*** Irritation of the nose and throat. Breathing vapors can cause cough, convulsions, and adverse effects on the liver. ***CNS:*** A central nervous system stimulant. Symptoms include: irritability, blurred vision, tremors, convulsions, and metal confusion.
Endrin	2 mg/m^3	0.1 mg/m^3 (skin) 0.1 mg/m^3 (skin) 0.1 mg/m^3 (skin)	***Skin:*** Liquid can pass through unbroken skin. Contact may cause irritation and rash. Symptoms of exposure include headache, nausea, abdominal pain, vomiting, and CNS stimulation. ***Eye:*** Severe irritation. ***Lung:*** Irritation of the nose and throat. Breathing vapors can cause cough, convulsions, and adverse effects on the liver. ***CNS:*** A powerful central nervous system stimulant. Symptoms of exposure may include agitation, aggressiveness, excitement, tremors, convulsions, blurred vision, mental confusion, delirium, and irritability.
Heptachlor	35 mg/m^3	0.5 mg/m^3 (skin) 0.05 mg/m^3 (skin) 0.5 mg/m^3 (skin)	***Skin:*** Liquid can pass through unbroken skin. Contact may cause irritation and rash. Symptoms of exposure of exposure include headache, nausea, abdominal pain, and vomiting. ***Eye:*** Severe irritation and possible damage on contact. ***Lung:*** Irritation of the nose and throat. Breathing vapors can cause cough, convulsions, unconsciousness, and death. ***CNS:*** A central nervous system stimulant. Symptoms include ataxia, excitement, tremors, convulsions, confusion, delirium and irritability.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Lindane	50 mg/m^3	0.5 mg/m^3 (skin) 0.5 mg/m^3 (skin) 0.5 mg/m^3 (skin)	***Skin:*** Liquid can pass through unbroken skin. Contact may cause irritation and rash. Symptoms of exposure include headache, nausea, abdominal pain, vomiting and CNS stimulation. ***Eye:*** Mild to severe irritation. Absorption through direct eye contact is possible. ***Lung:*** Irritation of the nose and throat. Breathing dusts or solution vapors can cause cough, convulsions, and adverse effects on the liver. ***CNS:*** A central nervous system stimulant. Symptoms include irritability, blurred vision, tremors, convulsions, and mental confusion.
Semivolatiles			
1,2-Dichlorobenzene	200 ppm	50 (Ceiling) 25 ppm 50 (Ceiling)	***Eye:*** Irritation ***Respiratory:*** URT irritation ***Target organs:*** Liver, kidneys
1,4-Dichlorobenzene	150 ppm Carcinogen	75 ppm 10 ppm Carcinogen	***Eye***: Irritation, periorbital swelling ***Inhalation/Ingestion:*** Rhinitis, headache, anorexia, nausea ***Target organs***: Kidneys, liver, respiratory system
3,3'-Dichlorobenzidine	Ca (ND) *OSHA Regulated Carcinogen	NE* ALAP** NE ** As low as possible	**Skin:** Skin sensitization, dermatitis, burns ***Eye:*** Irritation Inhalation/Ingestion: Headache, dizziness, frequent urination, blood in urine, GI upset, upper respiratory infection **Target organs:** Bladder, liver, lung, skin, GI Tract
1,2,4-Trichlorobenzene	ND	none 5 ppm (ceiling) 5 ppm (ceiling)	***Skin:*** Irritation with burning, itching, redness. ***Eye:*** Irritation with burning, itching and watering. ***Respiratory:*** Irritation of mucosa of respiratory tract. ***Other:*** Potential Teratogen

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Aniline	100 ppm (Ca)	5 ppm (skin) 2 ppm (skin) Lowest possible level	***Aniline is rapidly absorbed through all routes and induces methemoglobinemia. Symptoms include:*** *Cyanosis, headache, dizziness, weakness, lethargy, loss of coordination, shortness of breath.* ***Skin:*** Aniline causes skin irritation and mild skin sensitization. The skin is a major route of exposure. **Eye**: Severe irritation ***CNS****:* Severe headache, tremor, confusion. CNS depression may be a result of methemoglobinemia. ***Systemic****:* Chronic exposure may cause injury to kidney, bladder, liver and spleen.
Bis(2-ethylhexyl) phthalate	5000 mg/m^3	PEL: 5 mg/m^3 TLV: 5 mg/m^3	***Skin***: Irritation ***Eye****:* Irritation ***Other****:* Nausea, diarrhea ***Suspected human carcinogen***
Hexachlorobenzene	NE	NE 0.002 mg/m^3 NE	***Skin:*** UV sensitivity and skin damage, hyperpigmentation ***Inhalation/ingestion:*** CNS impairment, skin effects noted above
Naphthalene	250 ppm	10 ppm 10 ppm (skin) 10 ppm	***Skin:*** Irritation with burning, itching, and edema (buildup of fluid) redness and swelling. ***Eye:*** Severe irritation of the conjunctiva with burning, itching, and watering. Direct contact with heated fumes can irritate the eyes. ***Lung:*** Irritation of the bronchial tubes and other members of the respiratory tract. May cause headache, loss of appetite, nausea, vomiting, slowed reactions, and a loss of strength. ***CNS:*** Depression of the nervous system causing excitement, convulsions, and possibly coma.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
No. 6 Fuel Oil	100 ppm (H_2S)	20 pm (H_2S) 1 ppm (H_2S) 10 ppm (H_2S-ceiling)	***Skin:*** Skin irritation with prolonged contact ***Eye:*** Mild to moderate irritation ***Lung:*** Minimal inhalation hazard at ambient temperature, upon heating, vapors may result in respiratory tract irritation and CNS effects-see below ***CNS:*** Headache, dizziness, loss of balance and coordination, unconsciousness ***Other:*** Irritating and toxic hydrogen sulfide gas may be found in confined spaces. H_2S causes olfactory fatigue, so the characteristic rotten egg odor may not be detected, especially at higher concentrations. Thus, the odor is not a reliable warning indicator for exposure.
Phenol	250 ppm	5 ppm (skin) 5 ppm (skin) 5 ppm (skin)	***Skin:*** Severe irritation and possible burns. Will pass through unbroken skin to cause toxic systemic effects, including weakness, nausea, collapse, and death. ***Eye:*** Severe irritation with damage and possible loss of vision likely. ***Lung:*** Severe irritation to the respiratory tract and associated mucosa. Can cause weakness, nausea, confusion, collapse, coma and death (respiratory failure). ***CNS:*** A central nervous system depressant. Weakness, confusion, mood changes and other personality changes, nausea, collapse and death.
Polyaromatic hydrocarbons (PAHs) (coal tar pitch volatiles)	80 mg/m^3	0.2 mg/m^3 0.2 mg/m^3 0.1 mg/m^3	***Skin:*** Irritation with burning, itching, edema, redness and swelling. ***Eye:*** Severe irritation of the conjunctiva with burning, itching and watering. ***Lung:*** Irritation of respiratory tract, especially bronchial tubes. ***CNS:*** Slowed reactions, headache and loss of strength. ***Other:*** Loss of appetite, nausea, vomiting.

Contaminant	IDLH Level	PEL/TLV/REL	Acute Toxicological Symptoms
Polychlorinated Biphenyls (PCBs)	5 mg/m^3-	PEL:1 mg/m^3-1242 PEL:0.5 mg/m^3-1254 TLV:1 mg/m^3-1242 TLV:0.5 mg/m^3-1254 REL: 0.001 mg/m^3 – 1242 and 1254	***PCBs have low acute toxicity, but because they accumulate in the environment and in animal and human tissues, the potential for chronic toxicity is significant.*** ***Skin:*** Burning sensation and edema of the face and hands, skin thickening, hyper pigmentation, chloracne. ***Eye:*** Eye discharge, swelling of the eyelids. ***Ingestion:*** Severe abdominal pain, nausea, vomiting, diarrhea have been reported following acute and chronic exposures. ***Central and peripheral Nervous System:*** Headache, dizziness, depression and nervousness, weakness and numbness of the extremities.
Trimethyl benzene (mixed isomers)	ND	No PEL 25 ppm 25 ppm	***Skin***: Irritation ***Eye***: Irritation ***Lung***: Irritation of the throat, respiratory system, bronchitis ***CNS***: Headache, lassitude, drowsiness, dizziness, ***Gastrointestinal***: Nausea, vomiting
Other			
Methane	NA	NA	***-Simple asphyxiant-displaces oxygen in air and may cause rapid suffocation.*** ***-Odorless and does not provide adequate warning.*** ***-Highly flammable gas that may form explosive mixtures with air.***

Notes*:*
IDLH – Immediately Dangerous to Life or Health
mg/m3 – milligrams per cubic meter
NA – not applicable (no specific IDLH value has been determined)
ND – not determined (no IDLH value has been formally derived)
NE – not established
ppm – parts per million
STEL – Short-Term Exposure Limit

APPENDIX B – PHYSICAL HAZARDS

Asbestos Removal

- For asbestos removal, a Regulated Area will be constructed in accordance with Occupational Safety and Health Administration (OSHA)1926.1101.
- A digital vacuum pressure gauge must be maintained on-site during the removal activities, and the containment area must maintain a -0.020 inches of water column pressure.
- Warning signs and danger signs will be posted outside the containment area and all personnel performing the asbestos removal must wear safety toed shoes, hard hat, Tyvek (with booties and hoodie), nitrile gloves with polyvinyl chloride (PVC) over gloves, and full-face air purifying respirator.
- During the removal of asbestos containing materials, all asbestos materials must be sprayed with amended water and must be kept adequately wet during the removal process. Perform required personal air monitoring and provide results to staff members. Avoid over watering to control potential runoff. Capture any runoff on poly film and use towels or rags to soak up excessive water.
- Ensure best management practices are in place prior to work, and drains are protected.
- Ground-fault circuit interrupters must be utilized on all powered equipment.
- No cutting tools to be used for non-friable asbestos removal.
- Do not overload, throw, drop, or puncture bags.
- Good housekeeping must be maintained, and work area should be kept free of debris.
- If caulking needs to be cut, ensure to use cut resistant gloves and a self-retracting safety knife.
- A personal decontamination area must be on-site.
- All waste will be double bagged and must be properly marked with required U.S. Department of Transportation (DOT) hazard label, and all waste must be maintained on-site until the required manifest(s) are available and signed for transport and disposal.
- Personal air sampling is required for workers performing asbestos removal work.

Batteries

- Battery inspection: Determine the functionality and potential damage of each battery cell.
- Voltage checks: Implement procedures to check battery voltage before and after charging.
- Discharge prevention: Shut down the battery's ability to discharge below 20 volts.
- Alarm fixes: Improve the specificity of "battery abnormality" alarms and raise the threshold for maximum charge alarms.
- Standard work: Develop a Standard Operating Procedure (SOP), Job Hazard Analysis (JHA), or other standard work guidance for infrequent activities.
- Workplace exams: Conduct regular workplace examinations and eliminate hazardous conditions.
- Proper training: Train staff in the correct use and care of lithium battery-powered equipment, including appropriate personal protective equipment (PPE) usage.

- Follow instructions: Adhere to the manufacturer's guidelines for the storage, use, charging, and maintenance of lithium batteries.
- Fire safety: Keep fire extinguishing equipment readily available and ensure a smoke or heat alarm is installed in areas where devices are charged or stored.

Caught-In Or Between

Below are best practices to mitigate caught in between hazards:

- Ensure operating paths are clear of personnel or objects subject to hazard.
- Operate equipment safely and according to manufacturer's instructions.
- While heavy equipment is operating, all personnel on ground will wear reflective, high-visibility vests and hard hats.
- No one will approach equipment in operation unless the operator gives them positive indication that it is acceptable to do so.
- Put up barriers to avoid unnecessary contact with hazards.
- Ensure egress paths are clear and unobstructed.

Combustible Dusts

For a combustible dust explosion to occur, the following must be present:

- Combustible dust (fuel);
- Ignition source (heat);
- Oxygen in air (oxidizer);
- Dispersion of dust particles in sufficient quantity and concentration; and
- Confinement of the dust cloud.

Dust control:

- Minimize the escape of dust from process equipment or ventilation systems;
- Use dust collection systems and filters;
- Utilize surfaces that minimize dust accumulation and facilitate cleaning;
- Provide access to all hidden areas to permit inspection;
- Inspect for dust residues in open and hidden areas, at regular intervals;
- Clean dust residues at regular intervals;
- Use cleaning methods that do not generate dust clouds, if ignition sources are present;
- Only use vacuum cleaners approved for dust collection;
- Locate relief valves away from dust hazard areas; and
- Develop and implement a hazardous dust inspection, testing, housekeeping, and control program (preferably in writing with established frequency and methods).

Ignition control:

- Use appropriate electrical equipment and wiring methods;
- Control static electricity, including bonding of equipment to ground;
- Control smoking, open flames, and sparks;
- Control mechanical sparks and friction;
- Use separator devices to remove foreign materials capable of igniting combustibles from process materials;
- Separate heated surfaces from dusts;
- Separate heating systems from dusts;
- Proper use and type of industrial trucks;
- Proper use of cartridge activated tools; and
- Adequately maintain all the above equipment.

Damage control:

- Separation of the hazard (isolate with distance);
- Segregation of the hazard (isolate with a barrier);
- Deflagration venting of a building, room, or area;
- Pressure relief venting for equipment;
- Provision of spark/ember detection and extinguishing systems;
- Explosion protection systems;
- Sprinkler systems; and
- The use of other specialized suppression systems.

Compressed Air

Hazards of compressed air use include:

- Flying objects cutting or bruising the body.
- Foreign bodies getting lodged under skin.
- Air or foreign particles causing eye injury that can cause loss of sight.
- Injection of an air bubble into the blood stream that can lead to a fatal embolism.
- Air blown into the mouth that can rupture the esophagus or the lungs (can happen even at a low pressure of 5 psi).
- Noise from an air hose that can lead to hearing loss (sounds can reach 120-130 dB).

Safely working with and around compressed air involves the following:

- Stand clear of areas where compressed air is being used, whenever possible.
- Do not use compressed air to clean clothing or hair.
- Do not point a compressed air hose at yourself or another person.
- Check condition of hoses and lines before use.
- Wear PPE-safety glasses with side shields, full face shields, and hearing protection.
- Do not use compressed air for cleaning workspaces.
- Safety chains or other locking devices should be used at couplings of high-pressure hose lines where a connection failure would create a hazard.

Compressed Gases

- Compressed gases pose both physical and chemical hazards, depending on the type of gas. These gases can range from inert and non-reactive to highly toxic, flammable, or explosive. Due to the high pressure inside cylinders, any physical damage or exposure to high temperatures can result in serious incidents.
- All compressed gas cylinders must be:
  - Clearly labeled with the name of their contents in accordance with OSHA and other applicable regulations.
  - Accompanied by a Safety Data Sheet (SDS), which must be obtained and maintained for each type of gas.
- Always store, transport, and use cylinders upright with the valve facing up. Secure them using appropriate supports such as racks, straps, chains, or stands to prevent tipping or falling.
- Protect cylinder valves with standard caps when not in use—this applies to both full and empty cylinders.
- Never force a cap or regulator; caps should be hand-tightened only.
- Avoid exposing cylinders to excessive moisture, corrosive chemicals, or fumes.
- Do not store cylinders near combustible materials or in areas subject to extreme temperatures.
- Never transfer gases from one cylinder to another (except for dry ice or cryogenic materials). Do not attempt to refill compressed gas cylinders.
- Before use, slowly “crack” the valve to clear dust or debris—ensure the opening is pointed away from people. Take extra precautions with toxic or flammable gases.
- Do not stand in front of the regulator gauge when opening the valve.
- Always use the correct pressure regulator for specific gas. Never operate a cylinder without a regulator.
- Before opening the cylinder, ensure the regulator’s adjusting screw is released. Never allow gas to enter the regulator suddenly.
- If a leak occurs between the cylinder and regulator, close the valve before attempting to tighten the union nut.
- Never strike an electric arc on a cylinder.

Confined Space

A confined space is an area that has limited or restricted means for entry or exit, and it is not designed for continuous staff member occupancy. Confined spaces include, but are not limited to, underground vaults, tanks, storage bins, manholes, pits, silos, process vessels, and pipelines. OSHA uses the term "permit-required confined space" (permit space) to describe a confined space that has one or more of the following characteristics:

- contains or has the potential to contain a hazardous atmosphere;
- contains a material that has the potential to engulf an entrant;
- has walls that converge inward or floors that slope downward and taper into a smaller area which could trap or asphyxiate an entrant; or
- contains any other recognized safety or health hazard, such as unguarded machinery, exposed live wires, or heat stress.

Haley & Aldrich staff members should avoid entering confined spaces whenever possible. Confined spaces can only be investigated by opening the entryway, allowing adequate venting time, and viewing the interior using a flashlight without entering the space or breaking the plane of the opening of the space.

Work in confined spaces requires additional planning, training, permits, control of energy through lockout/tagout, and safety documentation. Confined space work cannot be performed without approval from Regional Safety Managers (RSMs). Contact an RSM for more information on safely completing confined space work.

Congested Areas

- Project sites may present congested working areas with other subcontractors and moving equipment.
- Be mindful of the work happening around you, pay attention to the hazards beyond those of just the task you are performing.
- Stay clear of vehicle pathways.
- Remain visible to operators and receive confirmation before moving across the pathway of any heavy equipment.

Construction Materials

- To prevent fires and hazards, separate incompatible materials during storage. Keep solvent waste, flammable liquids, and oily rags in fire-resistant containers until they are removed from the site.
- All materials stored in tiers must be securely stacked, racked, blocked, or interlocked. Cylindrical materials like steel, poles, and pipes need proper stacking and blocking to prevent shifting and potential injuries.
- Be aware of the maximum safe load limits for floors used as storage areas and never exceed these limits.
- Keep walkways and passageways clear and well-maintained to allow the safe movement of staff members and vehicles.
- When working on different levels, provide ramps or graded surfaces to prevent vehicle accidents and material spillage.

Control of Hazardous Energy

- A system for effective de-energization will be implemented, communicated to personnel, and verified prior to conducting operations in an area affected by energized equipment.
- Personnel will remain alert for energized equipment and will notify their supervisor immediately should they suspect anything around them is still energized or unsafe to approach.
- The steps of lockout/tagout are a primary method of isolating energy and include the following,
  - Lockout/tagout events to be discussed in tailgate safety meetings prior to implementation.
  - Notify all staff members within the immediate affected area that a lockout or tagout is going to be utilized and the reason why.
  - If the equipment is operating, shut it down in an orderly fashion using the normal stopping procedure.
  - Operate the switch, valve, or other energy isolating device(s) so that the equipment is isolated from its energy source(s).
  - Lockout and/or tagout the energy isolating devices with assigned individual lock(s) or tag(s).
  - Lockout devices and tagout devices are to indicate the identity of the employee applying the device(s).
  - Use the TAG Template available on the Haley & Aldrich Intranet Forms/Health & Safety/ Hazardous Energy Control page to generate staff member and equipment-specific tags.
  - Following the application of lockout or tagout devices, all potentially hazardous stored or residual energy shall be relieved, disconnected, restrained, or otherwise rendered safe. For example:
    - Prior to work on hydraulic or pneumatic systems, the energy source shall be turned off and existing pressure drained.
    - Release the tension on springs or block the movement of spring-driven parts.
    - Block parts that could move form loss of hydraulic and pneumatic pressure.
    - Drain process piping systems and close valves to prevent the flow of hazardous materials.
    - Verify a zero-energy state with applicable test equipment, gauges, etc.
    - Verify that timing devices and delayed starts have been isolated.
    - Attempt to energize the equipment.
    - Return the controls to its energy isolation position (i.e. “OFF”).
  - At this point the equipment is considered to be locked or tagged out.
  - If lockout is the energy control method utilized, the authorized staff member is to keep the key in their possession for the duration of the lockout period.

Dehydration

There may be instances where staff members become dehydrated while performing work. To reduce potential for dehydration, staff members should:

- Drink plenty of fluids, including drinks with electrolytes, at regular intervals. OSHA recommends at least one quart of water per hour on days that exceed 80 degrees F;
- Eat foods that are high in water, such as fruits and vegetables;

- Drink additional water in hot or humid weather to help lower your body temperature and to replace what you lose through sweating;
- Drink extra water in cold weather to combat moisture loss from dry air, particularly at higher altitudes; and,
- Ensure that project teams prepare for staff members to have access to adequate hydration.

If at any point staff members are experiencing extreme symptoms of dehydration, contact emergency medical services.

Drilling Hazards

There are several hazards associated with drilling. The primary hazards are the exposed moving parts where the soil borings are advanced. The size of the drill rig may present blind spots for the operator as well as workers moving around the drill rig. Noise is also a significant hazard, particularly when using air rotary equipment.

Use of a drill rig will require all personnel in the vicinity of the operating rig to wear steel-toed boots, hard hats, reflective safety vests, hearing protection, and safety eyewear. Personnel should not remain in the vicinity of operating equipment unless it is required for their work responsibilities.

Oversight personnel should follow these safety procedures:

- Do not stand near cables under tension, such as those lifting drill pipe
- Do not stand directly underneath a load suspended by a cable
- Stand clear as drill pipe is lowered into pipe rack by cable
- Keep away from drill rig unless required by task
- Do not approach equipment without first establishing eye contact with the operator
- Ensure that all machinery have operating back-up alarms
- Keep hands away from moving parts

Additionally, the following safety requirements must be followed:

- All drill rigs and other machinery with exposed moving parts must be equipped with an operational emergency stop device. Drillers and geologists must be aware of this device. This device must be tested before the job starts and periodically thereafter. The driller and helper shall not simultaneously handle augers unless there is a standby person to activate the emergency stop when necessary.
- The driller must never leave the controls of the rig while the tools are rotating unless all workers are kept clear of rotating equipment.
- A long-handled shovel must be used to clear drill cuttings away from the hole and from rotating tools. Hands and/or feet are not to be used for this purpose.
- A remote sampling device must be used to sample drill cuttings if the tools are rotating or if the tools are readily capable of rotating. Samplers must not reach into or near the rotating equipment. If personnel must work near any tools that could rotate, the driller must shut down the rig prior to initiating work.
- Drillers, helpers, and geologists must secure all loose clothing when in the vicinity of drilling operations.

- Only equipment that has been approved by the manufacturer may be used in conjunction with drilling equipment and specifically to attach sections of drilling tools together. Pins that protrude excessively from augers shall not be permitted.
- No person may climb the drill mast while tools are rotating.
- No person may climb the drill mast without the use of American National Standards Institute (ANSI)-approved fall protection or portable ladder that meets the requirements of OSHA standards.
- The drill rig must not be moved with the mast in a raised position.
- Elevated parts of the drill rig shall remain at least 20 feet from overhead power lines or follow the procedures outlined in Overhead Utilities below.

Drum Handling

- Use proper lifting and moving techniques to prevent back injuries.
- Make sure the vehicle selected has sufficient rated load capacity to handle the anticipated loads, and make sure the vehicle can operate smoothly on the available road surface.
- Have overpacks ready before any attempt is made to move drums.
- Before moving anything, determine the most appropriate sequence in which the various drums and other containers should be moved.
- Exercise extreme caution in handling drums that are not intact and tightly sealed.
- Ensure that operators have a clear view of the roadway when carrying drums.
- Pressurized drums are extremely hazardous. Wherever possible, do not move drums that may be under internal pressure, as evidenced by bulging or swelling.
- Make sure that all waste drums are managed according to state, federal, and PG&E regulations and requirements.

Economically Depressed Areas

- Economically depressed areas may have high crime rates.
- Staff could be subjected to crime that includes but may not be limited to thievery, vandalism, and violence.
- Prior to the start of work, staff need to understand the work locations and the potential for exposure to low level crime.
- Staff members should never work alone in these areas.
- A buddy system is required.
- Conduct work during daylight hours.
- Secure equipment and vehicles.
- If warranted, contact the local police department for a security detail.
- Leave the work area immediately and contact the local authorities if staff members feel threatened, or are threatened.

Electrical Hazards

- Electrical work of 120 volts or less can be completed by a qualified person, who has received training and has demonstrated skills and knowledge in the construction and operation of electric equipment and installations and the hazards involved.
- Electrical work of over 120 volts but under 600 volts can be completed by a qualified electrical worker as defined as a person with who by reason of a minimum of 2 years of training and experience with high-voltage circuits and equipment and who has demonstrated by performance familiarity with the work to be performed and the hazards involved.

Energized Equipment and Electrical Shock

- Document process to de-energize or isolate energy sources.
- Ensure staff are appropriately trained to conduct work requiring lockout/tagout.
- Affix lock and/or tag to equipment to ensure improper start-up or release of energy.
- Execute an Energy Isolation Permit.
- Electrical equipment and power tools must be operated and maintained in accordance with manufacturers' requirements.
- Electrical equipment, tools, switches, and outlets must be protected from environmental elements.
- Check manufacturers' requirements.

Equipment and Moving Vehicle Safety

The following procedures can be used to minimize the risk of traffic-related hazards and working near heavy equipment:

- Always wear seat belts when operating vehicles or heavy equipment, unless the equipment is designed for stand-up operation or lacks a rollover protective structure.
- Check vehicles and equipment before each shift to ensure all parts and accessories are in safe working condition.
- Obey all traffic laws and any additional on-site vehicle rules.
- Do not use cell phones or other distractions while driving.
- Allow adequate travel time to avoid speeding.
- Only drive on roadways or grades that are safely constructed and maintained.
- Do not exceed a vehicle's rated load or lift capacity.
- Do not carry passengers unless the vehicle is equipped with a designated and safe seating area.
- Operators must meet all requirements (e.g., licenses, certifications, training) for the specific equipment they are using.
- Do not drive in reverse with an obstructed rear view unless the vehicle has an audible reverse alarm, or a spotter signals it is safe.
- Use a dedicated spotter when operating or maneuvering heavy equipment.

- Set parking brakes when vehicles are parked, and chock wheels if on an incline.
- Lower or block buckets and place all controls in neutral when equipment is not in use.
- Ensure all vehicles have adequate braking systems and safety devices.
- Use traffic signs, barricades, or flaggers when working near public roadways.
- Clearly define work zones using reflective tape, traffic cones, or similar markers.
- Use orange flashing lights to alert drivers to hazards or changes in traffic patterns.
- Workers must wear high-visibility clothing.

Excavation and Trenching Safety

- Contact the proper utilities to obtain clearance. Prior to work, review the utilities in the area and be sure they have been staked properly. Before work begins, staff obtain multiple lines of evidence, using guidance found in OP1020 Working Near Utilities.
- Be aware that trenches and excavations deeper than 4 feet are considered confined spaces and require additional safety precautions, such as shoring. If an excavation exceeds 4 feet in depth, contact the Client or Site Safety Officer (SSO) to review the original Safe Work Permit and ensure that it is adequate. A 4-gas meter monitoring will be required in addition to the existing air monitoring plan.
- The walls and faces of all excavations and trenches more than 5 feet deep, in which a staff member is exposed to danger from moving ground, will be guarded by a shoring system, sloping of the ground, or some other equivalent means. The design of shoring systems in excavation greater than 20 feet must be done by a registered Professional Engineer as per 29 Code of Federal Regulations 1926 Part P.
- Excavated or other material will be effectively stored and retained at least 2 feet or more from the edge of any excavation or trench.
- Make daily inspections of excavations. If evidence of possible cave-ins or slides is apparent, all work in the excavation will cease until the necessary precautions have been taken to safeguard staff members.
- Trenches more than 4 feet deep will have ladders or steps available so no more than 25 feet of lateral travel is required.
- Determine soil composition (through soil sampling and/or soil maps) and other relevant site conditions, with special emphasis on conditions conducive to cave-ins.
- Monitor the atmosphere in and around excavation on a regular basis to check for explosive, toxic, or otherwise dangerous gases and vapors. Be sure sources of nuisance and harmful gas are not near the trenches; for example, do not park vehicles or equipment next to the excavation where the fumes can enter it.
- Water will not be allowed to accumulate in any excavation. Utilize ditches, dikes, pumps, or other means to keep surface water out of trenches.
- All open excavations must be well marked and barricaded.

- Staff members involved in the excavation activity have appropriate training in safe excavation practices, with emphasis on factors such as:
  - Utility line identification.
  - Cave-in prevention measures.
  - Recognition of conditions which may cause cave-ins.
  - Means of egress from trench.
- Excavations greater than 5 feet in depth are not to be entered unless properly sloped, shored, or benched.
- No project staff member is to enter the excavation unless they have determined that there is no potential for injury such as cave-ins and/or atmospheric hazards.
- It is the CM and project staff's responsibility to safeguard all affected workers from falling into any excavation by the use of barriers and warning signs.
- No excavation is to be left open or unattended when there is a possibility for anyone to fall into the excavation. Barricades will be placed around excavation where there is a fall hazard by workers and/or equipment; and if needed will be lighted at night. This is the responsibility of the subcontractor.
- Keep all equipment at a minimum of 10 feet away from energized electrical lines

Extended Shift

- Extended work hours may disrupt the body's regular schedule, leading to increased fatigue, stress, and lack of concentration.
- The degree to which an individual is exposed to fatigue risk factors depends upon the work schedule. As both the duration of the workday and the number of days worked increase, so do the fatigue risk factors.
- Staff Managers need to be aware of the fatigue risk factors and ensure projects are structured to mitigate these factors.
- Staff Members also have a responsibility to manage the personal fatigue risk factors that they can control outside of work (e.g., duration and quality of sleep, diet, drugs, and alcohol).
- Fatigue symptoms, both mental and physical, vary and depend on the person and degree of overexertion. Examples include:
  - Weariness
  - Sleepiness
  - Irritability
  - Reduced alertness
  - Lack of memory
  - Concentration and motivation
  - Increased susceptibility to illness
  - Depression
  - Headaches

  - Loss of appetite
  - Digestive problems

- When possible, managers should limit use of extended shifts and increase the number of days worked.
- Additional breaks and meals should be provided when working extended shift periods.
- Tasks requiring heavy physical labor or intense concentration should be performed at the beginning of the shift, if possible.
- Make efforts, when feasible, to ensure that unavoidable extended work shifts and shift changes allow affected staff members time for adequate rest and recovery.
- PMs need to plan to have an adequate number of personnel available to enable workers to take breaks, eat meals, relax, and sleep.
- Plan for regular and frequent breaks throughout the work shift.
- If at remote sites, ensure if possible, that there is a quiet, secluded area designated for rest and recuperation.
- Encourage use of micro breaks to change positions, move about, and shift concentration.
- Personnel should look to obtain an adequate quantity and quality of sleep.

Fuel Operations

Below are best practices to prevent spills or releases during fueling activities:

- Conduct fueling operations from a leak-proof nozzle or safety fuel can with a fitted gooseneck nozzle or funnel.
- Use screens to prevent debris and particles (from debris or ice in cold weather) from entering fuel tanks.
- Secondary containment shall be placed under the equipment during fueling to prevent any loss of containment to the ground surface.
- Store sorbent pads near fueling areas during all fueling operations to prevent fuel leaks or spills from travelling.
- Use flashlights in low light areas or in other than daylight hours to prevent over filling fuel tanks.
- Fuels will be stored in approved containers that are placed in a secondary containment capable of storing 100 percent of the spilled material.
- Equipment must be shut down before fueling, and drips will be wiped clean, and the fuel cap must be securely fastened before restarting the equipment.
- Store the fuel away from combustible materials and oxidizers.
- Fuel storage areas will be located away from common walkways and work areas.

Geoprobe® or Other Direct Push Rig Use

Use of a Geoprobe® or other direct push rig will require all personnel in the vicinity of the operating rig to wear steel-toed boots, hard hats, hearing protection and safety eyewear. Personnel should not remain in the vicinity of operating equipment unless it is required for their work responsibilities.

The following general guidelines apply to the operation and transport of any direct push rig.

Hazards

- Pinch points - Geoprobe® rod ends, auger joints, stacked rods, or augers
- Rotating augers - loose clothing
- Low head clearance when working directly over hole beneath mast
- Overhead and underground utilities
- Back strain - auger or Geoprobe® rod handling
- Use of cutting tools to open acetate core sleeves
- Accidentally engaging operating controls
- Noise

General Operating Procedures

- All drill rigs and other machinery with exposed moving parts must be equipped with an operational emergency stop device. Drillers and geologists must be aware of this device. This device must be tested before the job starts and periodically thereafter. The driller and helper shall not simultaneously handle augers unless there is a standby person to activate the emergency stop when necessary.
- Only equipment that has been approved by the manufacturer may be used in conjunction with drilling equipment and specifically to attach sections of drilling tools together. Pins that protrude excessively from augers shall not be permitted.
- Elevated parts of the drill rig shall remain at least 20 feet from overhead power lines or follow the procedures outlined in “Overhead Utilities” below.
- One person will be assigned the responsibility of lead operator. The lead operator will be responsible for operating the rig and performing the daily checklist. One additional personnel will assist with handling Geoprobe® rods. When augering, an additional person is recommended for assisting with auger flights. In either case, a separate person will be required for collection of soil samples or logging cores.
- A safe zone will be established using banner guard or physical barriers to prevent unnecessary personnel from entering the work area.
- Operations and crew will be familiar with the operator’s manual and will review the practical training on rig use.
- The driller must never leave the controls of the rig while it is running.
- Keep hands away from moving parts.
- Drillers, helpers, and geologists must secure all loose clothing when in the vicinity of drilling operations.
- Wear heavy gloves when handling Geoprobe® rods or augers.
- Hearing protection must be worn.
- Lifting tasks should be accomplished by using the legs to lift instead of the back (bending at the knees instead of at the waist). A second person should help lift the item if it is too large or heavy for one person.

- For protection of staff members from cut injuries during sampling activities or other activities where knives may be used, first evaluate whether hand knives are the correct tool for the task. Where possible, use a safer cutting tool such as scissors or snips. Cut resistant gloves such as Kevlar must be worn. If using a knife/ box cutter, it should have a self-retracting blade. When using a knife, cut away from oneself and end the knife stroke away from the body. Hold the item firmly and do not cut downwards and towards the body. Personal knives (e.g., pocket knives) are not to be considered as a tool for any type of work-related cutting.
- The rig and inspection logs will be maintained.
- During auger flight connection, pinch potential will be controlled by keeping hands away from the joint (heavy gloves must be worn). Constant communication between lead operator and helper must be maintained.

Oversight Personnel Should Follow these Safety Procedures:

- Do not stand near cables under tension such as those lifting drill pipe
- Do not stand directly underneath a load suspended by a cable
- Stand clear as drill pipe is lowered into pipe rack by cable
- Keep away from drill rig unless required by task
- Do not approach equipment without first establishing eye contact with the operator
- Ensure that all machinery have operating back-up alarms
- Keep hands away from moving parts

Daily Maintenance Checklist (for subcontractors):

- Check all fluids.
- Test kill switch.
- Ensure that back-up alarm is operating.
- Confirm mandatory underground utility clearances, double check for indications of utilities not reported by appropriate state clearing centers.
- Before raising mast, look up for overhead obstructions.
- Review location of kill switch and fire extinguisher with entire crew.
- Maintain safe distance from all electrical power lines.
- Decontaminate entire rig prior to leaving site.
- Check that fire extinguisher is charged.

Maintenance of Rig (for subcontractors):

- Regular inspection and maintenance will be performed by qualified personnel. A full check-out will be performed on a monthly basis and immediately following extensive use.
- The daily inspection will be completed by the rig operator.

Hand Tools

- Use tools that are in good condition.
- Ensure handles are not split, mushroomed, or otherwise damaged.
- Defective tools shall be removed from service.
- Use the proper tool for the job.
- Wear eye protection and hand protection depending on the potential hazard
- Maintain secure footing when using any hand tool
- Do not use fixed blade open knives, unless approval is obtained by Health & Safety. Examples include pocketknives, multi-tools, hunting knives, machetes, standard utility knives, and box cutters.
  - There are safer alternatives, such as self-retracting utility knives and guarded utility knives, that can effectively accomplish tasks while providing additional protection to staff members.
- For any tasks that involve cutting, staff must:
  - Wear cut score A2 cut resistant gloves.
  - Cut away from oneself and end the knife stroke away from the body. Hold the item firmly and do not cut downwards and towards the body. Cut into the air or onto something hard.

Heavy Equipment and Traffic – Working Around

- Before approaching a piece of heavy equipment or heavy equipment work zone, establish eye contact with the equipment operator. The equipment operator must put the equipment in a neutral energy state (e.g., coring equipment stopped from pushing or rotating) and then remove their hands from the controls.
- Once this is done, it is acceptable to approach the piece of equipment or heavy equipment work zone.
- The equipment operator must keep their hands off the equipment controls for the duration of time someone is in the work zone.
- Maintain visual contact with operators at all times and keep out of the strike zone whenever possible.
- Always approach heavy equipment with an awareness of the swing radius and traffic routes of each piece of equipment and never go beneath a hoisted load.
- Never position yourself between fixed objects and running equipment and/or between two running pieces of equipment.
- When possible, use a spotter or backer while equipment is being moved.
- Workers must be highly visible in all levels of light. Warning clothing, such as red or orange vests, are required while working near vehicles or any heavy mobile equipment. If worn for night work, clothing must be of reflective material.
- Be aware of traffic patterns and make sure to stay well away from these areas as a pedestrian.
- Be aware that debris may be kicked up or fly out of passing vehicles.
- Be especially careful and alert when working near heavy equipment since equipment failure or breakage can lead to accidents and worker injury.
- Avoid breathing fumes created by heavy equipment exhaust.

Heavy Equipment and Traffic – Operating

- Create a traffic control plan.
- Check vehicles before each shift to assure that all parts and accessories are in safe operating condition.
- Obey all traffic rules as well as an additional vehicle rules on-site.
- Do not speed. Make sure to leave adequate time to arrive at your destination on time.
- Do not drive a vehicle in reverse gear with an obstructed rear view, unless it has an audible reverse alarm, or another worker signals that it is safe.
- Drive vehicles or equipment only on roadways or grades that are safely constructed and maintained.
- Make sure that you and all other personnel are in the clear before using dumping or lifting devices.
- Lower or block buckets and leave all controls in neutral position when not in use (e.g., end-loader buckets, dump bodies).
- Set parking brakes when vehicles and equipment are parked and chock the wheels if they are on an incline.
- All vehicles must have adequate braking systems and other safety devices.
- Do not exceed a vehicle's rated load or lift capacity.
- Do not carry personnel unless there is a designed and safe place to ride.
- Use a dedicated spotter while heavy equipment is in operation.
- Use traffic signs, barricades, or flaggers when construction takes place near public roadways.
- Ensure work zones are defined (e.g., reflective tape, traffic cones).
- Use orange flashing lights to make drivers better aware of potential hazards and traffic pattern changes.
- If operating heavy equipment, personnel must meet all the requirements to operate that specific heavy equipment (e.g., licenses, certifications, training).

Hot Surfaces

- Cover hot surfaces with materials that can withstand high temperatures and prevent direct contact.
- Implement measures like ventilation, reflective shields, and air conditioning to reduce heat exposure.
- Provide appropriate gloves, heat-resistant clothing, and other gear to protect workers from burns.
- Identify potential hazards associated with hot surfaces and assess the risk of burns.
- Educate workers on the risks of hot surfaces, how to identify hazards, and how to use PPE effectively.
- Conduct routine inspections to ensure controls are effective and PPE is in good condition.
- Use clear warning signs, labels, and physical barriers to alert workers to hot surfaces.

Hot Work

Hot work activities include burning, welding, grinding, braising, soldering, and using fire or spark-producing tools. The main hazards associated with hot work are getting burned directly by the hot work activity or by fires or explosions that result from an accumulation of combustible materials.

Performing hot work in classified and non-classified areas may be considered a hazardous activity, and a Permit to Work may be required. In general, the Hot Work Permit has five purposes:

- To serve as written permission to do the work.
- To provide a minimum checklist prior to the commencement of hot work.
- To outline the steps necessary for making the work site safe for conducting hot work.
- To alert operating personnel to the hot work in progress.
- To provide a record of safe work practices performed during the permitted activity.

All work shall be conducted in accordance with OP1034 - Hot Work.

Combustible and flammable materials must be kept at least 35 feet from ignition sources. A trained person must act as a fire watch during hot work and 30 minutes after hot work is completed. A properly inspected and tagged fire extinguisher must be readily available to the fire watch.

Housekeeping

Haley & Aldrich strives to provide a clean, hazard-free and safe environment for staff members. Each staff member is expected to take an active role in maintaining a safe environment. Workplaces must be kept neat, clean and organized. Staff members are expected to observe good housekeeping practices at offices and project sites. Such practices must:

- Keep work areas clean and free of debris that might cause safety hazards.
- Maintain order on project sites to avoid tripping hazards. Equipment, tools, extension cords, coolers and supplies must be kept away from areas with foot traffic. These items must be put away and stored in a safe manner at the end of the workday.
- Prevent slip and fall hazards by keeping project sites clean and free from oil, grease, mud, and other slippery materials. Clear away snow and ice.
- Unsafe conditions at offices (e.g., ice on the sidewalk) shall be reported to the office manager to resolve with the property owner.
- Ensure that aisles, walkways, entrances, exits, and access to first aid and spill materials are kept free of obstructions.
- Collect and properly dispose of waste.
- All spills should be reported or cleaned up promptly.
- Avoid placing materials where they may become contaminated.
- Do not place tools or materials on elevated surfaces where they may fall and strike another worker.

- Remove sharp objects and debris including scrap metal, broken glass, nails, etc. from the work area.
- Ensure subcontractors keep the work area and the rest of the site in a neat and orderly fashion.

Periodic inspections of office areas and project sites are conducted by the Corporate Health & Safety Team. To protect against slips, trips, and falls, Haley & Aldrich requires its staff members to wear appropriate footwear (skid resistant soles) and watch for hazards such as uneven terrain, holes, ditches, wires, ropes, hoses, or any other materials or equipment in the path of travel. Tripping caused by wearing disposable footwear can be reduced by selecting properly sized disposable boots. Workers will also change work speed in muddy, snowy, or icy conditions.

Hunting Season

The PM will be responsible for identifying and informing the field crew of the potential for hunting activities in the general area of field activities.

- The field crew will be required to wear appropriate safety clothing such as, but not limited to, orange vests and/or hats when conducting field work during the hunting season in areas where it is likely to occur.

Inadequate Lighting

Some work may need to occur in areas that are not well lit. To reduce hazards of working in low light:

- temporary lighting can be used to brighten the site, such as flashlights or headlamps, if possible.
- Clear safety glasses should be worn under these conditions.
- If conditions are dark and visibility is poor, staff members should pause work and work with project teams to implement mitigations or postpone work until lighting is better.

Ladders

- Always maintain three points of contact when using a ladder.
- Only use ladders on a smooth and level surface.
- Do not exceed the maximum load rating of the ladder.
- Ladders shall always be maintained and kept in good condition.
- Lubricate metal bearings frequently (e.g., locks, wheels, pulleys).
- Safety feet and other auxiliary equipment shall be kept in good condition to ensure proper performance.
- Ladders shall be inspected prior to use, and those which have developed defects shall be withdrawn from service for repair or destruction and tagged or marked as "Dangerous, Do Not Use."
- Ladder rungs should be kept free of grease and oil.
- Fixed ladders that equal or exceed 24 feet must be equipped with ladder safety devices. If using multiple ladder sections, each ladder section is not to exceed 50 feet in length and must use a cage or well. Alternatively, self-retracting lifelines and rest platforms at intervals of 150 feet or less can be used.

- Side rails of through or side-step fixed ladders must extend 42 inches above the top level or landing platform served by the ladder.
- All on-site personnel shall review and be familiar with the requirements of OP1027 - Ladder Safety.

Lasers

- Restrict access and designate laser-controlled areas to restrict access to only authorized personnel who have completed laser safety training.
- Post appropriate warning signs at the entrance(s) of laser-controlled areas to warn about potential hazards.
- Ensure the area is set up and restricted to prevent laser beam transmission outside the controlled area.
- Never enter a laser-controlled area unless authorized and equipped with proper PPE.

Line of Fire - Lifting Equipment and Overhead Hazards

- Install barriers, shields, or screens around unsafe areas.
- Identify and maintain safe walking areas.
- Address overhead work concerns and avoid unsafe zones.
- Keep a safe distance between you and any suspended loads, heavy equipment, or other line of fire hazards.
- Never stand under a suspended load.
- Avoid areas where there is potential for dropped objects.
- Remain aware of stored energy, and where equipment or objects may fall when energy is released.

Line of fire - Lines Under Pressure

Injection hoses operating under pressure may release stored energy if connections fail or hoses otherwise become dislodged. Oversight personnel may be sprayed with injection chemicals and/or be injured by violent hose movements. Staff members shall maintain a safe distance from connection points during injection activities and wear safety glasses. Should the need arise to stand closer to connection points, a face shield should be worn over safety glasses. Contractors should be using whip checks to secure hoses that may become loose or dislodged.

Manual Lifting

Wherever possible, material handling will be done mechanically. Where manual handling is absolutely necessary, personnel will be instructed in safe handling techniques and will be instructed to use the appropriate protective gear to prevent abrasions, cuts, and struck-by accidents. Personnel will also be encouraged to request assistance from other site personnel when lifting large, heavy, or awkward objects (greater than 50 pounds).

The following steps describe the proper method for lifting:

- Get a good footing.
- Place your feet about shoulder-width apart.
- Bend your knees to pick up the load. Never bend from the waist.
- Keep your back straight.

- Get a firm hold. Grasp opposite corners of the load, if possible.
- Keep your back as upright as possible.
- Lift gradually by straightening your legs. Do not jerk the load.
- Keep the weight as close to your body as possible.
- When changing directions, turn your entire body, including your feet.
- Do not twist your body or make awkward moves forcing you to be off balance.

Mechanical Equipment

- Use physical guarding and barriers whenever possible to reduce potential for injury around mechanical equipment.
- Personnel should exercise caution when working with mechanical equipment to prevent entanglement with clothing, placing body parts near pinch points on equipment or using equipment on slopes or unstable surfaces.
- Site personnel and visitors who are not performing necessary work shall remain a distance of at least 15 feet away from moving parts on such equipment.

Material and Equipment Handling

The movement and handling of equipment and materials on site pose a risk to workers in the form of muscle strains and minor injuries. These injuries can be avoided by using safe handling practices; proper lifting techniques and use of safety equipment including steel-toed boots, hard hats and work gloves; and testing the weight of an object before attempting to lift or carry it. Where necessary, mechanical devices will be used to assist in moving equipment and materials.

Proper Lifting Procedure:

- Get a good footing.
- Place feet shoulder width apart.
- Bend knees to pick up load. Do not bend from waist.
- Keep back straight.
- Get a firm hold on load. Grasp opposite corners of the load if possible.
- Keep the back as upright as possible.
- Lift gradually by straightening the load-do not jerk the load.
- Keep the weight as close to the body as possible.
- When changing direction, turn the entire body, including the feet.
- Do not twist the body.

General Requirements for Powered Industrial Trucks are:

- They must be inspected daily before being placed into service, to detect safety problems.
- High-lift rider trucks must be fitted with an overhead guard to protect the operator from falling objects.

- If the load being carried obstructs forward view, the load must travel with the load trailing.
- When a unit is left unattended, the load must be fully lowered, the control Level positioned in neutral, the power shut off, and the brakes set.
- Trucks, trailers or railroad cars being unloaded or loaded with lift trucks must be secured by setting their brakes and placing wheel chocks under the rear wheels.
- Any driver-operated equipment (truck, tractor) used on a site with uneven terrain must have some form of rollover protection.

Naturally Occurring Radioactive Materials (NORM)

- Practice As Low As Reasonably Achievable (ALARA) principles by minimizing the time spent near, increasing your distance from, and using appropriate shielding materials, such as lead or concrete, to block radiation.
- Be aware that NORM can be present in various materials, like rocks, soil, and building materials, as well as in specific industrial equipment.
- Do not eat, drink, or smoke in areas where NORM may be present.
- Contamination Control:
  - Avoid activities that generate dust, such as cutting or grinding, which can contain NORM/ Technologically Enhanced Naturally Occurring Radioactive Material (TENORM).
  - Protect any wounds or cuts to prevent potential contamination.
  - Practice good hygiene, including hand washing after working with or around NORM/TENORM.
- Monitoring and Decontamination:
  - Use appropriate radiation monitoring equipment, like alarming dosimeters, to track exposure.
  - Ensure workers are checked for contamination before leaving a NORM/TENORM work area.
  - Decontaminate equipment and work areas appropriately.
- PPE: Utilize PPE like respirators, coveralls, boots, and gloves, as needed.

Needles

- If you find a discarded needle, stop and do not approach or attempt to pick up the needle.
- Notify the client, controlling employer, or other designated safety personnel immediately.
- Clearly describe the location of the needle to ensure it can be safely addressed.
- Treat all needles, blood, and other potentially infectious materials as if they are infectious.
- When discarded, needles must be placed in SHARPS containers and not in traditional trash.

Night Work

- Minimize or redesign routine administrative tasks to ensure staff members can focus on core duties during their night work.
- Limit consecutive night shifts worked.
- A project requiring staff to work beyond four nights requires a fatigue management plan.

- Ensure that rosters allow for at least two full night's sleep after the last night shift before returning to day shift.
- Regular and frequent breaks are needed.
- Comprehensive training on fatigue risk factors for the staff members assigned to the project.
- Identify optimal sleeping conditions during the day (e.g., dark/quiet rooms, minimize distractions, light meal prior to sleep).

Noise Exposure (e.g., loud equipment, impact noise, noise from tools)

- Wear appropriate hearing protection such as earplugs or earmuffs in high-noise areas.
- Check the Noise Reduction Rating (NRR) of your hearing protection to ensure it provides adequate protection for the noise levels you're exposed to.
- Maintain distance from loud equipment whenever possible to reduce noise exposure.
- Take regular breaks in quiet areas to give your ears time to recover from prolonged noise exposure.
- Use tools like the NIOSH Sound Level Meter app to monitor and understand noise levels on-site and assess the need for hearing protection.
- Be aware of the potential for sudden loud noises. While it's best to wear hearing protection at all times in noisy environments, always follow posted signage indicating when hearing protection is required.

Operations On, Over, or Near Water

Staff are not permitted to perform work on, over, or near water without first informing their RSM and developing additional safety plans. Coast Guard approved flotation devices will be worn to protect staff members from the risk of drowning.

General hazards of walking around water bodies:

- Enhanced potential for slips, trips, and falls.
- Engulfment/entrapment.
- Rising water conditions.
- Enhanced physical exertion from wearing waders, hauling equipment to areas inaccessible to vehicles; this will increase the likelihood of fatigue leading to muscle strains, heat stress, or cold stress.
- Increased chance of encounters with biological hazards.

Protective measures for working around water:

- Ensure team has proper PPE, such as Coast Guard approved floatation devices, lifeline, life buoy, or audible alarms.
- Use the buddy system.
- Conduct daily inspections of work areas, equipment, and safety devices.
- Install guardrails or other barriers to reduce potential for falling into water.
- Ensure team is aware of emergency procedures and prepared to respond in the event of an emergency.

- Be vigilant about weather changes.
- Dress appropriately for weather and other conditions of the site.
- Remain hydrated and maintain electrolyte balance.
- Make sure all walking areas and work surfaces are clean, dry, clear of debris, etc.
- Keep all gear secure when not in use.
- Keep stairs, ladders, doorways, ramps, walkways, and gangways clear.
- Safely secure ramps or gangways when loading and offloading.
- Check for items such as loose boards or nails that stick out on docks and repair as necessary.
- Wear footwear with slip-resistant soles.
- Use a non-skid deck compound where possible.
- Do not allow any part of your body to be between the dock and the boat.

Overhead Utilities

Before beginning equipment operations, the contractor will identify the work zone and determine if any part of the equipment, load line, or load could get closer than 20 vertical or horizontal feet to a power line. If so, the contractor will:

- Determine the line's voltage and maintain clearance distances both vertical and horizontal in accordance with the table below, or
- Arrange with the utility to de-energize and ground the power line, or
- Modify the work to maintain a 20 foot clearance distance by implementing the measures specified in 29 CFR 1926.1408(b) including conducting a planning meeting, erecting, and maintaining an elevated warning line, barricade, or line of signs in view of the operator equipped with high visibility markings at the minimum clearance distance, using a proximity alarm, dedicated spotter, or range limiting device.

Table B. Equipment Travel Clearance Distances for Uninsulated Power Lines	
Voltage (nominal, kV, alternating current)	**Minimum clearance distance (feet)**
up to 50	10
over 50 to 200	15
over 200 to 350	20
over 350 to 500	25
over 500 to 750	35
over 750 to 1,000	45
over 1000	As established by the utility owner operator or registered PE who is a qualified person with respect to electrical power transmission and distribution

Pinch Points

A pinch point is a place where a body part can get caught between moving parts of a machine, between moving and stationary machine parts and between moving parts and other materials. Other situations where pinch point injuries can occur include:

- Catching fingers, hands, toes, or feet under or between heavy crates or equipment or drums while moving them
- Slamming fingers or hands in a door
- Nipping fingers or hands with hand tools
- Nipping fingers or hands with equipment that has sliding parts or hinges
- Nipping fingers or hands while closing a container
- Getting clothing, hair, or jewelry tangled in a pinch point

Pinch point injuries can be prevented by:

- Using machine guards
- Never reaching around, under, or through a guard
- Reporting guards that are missing, damaged, or not working properly
- Lockout/tagout of equipment before repairing or servicing
- Looking for pinch points before starting any task
- Lifting the edge of a heavy item slightly before picking it up to assess its weight and if you are able to lift the weight unassisted-an awkward or heavy load can slip and pinch or crush hands or feet. Get help or use material handling aids to move heavy or awkward items.
- When placing a heavy item on a shelf, pallet, floor etc. or closing a heavy door, make sure there is enough room so the item will not land on your feet
- Sliding the item into place while moving feet and hands out of the way
- Concentrating on the task at all times

Power Tools

Potential Hazards:

- Electrical
- Eye injuries
- Hand injuries
- Musculoskeletal Injuries
- Dust inhalation

Safe Work Practices and Control Measures:

- Personnel will be trained in the use operation and proper handling of portable power tools before they use them.
- Ensure the right tool is used to perform the job.
- Before use, the tool must be inspected for loose power cord connections and frays, and damage to the casing, cord, and plug. The grounding prong must be in good condition, if present.
- Electric tools must be grounded (i.e., have three pronged plugs) and be plugged into a properly grounded outlet or be double insulated. Always hold the tool by the insulated gripping surface instead of the cord.
- Extension cords with three-pronged grounding plugs must be plugged into a three-pronged outlet when using grounded tools.
- When using power tools, grounded surfaces such as pipes or radiators shall not be touched, as there is a greater risk of electric shock if your body is grounded.
- When working with electric tools in damp or wet locations, ground fault circuit interrupter (GFCI) protection must be used.
- Do not abuse the cord, carry the tool by its cord, or pull the cord to unplug it. Keep cord from heat, oil, sharp edges, or moving parts. Replace damaged cords immediately.
- Unplug the tool immediately after use, before removing or changing bits, and before performing any service or maintenance on the tool.
- During inspection, ensure that the trigger turns the tool when “on” and stops the tool in the “off” position. Make sure the chuck is tightly secured to the spindle.
- Loose clothes, gloves, jewelry, or hair can be caught in moving parts, so they must not be worn or be controlled (tie back hair).
- Always wear safety glasses or goggles and, if needed, a face shield.
- Wear hearing protection and a particulate respirator, if dust is generated.
- Hold or brace the tool securely. Brace against stationary things for maximum control.
- Avoid bending the wrists and other awkward body postures, such as twisting the torso.
- Take frequent breaks from tool use to avoid musculoskeletal injuries from repetitive motion.
- Use proper lifting techniques for handling tools. Do not lift more than 55 pounds alone, and use proper body mechanics - lift with your legs, not your back. Do not twist the body when picking up a load.

Projectiles

- Be aware that many tasks, such as cutting, grinding, or prying, can create flying debris or projectiles.
- Implement controls to prevent tools and materials from falling, such as securing tools, using toe boards, guardrails, debris nets, or canopies.
- Secure materials that could become airborne or projectiles.
- Use tool tethers to prevent tools from falling from heights and becoming projectiles.

- Barricade areas where work creating projectiles is taking place and prevent unauthorized personnel.
- Wear proper PPE, including hard hats, eye protection, and safety-toed boots.

Public Right Of Way

- Haley & Aldrich staff and their subcontractors conducting work on public roads and/or rights of way can be exposed to vehicular traffic and expose the public to the hazards of the job site.
- Where a hazard exists to site workers because of traffic or haulage conditions at work sites that encroach public streets or highways, a system of traffic controls in conformance with the Manual on Uniform Traffic Control Devices for Streets and Highways (MUTCD), or state program, is required.
- A Temporary Traffic Control Plan (TCP) describes traffic controls to be used for facilitating vehicle and pedestrian traffic through a temporary traffic control zone. TCPs are required to provide for worker protection and safe passage of traffic through and around job sites with as little inconvenience and delay as possible.
- The plan may range in scope from being very detailed, to merely referencing typical drawings contained in the MUTCD.
- The degree of detail in the TCP depends entirely on the complexity of the situation, and TCP's should be prepared by persons knowledgeable about the fundamental principles of temporary traffic control and the work activities to be performed.
- Haley & Aldrich PMs or their subcontractors need to establish appropriate control measures and obtain any permits when project work is on or encroaches public roadways.
- You may need flaggers or police details.
- Cease work and notify the field supervisor immediately if any conditions are such that safety is jeopardized.
- Utilize protective vehicles whenever appropriate, or position equipment so it is in between the work and oncoming traffic.

Railroad Right Of Way

- Typically, the presence of anyone, equipment, and/or material is "in the railroad area" (closer than 15 feet of the nearest railroad rail, centenary, electrical wire, signal wire, or other signal facility) must have permission of the railroad company prior to initiating work. All work must avoid causing damage to trains, tracks, or other facilities of the railroad company, and not to interfere with movement of trains or railroad track ballast.
- Railroad companies may require a flagger when construction operations encroach "the railroad area."
  - The flagger is assigned to a construction project to ensure safe passage of trains or on-track equipment by communicating with trains regarding the movement of construction equipment on/near active tracks.
  - Notify the flagger whenever necessary to be "in the railroad area" and proceed only after the flagger has indicated it is safe to do so.
  - The tracks must be cleared promptly anytime the flagger indicates to do so.
- Trains may need 1.5 miles to come to a complete stop.

- New trains are quiet and can cover a mile in less than 30 seconds producing enormous amounts of wind turbulence.
- The slipstream is powerful enough to drag you under the wheels of the train if standing next to the track.
- Communication protocols need to be established prior to being "in the railroad area."
- Tracks are clearly defined and known by all parties involved.
- If a flagger is required, they must inform all workers how they will be warned of approaching trains.
- Complete safety and security awareness training through e-railsafe or designated vendor.

Remote Work Area

- Establish frequent check-ins and meetings to maintain clear communication among team members.
  - Project team members must regularly touch base with staff members performing remote work to ensure they are safe.
  - When working in remote areas, cell phone reception may be limited.
  - Ensure you have designed communication systems that are adequate for the working environment.
- Encourage the use of instant messaging and email for quick updates.
- Establish adequate emergency response processes, including who to call, how to call, and directions to your location.

Repetitive Motion

- Trade-off job duties to minimize repetitive stress of joints or muscles.
- Encourage job rotation for repetitive or monotonous work in the field.
- Take breaks at the intervals defined during the task risk assessment to prevent fatigue and repetitive motion injuries.
- Use proper lifting and hand positioning to avoid sprains and repetitive motion injury.
- Prior to work, stretch legs, arms, and back.

Rotating Equipment

- Personnel should exercise caution when working near rotating equipment to prevent entanglement with clothing, placing body parts near pinch points on equipment, or using equipment on slopes or unstable surfaces.
- Site personnel and visitors who are not performing necessary work shall remain a distance of at least 15 feet away from moving parts on such equipment.

Rough Terrain

- Wear proper footwear with tread patterns designed for rough terrain.
- Before stepping, lightly tap the ground with your foot to assess its stability.
- Reduce your walking speed to allow for better balance and reaction time.
- Keep your feet slightly apart to increase your base of support.

- Actively scan the path ahead for obstacles, loose rocks, or uneven surfaces.
- Carry lightly: Avoid carrying heavy loads that could affect your balance.
- Take extra caution in wet, icy, or snowy conditions.

Sharp Objects

- Staff members are required to wear ANSI cut score A2 gloves whenever there is potential for cutting hazards on site.

Silica Dust

- Use of wet methods must be utilized to minimize dust production.
- Ensure that enough water is supplied.
- A water-integrated delivery system that supplies water to cutting surface will be a requirement for stationary mason saws, hand-held power saws, walk behind saws, drivable saws, rig mounted core saw/drills, jackhammers, and hand-held grinders.
- All water must be collected for sampling and disposal off-site.
- All drain inlets will need to be identified and secured with plastic sheeting and absorbent snakes or sandbags, and will need to be identified by the use of a cone or delineator.
- Personnel must ensure drain valves are safely secured, and washed soils will be shoveled from the unit and will be transported into appropriate labeled drums.
- Evaluation must be completed to determine whether a dusk mask/respirator is required to prevent inhalation of particles (based on equipment used – see below).
- The slurry from utilizing the wet methods must be removed before it dries to prevent the dried material from generating dust that can spread to other areas of the site.

Simultaneous Operations (SIMOPS)

- SIMOPS are described as the potential class of activities which could bring about an undesired event or set of circumstances (e.g., safety, environment, damage to assets, schedule, commercial, financial, etc.).
- SIMOPS are defined as performing two or more operations concurrently.
- SIMOPS should be identified at an early stage before operations commence to understand issues such as schedule and physical clashes, maintenance activities, failure impacts, interferences between vessels, contracts and third part interfaces and environmental impacts.
- Coordinate project with site activities.
- Identify and understand the hazards associated with the host and client's activities.
- Integrate site emergency response protocols, where appropriate, and communicate to all project staff.
- Integrate site communication protocols and communicate to all project staff.

Site Control

- Work Zones
  - To prevent both exposure of unprotected personnel and migration of contaminated materials due to tracking by personnel or equipment, work areas along with PPE requirements will be clearly identified.
  - Haley & Aldrich designates work areas or zones in accordance with the "Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities" (NIOSH/OSHA/USCG/EPA).
  - They recommend the areas surrounding each of the work areas to be divided into three zones:
    - Exclusion zone
    - Contamination Reduction Zone (CRZ)
    - Support Zone
- Exclusion Zone
  - The areas of site activities discussed in the mobilization section of this HASP which contain or are suspected to contain hazardous material will be considered the exclusion zone.
  - The exclusion zone for soil excavation will be a radius of 20 feet around each area to be excavated whenever possible and where applicable.
  - The exclusion zone for drilling activities will be at least a radius equal to the height of the drill rig mast.
  - This radius may be increased if air monitoring reveals that Level C respiratory protection is required during these activities.
  - Personnel in the exclusion zone must have the required OSHA training, medical authorization, and have the appropriate PPE.
  - The locations of the exclusion zones will be determined in the field by the SSO and will be marked using caution tape, snow fence, and/or signs, where appropriate.
- Contamination Reduction Zone (CRZ)
  - The CRZ will be established upwind of the exclusion zone and will provide for personnel and equipment decontamination where necessary. The decontamination area will be determined in the field by the SSO.
- Support Zone
  - The support zone is located in an area that is not contaminated.
  - This area is established to provide areas for breaks, administration of first aid, personnel monitoring, administrative functions, and equipment storage.

Slips, Trips, and Falls

- Keep site and walking pathways clear of obstacles and debris.
- Keep work surfaces dry where possible.
- Wear appropriate footwear for the conditions. Add traction devices (ice cleats, 'Yaktrax', etc.) to footwear as needed.

- Take your time, stay alert, and be aware of the conditions.
- In mixed vegetation, rubble or debris, or walking over snow/ice use a walking stick/staff to 'investigate the ground' of your path.

Steep Slopes

- All machines have limits for the slopes on which they can safely operate.
- Machines must not be operated on slopes outside of the manufacturer's recommendations.
- If excavations are on a steep slope, evaluate operations to ensure compliance with excavation/ trenching regulations.
- Work should be evaluated for fall protection, and any heavy equipment should be evaluated to see if appropriate for the work.
- When planning how the work should be done on a specific site, consider:
  - Soil/ground conditions,
  - Ground roughness,
  - Underlying material,
  - Machine design/limits,
  - Terrain-slope measurements,
  - Visibility,
  - Weather conditions,
  - Water on site/sinking/floods.
- When working on a steep slope, additional care should be taken to prevent slips, trips, or falls.

Struck-By

- Ensure operating paths are clear of personnel or objects subject to hazard.
- Operate equipment safely and according to manufacturer's instructions.
- No one will approach equipment in operation unless the operator gives them positive indication that it is acceptable to do so.
- Put up barriers to avoid unnecessary contact with hazards.
- Ensure egress paths are clear and unobstructed.
- Vessel operator will clearly notify anyone boarding the vessel of any pinch point hazards associated with the operation of the vessel and sample equipment.

Underground Utilities

Pre-planning is required for understanding the limitations of the site and identifying whether subsurface intrusive work can be completed safely. The following items, which are provided in OP1020 - Underground Utilities may

need to be completed or reviewed prior to intrusive work based on the type of work as well as local, state, federal, and client requirements:

- Dig-In Prevention Policy;
- A health and safety plan and related health and safety documents/considerations;
- OP1020 Attachment C - Subsurface Clearance Field Checklist;
- Utilities marked with ANSI Z 535.1 Color Matrix;
- Confirm utilities with a private utility locator;
- Establish Tolerance Zones;
- A site walkthrough with staff members, subcontractors, and others who know utility locations, as applicable;
- State One Call – 811, three days prior to starting ground disturbance activities; and/or
- State One Call Ticket/Permit kept at site or readily accessible.

Welding

- Stay at least 35 feet away from the welding area, when possible, especially if not involved in the welding operation.
- If you must be in the vicinity, ensure you are shielded from the direct line of the welding arc, do not look at the welding arc, or wear appropriate eye protection.
- Flammable materials should be removed from the area or protected with fire-resistant shields or blankets.
- Unauthorized people should not enter the welding area.
- If staff will be performing welding activities, they must complete a task-specific JHA, hot work permit, and contact H&S.

Working at Heights

- Any project teams performing work that requires fall protection must work with their RSM for proper planning and training.
- Fall protection such as guardrail systems, safety net systems, or personal fall arrest systems can be used to protect staff while working at height.
- Staff required to wear fall arrest systems shall have received appropriate training and will inspect their gear prior to use to ensure it is in good working order.

Working in Phragmites Marshes

Special precautions should be taken when working in and around Phragmites marshes. These precautions include:

- Phragmites marshes pose significant slip, trip, and fall hazards. These marshes have soft and wet soil which can cause slip hazards. Terrain may be uneven, and changes in terrain may be hidden by ground cover. Additionally, features including muskrat burrows and ditches may create hazardous walking conditions. Use caution when walking through these areas.

- Dense Phragmites growth limits visibility, making slip, trip, and fall hazards such as ditches or waterway banks difficult to negotiate.
- The limited visibility can affect orientation. Site personnel entering areas of dense Phragmites growth should travel in pairs and maintain constant awareness of the way to exit the area.
- Phragmites leaves are sharp edged and cut skin. Additionally, Phragmites pose significant threat to eyes. Site personnel should wear work gloves and eye protection (spoggles) if the activity involves walking through Phragmites.
- When working in marshes and other limited visibility areas, field staff should ensure that they make another team member aware of their location. Radios or cell phones should be used to maintain communication. Have readily available telephone numbers for emergency services such as fire and police departments.
- Dry Phragmites stalks and leaves can catch fire. See section on Brush Fires in Appendix C for more information.

Workplace Harassment

- Staff members that encounter any sort of harassment during work should first try to deescalate the situation, if possible, and remove themselves from any threat as soon as possible;
- Do not attempt to engage or antagonize a person threatening violence;
- Report any threats, physical or verbal, and/or any disruptive behavior of any individual to HP and H&S as soon as possible either through phone call, email, or the use of the Sensitive Information Reporting Form; and
- Following an incident, staff may be asked to participate in any subsequent investigation of the workplace harassment incident.

APPENDIX C – WEATHER-RELATED HAZARDS

Brush Fires

Fires are a concern due to the large amount of vegetative material. Dry weather and strong winds contribute to the threat of fires. The following precautions should be taken during these periods:

- Prior to field activity, personnel should review all available weather information for warnings of increased fire potential.
- For extended work activities in the wetlands, a minimum of two evacuation routes should be established and cleared. The evacuation routes must be identified to field personnel working in the wetlands.
- A fire extinguisher will be carried into the wetlands.
- One field team member must return to the entry point to check the surroundings for signs of brushfire (smoke, etc.) and changing wind conditions on intervals not exceeding twenty minutes.
- Navigation tools, such as a map and compass, and two separate means of communication (cell phones, two-way radios, etc.) will be required safety equipment and must be immediately accessible to all members of each field team during all activities.
- Work involving the use of a potential ignition source (e.g. generators, halogen lighting) will not be performed.
- No smoking is allowed in wetland areas.
- In the event that warning signs of a brush fire are noticed by any field team member (fire sighting, smoke smell or sight, warning from elsewhere on site), all work will be suspended, and field teams will evacuate the site immediately. After evacuation, the priority will be achieving confirmation of evacuation from all field personnel. Emergency services will be contacted as per the HASP.
- All activities will be suspended until positive confirmation can be obtained that the fire has been completely controlled and extinguished.

Cold Stress

- Make sure workers are aware of cold stress symptoms, consequences, and prevention methods.
- Dress appropriately for wet/cold weather.
- Dress in layers and wear waterproof clothing as needed and proper cold weather PPE.
- Stay inside, as practicable, and use a buddy system to monitor cold stress effects.
- Have a warm area or vehicle available for breaks from the cold.
- Schedule work at warmer times of day or during milder weather times, if possible.
- If staff members feel the effect of weather stress, seek a warm location and drink warm non-caffeinated beverages.

Earthquakes

- Remember to Drop, Cover, and Hold on.
  - Drop: Wherever you are, drop down to your hands and knees and hold onto something sturdy. If you're using a wheelchair or walker with a seat, make sure your wheels are locked and remain seated until the shaking stops.
  - Cover: Cover your head and neck with your arms. If a sturdy table or desk is nearby, crawl underneath it for shelter. If no shelter is nearby, crawl next to an interior wall (away from windows). Crawl only if you can reach better cover without going through an area with more debris. Stay on your knees or bent over to protect vital organs.
  - Hold on: If you are under a table or desk, hold on with one hand and be ready to move with it if it moves. If seated and unable to drop to the floor, bend forward, cover your head with your arms, and hold on to your neck with both hands.
- If an earthquake happens, protect yourself right away:
  - If you are in a car, pull over and stop. Set your parking brake.
  - If you are in bed, turn face down and cover your head and neck with a pillow.
  - If you are outdoors, stay outdoors away from buildings.
  - If you are inside, stay and do not run outside and avoid doorways.
- There can be serious hazards after an earthquake, such as damage to the building, leaking gas and water lines, or downed power lines.
  - Expect aftershocks to follow the main shock of an earthquake. Be ready to Drop, Cover, and Hold On if you feel an aftershock.
  - If you are in a damaged building, go outside and quickly move away from the building. Do not enter damaged buildings.
  - **If you are trapped, send a text or bang on a pipe or wall.** Cover your mouth with your shirt for protection and instead of shouting, use a whistle.
  - If you are in an area that may experience tsunamis, go inland or to higher ground immediately after the shaking stops. Avoid contact with floodwaters as they can contain chemicals, sewage, and debris.

Fires

- Small
  - Use fire extinguisher if safe and qualified to do so.
  - Notify PM, Site Safety Officer, contact 911.
- Large
  - Evacuate immediately.
  - Notify PM, Site Safety Officer, contact 911.

Flooding

- Move to higher ground.
- Avoid floodwater, don't walk, drive, or swim in flooded areas.

- Don't touch any electrical equipment.
- If heavy rains are expected and there is potential for flooding, consider rescheduling work to avoid unsafe areas.
- Evacuate when instructed to do so.

Heat Stress

- Make sure workers are aware of heat stress symptoms, consequences, and prevention methods.
- Drink plenty of hydrating fluids (i.e., water), provide enough water for each staff member to drink one quart per hour for days when heat exceeds 80 degrees F using the Heat Index.
- Use cooling devices, as needed.
- Acclimatize workers before performing work in extreme heat.
- Have a shaded area or vehicle available for breaks from the heat.
- Use a buddy system to monitor heat stress effects.
- Schedule work at cooler times of day or during milder weather times, if possible.
- Monitor the Heat Index using the OSHA NIOSH Heat Safety Tool mobile app, complete the Heat Injury and Illness Prevention Plan when heat exceeds 80 degrees F using the Heat Index, and consult Health and Safety.

High Winds

- The table below summarizes the stop work action levels for general site work and various high-risk project activities with respect to wind speed.

Work Activity	Wind Speed Stop Work Action Levels	
	Sustained[1]	Instantaneous
General Site Work	25 mph	40 mph or greater
High Risk Activities		
Working at Heights	15 mph	25 mph or greater
Critical Lifting Operations	15 mph	25 mph or greater
Lifting Operations	25 mph	35 mph or greater
Over Water Work	25 mph	35 mph or greater
Heavy Equipment with Elevated Masts	25 mph	35 mph or greater

- It should be noted that when using specific heavy equipment, that the manufacturer's recommended wind speed for cease of operations (if defined) supersedes the wind speed action levels stated above, if the manufacturer's guidelines are more restrictive.
- Task specific JHAs must reflect manufacturer's specific guidelines, or if exact make and model of equipment is not known during initial JHA development, they will be updated in the field to reflect requirements.

Hurricanes

- Pay attention to emergency information and alerts.
- If you live in a mandatory evacuation zone and local officials tell you to evacuate, do so immediately.

- Determine how best to protect yourself from high winds and flooding.
- Take refuge in a designated storm shelter or an interior room for high winds.
- Go to the highest level of the building if you are trapped by flooding. Do not climb into a closed attic. You may become trapped by rising flood water.
- Do not walk, swim, or drive through flood waters. Turn Around. Don't Drown! Just 6 inches of fast-moving water can knock you down, and 1 foot of moving water can sweep your vehicle away.

Lightning Storms

- If lightning is observed to be within 6 miles of the project, suspend operations, depending on the speed of storm approach.
- You can use "Spark" by WeatherBug, or another recognized lightning warning app to track lightning.
- National Severe Storms Laboratory (NSSL) recommends the "Flash to Bang" method. Simply count the seconds from the time the lightning is sighted to when the clap of thunder is heard.
- Divide the number by five to obtain how far away in miles the lightning is occurring.
  - For example, if the lightning flash is seen and then 15 seconds later the bang of thunder is heard, the lightning is 3 miles away. It is important site personnel monitor not only how far away the lightning is, but also how fast it is approaching.
- The NSSL recommends by the time the Flash to Bang count reaches 30 seconds, all individuals should have left the site and reached a safe structure, such as a building or job trailer.
- If caught in the open by an electrical storm, immediately seek shelter in a vehicle on land.
- If a vehicle is inaccessible: move to a topographically low area away from tall objects and conductors (e.g., trees, transformers, fences, pipelines, power lines, metal sheds) and wait for the storm to leave the area.
- If you feel your hair stand on end (an indicator lightning is about to strike), drop to your knees and bend forward, putting your hands on your knees. Do not lie flat on the ground (be wary of seeking shelter in washes, ravines, or gullies during heavy downpours because of the risk of flash floods).
- In the event of extreme weather conditions which may pose a health and/or safety risk to workers, field activities will cease until the SSO determines conditions are safe to resume operations.
- Wait at least 30 minutes after the last lightning strike within 6 miles before resuming work.

Snowstorms

- Assess the weather before traveling and ensure emergency supplies are packed in the vehicle.
- Provide sufficient time for travel delays due to snow/ice covered roads or accidents.
- Inform someone of your travel plans and expected time of arrival.
- Wear layered clothing, including jackets, hats, and mittens, or keep these items in the vehicle.
- If you get stuck inside your vehicle:
  - Stay inside the vehicle.
  - Run the heater periodically but be cautious of carbon monoxide poisoning.

Sun Exposure

- Exposure to ultraviolet light from working outdoors can cause sunburn and can lead to skin cancer. Staff members are encouraged to liberally apply sunscreen, with a minimum sun protection factor of SPF 30 when working outdoors. Sunscreen selected should protect against both UVA and UVB rays. Staff members should work in the shade whenever practical.
- Prevention of over-exposure and sunburn will be completed by wearing appropriate clothing and using sunscreen. Long-sleeve breathable shirts are recommended to avoid sun exposure to the arms. All staff members are required to wear long pants and boots as part of their PPE and will eliminate sun exposure to legs and feet.
- Additionally, staff wear safety glasses offering protection from ultraviolet A/ultraviolet B rays.

Tornadoes

- Watch areas where tornadoes could become present.
- Review and discuss your emergency plans, communication/sirens in case of emergency and review shelter locations.
- If site is prone to tornadoes, monitor weather reports during work shifts.
- Act quickly if a warning is issued or you suspect a tornado is approaching.
- If a tornado has been sighted or indicated by weather radar, imminent danger to life and property exists.
- Go to shelter locations listed in Safety Plan. Go immediately under ground to a basement, storm cellar or an interior room (closet, hallway, or bathroom).
- If caught outdoors when a tornado is threatening, seek shelter in a basement or a sturdy building. If one is not within walking distance, try to drive to the nearest shelter.
- If flying debris is encountered while in a vehicle, there are two options:
  - Stay in the vehicle with the seat belt on, keep your head below the windows, and cover it with your hands or a blanket, or
  - If there is an area that is noticeably lower than the roadway, lie in that area and cover your head with your hands.

Tsunami

- If you are under a tsunami warning:
  - Know evacuation routes (often are marked by a wave with an arrow in the direction of higher ground) and follow them to higher ground.
  - Contact your project manager when it is safe to do so to inform them that you have evacuated.
  - Leave immediately if you are told to do so. Evacuation routes often are marked by a wave with an arrow in the direction of higher ground.
  - If you are in the water, grab onto something that floats, such as a raft or tree trunk.
  - If you are in a boat, face the direction of the waves and head out to sea. If you are in a harbor, go inland.

Wildfires and Air Quality Concerns

- Monitor and assess the air quality index (AQI) for particulate matter at 2.5 microns (PM 2.5) utilizing AirNow.
- Utilize engineering or administrative controls to limit staff member exposure to an AQI level of 150 or lower if possible.
- Review our Wildfire Smoke and Air Quality program materials.
- Ensure all staff members have completed Air Quality Training.

AQI for PM 2.5	Response Action
0 to 50	Normal working conditions. Monitor PM 2.5 conditions and forecasts.
51 to 100	Normal working conditions. Sensitive groups should begin to monitor physical condition. Notify staff of air quality conditions. Ensure only trained staff members are working in the field.
101 to 150	NIOSH approved N95 respirators will be available in all offices for staff members that have completed the voluntary use form. Engineering controls and administrative controls will be implemented. Sensitive groups should continue to monitor conditions.
151 to 200	NIOSH approved N95 respirators will remain available in all offices for staff members that have completed the voluntary use form. Monitor physical conditions throughout the day and remain aware of immediate health impacts of smoke inhalation. PMs should begin discussions about rescheduling work. Recommended that outdoor staff take frequent breaks in areas where filtered AQI is less than 101.
201 to 250	Respirators will remain available for all staff if voluntary use form is completed. All outdoor staff are required to take frequent breaks in areas where filtered AQI is less than 101. Sensitive persons are not to work outdoors in these conditions.
251+	All outdoor projects will be stopped in the impacted areas until the AQI has dropped below 251. Outdoor project work will be rescheduled.

APPENDIX D – BIOLOGICAL HAZARDS

Bacteria

- Wear appropriate personal protective equipment (PPE), such as gloves, eye protection (goggles or face shields), and disposable clothing, when working with potentially contaminated materials or in areas where bacterial exposure is possible.
- Wash your hands and any exposed skin with soap and water immediately, or as soon as feasible, after contact with potentially infectious materials or contaminated surfaces.
- Disinfect potentially contaminated surfaces to reduce potential for bacteria to spread.

Bees, Wasps, and Hornets

- Bees generally fly in straight lines between flower and hive, hence collision with unsuspecting individuals occurs.
- If a single bee approaches, STAY STILL, do not try to swat the insect as this may cause it to react.
- If it lands, gently try to blow it off the skin.
- If a swarm of bees approach, run for shelter. Bees release a chemical when they sting, which may attract other bees to sting.
- If stung, try to look for the stinger and carefully remove it by flicking it or scratching it out of the skin from the stinger sack.
- Stings to the head and neck are more dangerous.
- Life threatening reactions are more likely to occur in people who are already known to be very allergic to bee venom, older people with pre-existing heart and chest complaints, or with multiple stings.
- When stung immediately apply ice or cold compresses to the sting site.
- Wasps and bees are drawn to flower fragrances and clothing with bright colors (white is safest), perfumes, fruit juices and eating fruit outdoors, hair tonics, suntan lotions, and floral odors.
- Hornets are drawn to food and sources of moisture.
- Carefully shake out any clothing left on the ground.
- Cover open containers and any food outdoors.
- Individuals who are prone to severe reactions to bee stings should notify the SSO and carry their prescribed medication(s) with them for self-administration.
- Emergency medical treatment should be sought immediately for individuals who are allergic to bee stings or other individuals who exhibit severe reactions described above.

Bird and/or Bat Droppings

- Avoid situations where bird or bat droppings may exist.
- Use dust suppression methods to reduce airborne particulate levels.
- Wear respiratory protection and PPE to curtail particulate adhesion to clothing.

Bloodborne Pathogens (BBPs)

- While Haley & Aldrich staff members should not anticipate occupational exposure to bloodborne pathogens in their normal daily activities, there is always potential for an incident that could lead to exposure.
- Bloodborne pathogens are bacteria, viruses, and other pathogenic microorganisms that can only be detected by medical tests.
- Any time you come in contact with another individual's blood or other potentially infectious material (OPIM), you are at risk of becoming infected.
- Therefore, staff members must treat all blood and OPIM as if they could get infected.
- Offices are equipped with BBP response kits, and field staff members have the option to collect the same BBP kits to have on hand as a resource.
- Proper PPE, disposal, and handwashing are critical to limit exposure to BBP.
- For more information, see OP1058 - Bloodborne Pathogens.

Contaminated water

- Don PPE to reduce exposure, such as gloves, safety glasses, and protective clothing.
- Implement work practices to limit contact with contaminated water, such as avoiding walking through contaminated areas and using remote handling techniques, where feasible.
- Use signage that clearly indicates the presence of contaminated water and warns against contact or use.
- Identify handwashing facilities or areas where staff members can thoroughly wash hands following any potential exposure.

Disease

- The primary routes of infectious disease transmission are contact, droplet, and airborne.
- General practices, such as the following, can help reduce the risk of disease:
  - Regular handwashing
  - Covering coughs and sneezes
  - Proper cleaning and disinfection of surfaces
  - Staying home when sick
  - Wearing face coverings
  - Wearing gloves

Mammals - Large

- Avoid contact with animals whenever possible.
- If an animal displays aggressive behavior and charges, do not run or turn your back.
- When confronted by a large mammal, it is important not to run or exhibit any behavior that may be construed as a challenge (e.g., looking the animal in the eyes, showing your teeth, etc.).

- Stand still and place your work bag between you and the animal and then begin to move slowly away from the animal, while not turning your back.

Mammals - Small

- Avoid contact with rodents, if possible.
- Avoid contact with rodent excrement.
- Do not eat food or water that may have encountered rodent excrement.
- If exposed, wash hands and avoid touching your face with your hands.

Mosquitoes

- Take appropriate precautions to minimize the potential transmission of viruses carried by mosquitoes.
- Use of PPE techniques is essential to prevent mosquito bites, especially when working at sites where mosquitoes may be active and biting.
- Use repellents containing DEET, picaridin, IR3535, and some oil of lemon eucalyptus and para-menthane-diol products provide longer-lasting protection. To optimize safety and effectiveness, repellents should be used according to the label instructions.
- Cover as much of your skin as possible by wearing shirts with long sleeves, long pants, and socks whenever possible.
- Avoid use of perfumes and colognes when working outdoors during peak times when mosquitoes may be active; mosquitoes may be more attracted to individuals wearing perfumes and colognes.
- Treat bites by cleaning the affected area with warm soapy water.
- Place an ice pack on the affected area to reduce swelling.
- Take or apply an antihistamine to reduce itchiness.
- Avoid scratching the bite to reduce risk of infection.

Marine Animals

- Maintain a minimum 50-yard distance from marine mammals.
- Marine mammals can be aggressive if bothered, and many have environmental protections.
- Do not approach marine mammals and should maintain their distance.
- If marine mammals are in the work area and impede work, the team should contact the harbormaster and project management.

Poisonous Plants

Poisonous plants that can cause skin irritation include poison ivy, poison oak, and poison sumac. Poisonous plants are found throughout the United States (except Alaska and Hawaii).

<u>Poison Ivy</u>

Eastern poison ivy is typically a hairy, ropelike vine with three shiny green leaves budding from one small stem. The leaves may be red in the fall.

Western poison ivy is typically a low shrub with three leaves that does not form a climbing vine. It may have yellow or green flowers and white to green-yellow or amber berries.

Poison Oak

Poison oak is typically a shrub with three leaves, like poison ivy. Pacific poison oak may be vine-like. It may have yellow or green flowers and clusters of green-yellow or white berries.

Poison Sumac

Poison sumac is a woody shrub that has stems with 7 to 13 leaves arranged in pairs. It may have glossy, pale yellow, or cream-colored berries

Types of Exposure

Poison ivy, poison oak, and poison sumac release an oil (urushiol) when part of the plant is damaged or burned. When the oil gets on the skin, most exposed people have an allergic reaction (contact dermatitis). Workers can become exposed to urushiol through:

- Direct contact with the plant.
- Indirect contact, such as touching tools, livestock, or clothing that have urushiol on them.
- Inhaling particles containing urushiol from burning plants.

Prevention

- Wear long sleeves, long pants, boots, and gloves. Wash exposed clothing separately in hot water with detergent.
- Use barrier skin creams, such as Ivy X pre contact towelettes.
- Clean tools with rubbing alcohol (isopropanol or isopropyl alcohol) or soap and lots of water. Urushiol can remain active on the surface of objects for up to five years. Wear disposable gloves while cleaning.

Symptoms of contact with poisonous plants include:

- Red rash within a few days of skin contact
- Bumps, red patches or streaking, or weeping blisters
- Note: fluids in blisters will not cause blisters to spread on you or others
- Swelling
- Itching

First Aid

- Rinse skin immediately.
- Use rubbing alcohol, poison plant washes (Ivy X Contact wipes), or dishwashing soap, and lots of water.
- Rinse often to prevent wash solutions from drying on the skin and further spreading the urushiol.
- Apply cold compresses, follow directions on treatment wipes, do not apply to broken skin
- Emergency medical treatment should be sought immediately for individuals who are allergic to poisonous plants or other individuals who exhibit severe reactions described above.

Snakes

The degree of toxicity resulting from snakebites depends on the potency of the venom, the amount of venom injected, and the size of the person bitten. Poisoning may occur from injection or absorption of venom through cuts or scratches.

The most effective way to prevent snakebites is to avoid snakes in the first place. Personnel should avoid walking at night or in high grass and underbrush. Visual inspection of work areas should be performed prior to activities taking place.

The use of leather boots and long pants will be required, since more than half of all bites are on the lower part of the leg. No attempts at killing snakes should be made; many people are bitten in such an attempt.

If someone is bitten by a potentially poisonous snake, the following treatment should be initiated:

- Keep patient calm.
- Notify emergency medical services.
- Wash the wound and keep the affected body part still.
- Apply direct pressure to site of bite if bleeding is extreme.
- Keep the affected area lower than the heart.
- Carry a victim who must be transported or have them walk slowly.
- Transport to the closest medical facility.

Ticks

- Wear light-colored clothing so ticks stand out, and long-sleeved shirts and long pants to reduce skin exposure.
- Tuck your shirt into your pants and tuck your pants into your socks to close gaps.
- Use repellent containing 20-30% DEET (N, N-diethyl-m-toluamide) on exposed skin and clothing.
- Avoid hands, eyes, and mouth and wash off repellent when back indoors.
- Treat clothing with or purchase clothing with products containing 0.5% permethrin.
- Conduct frequent tick checks on clothing and skin. Have others check your back, scalp, and behind your ears and check gear for "hitchhikers."
- As soon as you return indoors, take a bath or shower. and do a full-body inspection using a mirror.
- Wash field clothes and tumble dry on high to kill any ticks that may be hidden.
- If working in an area of significant tick habitat, PPE may need to be upgraded to a Tyvek suit.
- Implementation of controls is crucial to minimize or eliminate the possibility of a tick bite.
- If a staff member has been bitten contact Corporate H&S and Work Care at 888-449-7787 to initiate the Tick Management Protocol. Once bitten, it takes approximately 48 hours to transmit Lyme Disease,
- For removal, a fine-tipped tweezer is recommended as a tick removal tool and should be in the first-aid kit. Follow these steps:
  - Pull upward with steady, even pressure.
  - Do not twist or jerk the tick; this can cause mouth parts to break off and remain in the skin.
  - If this happens, remove the parts with tweezers.
  - If unable to remove easily with tweezers, leave them alone and let the skin heal.
  - After removing the tick, thoroughly clean the bite area and hands with rubbing alcohol, iodine scrub, or soap & water.
  - Dispose of live ticks by submersal in alcohol, placing it in a sealed bag/container, wrap it tightly in tape, or flush it down the toilet.

- Never crush ticks with your fingers.
- Do not attempt to use nail polish remover, petroleum jelly, lotion, or heat to try to get the tick to exit skin.
- Swift removal is key.

Venomous Spiders

- Inspect or shake out any clothing, shoes, towels, or equipment before use.
- Wear protective clothing, such as a long-sleeved shirt and long pants, hat, gloves, and boots when handling stacked or undisturbed piles of materials.
- Minimize the empty spaces between stacked materials.
- Remove and reduce debris and rubble from around the outdoor work areas.
- Trim or eliminate tall grasses from outdoor work areas.
- Store apparel and outdoor equipment in tightly closed bags.
- Keep tetanus boosters up to date (every 10 years).
- Spider bites can become infected with tetanus spores.
- Additional information in the case of bites can be obtained from the Poison Center (1-800-222-1222).

Wildlife Droppings

- Do not touch droppings with unprotected hands.
- Avoid disturbing the droppings and generating dust.
- Staff member work practices and dust control measures that eliminate or reduce dust generation during removal of manure from a building will also reduce risks of infection and development of disease.
- Use an industrial vacuum cleaner with a high-efficiency (HEPA) filter to bag contaminated material.

APPENDIX E – EMERGENCY RECOGNITION AND PREVENTION

Emergency Type	Notification	Response Action
Chemical Exposure	Report event to PM immediately	Refer to Safety Data Sheet for required actions
Fire- Small	Contact 911 and notify PM when safe to do so	Use fire extinguisher if safe and qualified to do so
Fire - Large/Explosion	Contact 911 and notify PM when safe to do so	Evacuate Immediately
Medical - Bloodborne Pathogen	Report using H&A's reporting tool	If qualified and trained dispose in container or call client/responsible party for further instruction
Medical - First Aid	Call Acuity (1-888-397-8099) and notify PM when safe to do so	If qualified, perform first aid duties. Contact Acuity for future management.
Medical - Trauma	Call 911 immediately, and notify PM when safe to do so	Wait at site entrance for ambulance.
Security Threat	Notify PM, who will call 911 as warranted	Hide all valuables and delineate all work zones. Double check and secure access points to site.
Weather - Earthquake/ Tsunami's	Stop Work, evacuate the site if necessary, and notify PM	Turn off all equipment and evacuate as soon as it is safe to do so.
Weather - Lightning Storm	Stop Work and seek shelter, and notify PM	Work may resume 30 minutes after the last observed lightning.
Weather - Tornadoes/ Hurricanes	Monitor weather conditions, Stop Work, seek appropriate shelter, and notify PM when safe to do so	Evacuate to shelter location or shelter in place immediately.
Weather - Fog	Monitor Weather conditions	Pause work until fog dissipates and site lines can be re-established.
Weather - Wind	Notify PM and all equipment operators	Monitor conditions and be prepared to Stop Work.
Weather - Floods	Notify PM and monitor weather conditions	Immediately Stop Work and evacuate.
Hazardous Materials Spill - Small	Contain and clean the spill if it is safe to do so, and notify the PM	Properly trained staff can clean up the spill.
Hazardous Material Spill - Large	Stop Work, secure the area, and notify the PM when safe to do so	Spill will be reported to the correct agency, and cleanup will be at their discretion

APPENDIX E
Community Air Monitoring Plan

COMMUNITY AIR MONITORING PLAN (CAMP)
FORMER MONARCH CHEMICALS FACILITY
61 GATES AVENUE
GENEVA (ONTARIO COUNTY), NEW YORK 14456
VCA NO.V00119-8

Prepared For:

H.B. Fuller Company
1200 Willow Lake Blvd
PO Box 64683
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Pinnacle Project No:

R013023.000

Date:

August 22, 2017

Pinnacle Engineering

Pinnacle Engineering, Inc.
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COMMUNITY AIR MONITORING PLAN

FORMER MONARCH CHEMICALS FACILITY
61 GATES AVENUE, GENEVA, NEW YORK 14456
VCA NO.V00119-8

1.0 INTRODUCTION

1.1 BACKGROUND

During potential soil excavation or development activities at the Former Monarch Chemical Facility located at 61 Gates Avenue in Geneva, NY including during soil excavation and removal, air and dust emissions will be monitored and controlled to protect the surrounding environment from exposure to potential airborne contaminants. This *Community Air Monitoring Plan* (CAMP) is an integral part of the Excavation Workplan (EWP – Appendix C), a component of the Site Management Plan (SMP). This CAMP is intended to provide a measure of protection for on-Site and downwind community (i.e., off-Site receptors including residences and businesses, and on-Site workers not directly involved with the subject excavation or development activities) from potential airborne contaminant releases as a direct result of the intrusive work activities in this area.

This CAMP has been developed to address particulates (dust) and potential subsurface volatile organic compounds (VOCs), existing as soil vapors, that may be generated or released to air during implementation of soil excavation or intrusive development activities. This CAMP was prepared in accordance with New York State Department of Health (NYSDOH) requirements presented in *Appendix 1A* of the *DER-10 Technical Guidance for Site Investigation and Remediation* (NYSDEC – May 3, 2010). The CAMP requires real-time monitoring for dust and organic vapors at the downwind perimeter of the designated work area for the benefit of downwind adjoining properties that may contain sensitive receptors (e.g., Gates Avenue, Lehigh Street, and the open space and residential area east of the Finger Lakes Railroad rail line). There is one (1) potential Work Area that exceeds the Unrestricted Soil Use standard for volatile organic compounds (VOCs), in particular Tetrachloroethene (PCE). Measures included in this CAMP provide a level of protection for the occupants of the neighborhood properties and residences, as well as the downwind community, from potential airborne releases. This CAMP sets forth specific action levels for determining monitoring frequency and the appropriate corrective actions, including work shutdown.

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1.2 PURPOSE AND OBJECTIVES

The principal purpose of this CAMP is to monitor air quality at the Site where soil may be disturbed or loaded during soil excavation or development activities where remaining contamination may be encountered. This CAMP describes monitoring of dust and vapors on a real-time basis. Air monitoring will involve standard monitoring functions for environmental projects including real-time air monitoring for particulate matter less than ten (10) micrometers in size (PM-10), and VOCs. These monitoring and observations of visible dust emissions and detected odors include routine inspection and monitoring of contractor's work practices, and include the reporting the results to NYSDEC and the New York State Department of Health (NYSDOH).

Monitoring will be performed during all ground intrusive activities. Ground intrusive activities include, but are not limited to, soil excavation and handling. Reliance on this CAMP should not preclude simple, common-sense measures to keep VOCs, dust, and odors at a minimum around the work areas.

Principal objectives of this CAMP are as follows:

- Monitor dust as PM-10 in real-time, such that dust associated with the soil excavation or development activities are maintained below action levels;
- Monitor organic vapors as VOCs in real-time, such that potential vapors associated with the soil excavation or development activities are maintained below action levels;
- Monitor odors and dust emissions (based on olfactory and visible evidence) so that vapors and dust from work areas do not leave the Site; and,
- In the event that PM-10 or VOC concentrations exceed action levels, Site personnel will be immediately notified so that necessary corrective actions can be taken.

2.0 AIR MONITORING PROGRAM

The perimeter air-monitoring plan is a stand-alone document and will be available on-Site. The VOC Monitoring, Response Levels, and Actions are presented as follows.

Air Monitoring Response Levels and Actions	
VOCs	
Response Level	**Actions**
>5 ppm above background for 15-minute average	• Temporarily halt work activities • Continue monitoring • If VOC levels decrease (per instantaneous readings) below 5 ppm over background, work activities resume
Persistent levels >5 ppm over background but <25 ppm	• Halt work activities • Identify source of vapors • Corrective action to abate emissions • Continue monitoring • Resume work activities if VOC levels 200 feet downwind of the exclusion zone, or half the distance to the nearest residential/commercial structure, whichever is less, is <5 ppm for a 15-minute average • If VOC levels are >25 ppm at the perimeter of the work area, activities must be shutdown
Particulates	
Response Level	**Actions**
>100 ug/m^3 above background for 15-minute average or visual dust observed leaving the Work area	• Apply dust suppression • Continue monitoring • Continue work if downwind PM-10 particulate levels are <150 ug/m^3 above upwind levels and no visual dust the Work area
>150 ug/m^3 above background for 15-minute average	• Stop work • Re-evaluate activities • Continue monitoring • Continue work if dust suppression measures and other controls are successful in reducing downwind PM-10 particulate levels to <150 ug/m^3 above upwind levels, and no visual dust leaves the Work area

During excavating and material handling operations, air in Work areas will be monitored continuously for the presence of VOCs and particulates. A portable photoionization detector (PID) equipped with a datalogger will be utilized to record levels of VOCs in ambient air. The PID will be equipped with an audible alarm to indicate exceedance of the action level. The instrument will be calibrated in accordance with the manufacturer's operating instructions on a daily basis and documented in a dedicated field logbook.

A Mini RAM™ PM-10 (or equivalent) particle detector will be used to continuously monitor inhalable particles (0.1-10 micrometer range) of dust during fieldwork. The particulate monitors will consist of, at a minimum, of the following functions:

- Particulate monitoring will be performed using real-time particulate monitors for particulate matter less than ten (10) microns (PM10) with the following minimum performance standards:
- Objects to be measured: Dust, mists or aerosols;
- Measurement Ranges: 0.001 to 400 mg/m^3 (1 to 400,000 ug/m^3);
- Precision (2-sigma) at constant temperature: +/-10 g/m^3 for one (1) second averaging; and +/-1.5 g/m^3 for sixty (60) second averaging;
- Accuracy: +/-5% of reading +/- precision (Referred to gravimetric calibration with SAE fine test dust (mmd= 2 to 3 m, g= 2.5, as aerosolized);
- Resolution: 0.1% of reading or 1 g/m^3, whichever is larger;
- Particle Size Range of Maximum Response: 0.1-10;
- Total Number of Data Points in Memory: 10,000;
- Logged Data: Each data point with average concentration, time/date and data point number;
- Run Summary: overall average, maximum concentrations, time/date of maximum, total number of logged points, start time/date, total elapsed time (run duration), STEL concentration and time/date occurrence, averaging (logging) period, calibration factor, and tag number;
- Alarm Averaging Time (user selectable): real-time (1-60 seconds) or STEL (15-minutes), alarms required;
- Operating Time: 48-hours (fully charged NiCd battery); continuously with charger;
- Operating Temperature: -10 to 50^o C (14 to 122^o F); and,

- Particulate levels will be monitored at one (1) upwind, and two (2) locations, immediately downwind of the Work area, and integrated over a period not to exceed fifteen (15) minutes.

Upwind 15-minute average background VOC levels will be subtracted from the downwind 15-minute average VOC levels to establish ambient VOC concentrations reflective of work activities at any particular point in time. Therefore, the "*background*" level is the most recent upwind 15-minute average reading.
Whenever practical, work areas should be positioned upwind of organic vapor and dust sources to reduce the potential for worker exposure. Monitoring locations will include, at a minimum, one (1) upwind location, and two (2) downwind locations. Monitoring stations will be located on the Property perimeter fence, and may be adjusted during the day for changes in wind direction.

Dust suppression techniques may include the following, or combinations of the following:

- Applying water on haul roads;
- Wetting equipment and excavation faces;
- Spraying water on buckets during excavation and dumping;
- Hauling materials in properly tarped or watertight containers;
- Restricting vehicle speeds to 10 mph;
- Covering excavated areas and material after excavation activity ceases; and,
- Reducing the excavation size and/or number of excavations.

Workers may use dust masks or respirators with the approval of the on-Site Safety Officer (SSO).

3.0 RECORDKEEPING AND REPORTING

3.1 RECORDKEEPING

The qualified Pinnacle SSO or on-Site Technician will ensure that all air monitoring data is recorded in a dedicated logbook. Documentation shall be made clear, concise, and provide the monitoring data, time of entry, location, personnel, weather conditions, and background concentrations for each monitoring station. Documentation will also include observational data that has the potential for impacting results, such as damage to instruments, site equipment problems, off-Site interferences, on-Site interferences, or weather-related interferences.

A windsock will be temporarily installed on-Site to monitor and determine wind direction throughout each Work day. Meteorological wind rose or graphic data will be available for use as a reference for assessing how wind speed and direction are typically distributed (i.e., the frequency of possible wind directions) in the general vicinity of the Site. Area weather data such as wind speed and relative humidity shall be obtained on a daily basis while work is progressing and documented in the dedicated field logbook.

Real-time data (e.g., PM-10 and VOCs) will be downloaded from the respective equipment dataloggers at the end of each work day. Fifteen-minute averages from each station and instantaneous readings, if any, used for decision purposes will be recorded.

NYSDEC and NYSDOH will be notified promptly via phone and/or eMail of any exceedance of an action level, and of the corrective actions taken in connection with the exceedance. All recorded dust and organic vapor readings will be available for State (NYSDEC and NYSDOH) personnel review.

3.2 REPORTING

Air monitoring equipment will be operated by trained and qualified personnel. Personnel who perform air monitoring functions described in this CAMP will be experienced in the use of field air monitoring equipment and equipment calibration methodologies, as well as in the air monitoring procedures described above. There will be appropriate staff (industrial hygienist or environmental scientist) for assessing the results of air monitoring and advising the Pinnacle SSO, Univar on-Site Manager, and the on-Site construction management representative of air quality considerations.

All air monitoring equipment will be calibrated on a daily basis in accordance with the manufacturer's operating instructions. A dedicated logbook for each monitoring unit will be maintained that details the serial number, date, time, calibration gas or other standard, and the name of the person performing the calibration.

A summary Report will be generated at the culmination of the soil excavation or development activities that requires the implementation of this CAMP, which will include daily summaries of data collected, and compilation of the CAMP results. A copy of this report will be retained on-Site, and submitted to NYSDEC and NYSDOH.

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4.0 REFERENCES

New York State Department of Environmental Conservation, 1989. *Technical and Administrative Guidance Memorandum - Fugitive Dust Suppression and Particulate Monitoring Program at Inactive Hazardous Waste Sites*, October 27, 1989.

Pinnacle Engineering, 2016. *Site Management Plan – Former Monarch Chemical Facility, 61 Gates Avenue, Geneva, NY*. April 2016.

APPENDIX F
Sampling and Analysis Plan

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27 April 2026
File No. 0211081-001

New York State Department of Environmental Conservation, Region 8
6274 East Avon-Lima Road
Avon, New York 1414-9516

Attention: Kathryn V. Lovell
Project Manager

Subject: Former Monarch Chemical Facility
Site Management Plan
Appendix F - Sampling and Analysis Plan

Dear Ms. Lovell:

The following Sampling and Analysis Plan (SAP) is an appendix to the Facility's Site Management Plan for the Former Monarch Chemicals located at 61 Gates Avenue, Geneva, New York. (Site). The following SAP was generated by ECOR Solutions, Inc. (ECOR) on October 30, 2007. This SAP remains applicable with the exception of Table 2 and Table 7. Attached Tables I and II hereby replace Tables 2 and 7 of the original ECOR SAP. Also included is the current sampling log form as requested in the New York State Department of Environmental Conservation's letter dated March 27, 2026.

Sincerely yours,
HALEY & ALDRICH OF NEW YORK

Santa E. McKenna, P.G.
Assistant Project Manager

Attachments:
Table I – Groundwater Monitoring Schedule
Table II - Groundwater Monitoring Locations
Groundwater Field Sampling Form

Table I
Groundwater Monitoring Schedule
Former Monarch Chemicals Facility
61 Gates Avenue
Geneva, New York

Sample Location	Bi-Annual Sampling Event	Bi-Annual Gauging Event
MW-114	X	X
MW-115	X	X
MW-115d		X
MW-116		X
MW-117	X	X
MW-118	X	X
MW-121		X
MW-122	X	X
MW-124s		X
MW-124d	X	X

Notes:
Bi-annual sampling and gauging events will be conducted between March and May.
Performance monitoring wells will be analyzed for VOCs, field parameters will be recorded.

Table II
Groundwater Monitoring Locations
Former Monarch Chemicals Facility
61 Gates Avenue
Geneva, New York

Bi-Annual[1] On-Site Groundwater Sample Locations
MW-114
MW-115
MW-117
MW-118
MW-122
MW-124d

Notes:
[1] Groundwater sampling events to be completed every two years.

GROUNDWATER FIELD SAMPLING FORM

Page of

PROJECT		**H&A FILE NO.**	
LOCATION		**PROJECT MGR.**	
CLIENT		**FIELD REP.**	
CONTRACTOR		**SAMPLING DATE**	

Sampling Data:

Well ID: _____ Well Depth: _____ ft Initial Depth To Water: _____ ft

Start time: _____ Depth To Top Of Screen: _____ ft

Finish Time: _____ Depth To Bottom Of Screen: _____ ft

Elapsed Time (24 hour)	**Depth To Water From Casing (ft)**	**Cumulative Purge Vol. (liters)** or **(gal)**	**Temp-erature (°F)** or **(°C)**	**pH**	**Conduct-ivity (us/cm)**	**Dissolved Oxygen (mg/L)**	**Turbidity (NTU)**	**ORP/eH (mv)**	**Comments**

Sample ID: _____

Sample Time: _____

Analysis: _____

ENVIRONMENTAL CONSTRUCTION OPERATIONS & REMEDIATION

Sampling and Analysis Plan
Former Monarch Chemicals Facility
61 Gates Avenue
Geneva, New York

October 30, 2007

ECOR Solutions, Inc.
3191 Copper Oaks Pl.
Woodbury, MN 55125

ECOR Solutions, Inc.

Sampling and Analysis Plan
Former Monarch Chemicals Facility
61 Gates Avenue
Geneva, New York

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Sampling and Analysis Plan
Former Monarch Chemicals Facility
61 Gates Avenue
Geneva, New York

1.0 Introduction

1.1 Purpose

This *Sampling and Analysis Plan* (SAP) presents the groundwater monitoring program and site sampling plan developed by ECOR Solutions (ECOR) for the former Monarch Chemicals Facility (Facility) located in Geneva, New York. The Facility monitoring program follows the methods and requirements set forth in this SAP, which is based on currently available data for the site. This SAP is designed to incorporate the evolution of the groundwater monitoring program, anticipated future investigation activities, operation monitoring and maintenance, as well as the flexibility to accommodate future sampling locations. Monitoring and sampling activities at the Facility have been performed since 1996.

1.2 Background

ECOR Solutions, Inc. (ECOR), on behalf of the HB Fuller Company (HB Fuller) has developed this *Enhanced Reductive Dechlorination In-Situ Bioremediation Interim Measures Workplan* (Workplan) to implement an Interim Remedial Measure (IRM) for an in-situ enhanced reductive dechlorination (ERD) bioremediation program at the Former Monarch Chemicals Facility (Site) located at 61 Gates Avenue in the city of Geneva, Ontario County, New York (Figure 1). The purpose of this Workplan is to provide the New York State Department of Environmental Conservation (NYSDEC) with a summary of the data supporting the extent of the proposed IRM, a chronological description of the anticipated IRM activities to be completed at the Site, and a schedule for performance of the IRM activities, including the submission of a final report.

Groundwater beneath portions of the Facility has been found to contain volatile organic compounds (VOCs) exceeding the NYSDEC groundwater cleanup. Several phased subsurface investigations have been initiated since 1996, which has lead to the installation of groundwater monitoring wells, the advancement of soil borings, surface water sampling, and the collection of soil vapor from indoor air and permanent vapor probes. The purpose of this SAP is to ensure that data of sufficient quantity and quality is collected to monitor the impacted media as well as the extent and rate of migration of groundwater impacted with the compounds of concern (COCs) related to Facility operations.

1.3 Site Location

The Site is located on a parcel of property approximately 1.75-acres in size, located at 61 Gates Avenue in the city of Geneva, Ontario County, New York (Latitude 042^{o} 52’ 54” N, Longitude 076^{o} 58’ 52” W (Figure 1). The Site is industrially developed with a manufacturing plant occupying

approximately 33,700 ft^2, with accompanying loading and unloading areas, parking lots, and an above ground storage tank (AST) containment area. The Site is typical of the gently rolling glacial topography of the Finger Lakes region, the result of repeated glaciations, the most recent 14,400 years ago during the Wisconsonian Glacial Epoch (Mullings, 1996).

Access to the Site is via Gates Avenue, which is contiguous on the southern property boundary (Figure 2). The Site is located in an industrial area of Geneva, north of Seneca Lake. Properties bordering the Site include a furniture manufacturer (CCN International) to the north, and railroad tracks (Finger Lakes Railroad) and an intermittent stream and adjacent vacant lots and residences to the east, and vacant manufacturing facilities and residential developments are located to the south and west.

The property was developed around 1910 with the construction of part of the existing manufacturing plant, with construction addition and improvements noted in the 1930s and 1950s. A boiler and circuit board manufacturer operated on the property until it was purchased in 1979 by Monarch Chemical Company (Monarch), a former operating division of the H.B. Fuller Company (HB Fuller). Monarch used the Site to manufacture food grade cleaners and sanitizers used in the dairy and related industries. In 1996, HB Fuller sold the property and business to Haltrachem. Haltrachem continues to engage in similar production activities that were employed by Monarch. Information and aerial maps indicated that in 1967, a Sanborn map indicated the site was occupied by Magnetic Components, Inc., where in the northern portion of the manufacturing plant was used for circuit board manufacturing and impregnation of capacitors and associated board components.

The Site currently consists of the 33,700 ft^2 manufacturing plant, an exterior bulk liquid powder loading and unloading areas, parking and truck turnaround areas, and a railroad spur. Currently, Haltrachem stores, mixes, and packages powdered and liquid cleaners and sanitizers on-site. The components of this operation include acids, caustics, sodium hypochloride and surfactants. Bulk liquids are delivered by tanker truck, connected to piping located outside the east portion of the plant within a concrete secondary containment area. Additional raw materials are delivered via truck in drums and totes and unloaded at a dock along the southeast corner of the manufacturing building. The mixing tanks are connected via piping to the bulk loading area along the northeast portion of the manufacturing plant.

1.4 Site Geology

The Site is underlain by several unconsolidated units of varying composition and thickness. The subsurface sediments are typical of the gently undulating glacial topography in the Finger Lakes Region of upstate New York. The near surface sediments between 0 and 4 feet bgs consist of a mixed fill (gravel to silt) and extends across the Site. This fill layer is underlain by native silt and clays ranging in thickness from 0 to 8 feet thick. A sand and sandy loam underlies this unit and ranges from 2 to 10 feet thick. Each of these units is variable in composition, and interbedded laterally with other layers, common of a deltaic depositional environment. Beneath these granular sediments is a clay and silty clay unit encountered between 9 and 14 feet bgs. This lower confining unit was not encountered at MW-116 and MW-120, or at off-site locations.

1.5 Site Hydrogeology

As of September 30, 2007, 10 (ten) groundwater monitoring wells on- and off-site are used to characterize the hydrogeology at and near the Facility. Groundwater elevation and groundwater quality data are obtained from the existing monitoring well network. Table 1 summarizes the well construction details for Facility groundwater monitoring wells. All the wells have screened intervals completed entirely in the glacial overburden sediments. Groundwater is found in the shallow unconfined sands and sandy clay units below the Site. The depth to groundwater generally follows the surface topography, typical of shallow unconfined aquifers, and at the Site the greatest depth to groundwater is on the eastern portion of the property near the drainage ditch where the hydraulic gradient steepens near the channel. Groundwater flow is somewhat radially outward from the manufacturing plant, with a general easterly flow direction across the Facility. Groundwater is encountered at depths ranging from approximately 4 feet to approximately 14 feet bgs. Figure 2 provides the location for the groundwater monitoring wells at and near the Facility.

1.6 Site Hydrology

The various site investigations established three (3) surface water sampling locations (SW-1, SW-2, SW-3) along the eastern drainage ditch. This ditch may temporarily act as a groundwater discharge point for the shallow groundwater flow system. Sampling for water quality parameters often indicates VOCs are generally detected in all surface water samples, with the highest concentrations detected at the upgradient location. The concentrations of TVOCs, and individual constituents, generally decease downstream along the Facility.

2.0 Project Organization

The project organization is designed to identify the parties involved in the project and establish the chain of authority for decision making. The project organization is as follows:

Individual	Organization	Function
Kristin Colberg	**HB Fuller**	-Senior Environmental Engineer, CHMM
Keith Rapp	**ECOR Solutions**	-Project Manager
William Anthony	**ECOR Solutions**	-Geologist, Technical Manager
Jennifer Good	**ECOR Solutions**	-Geologist, SHSC
James Craft	**NYSDEC**	-Engineering Geologist
Bartholomew Putzig	**NYSDEC**	-Project Manager\Environmental Engineer
Debbie McNaughton	**NYSDOH**	-Remedial Project Manger

The analytical laboratory and data validation organizations included in this SAP include:

Analytical Laboratories	Location
• TestAmerica	-Buffalo, New York
• Microbial Insights, Inc.	-Rockford, Tennessee
• Microseeps	-Pittsburgh, Pennsylvania
Data Validation	**Location**
• Alpha Environmental Consultants, Inc.	-Clifton Park, New York
• Environmental Standards, Inc.	-Valley Forge, Pennsylvania

TestAmerica will be the principle laboratory conducting soil, groundwater, air and soil vapor analysis under the SAP. Microbial Insights is a specialty laboratory in the bioremediation industry with molecular biology sampling and analytical services. The Microseeps laboratory conducts analytical services related to pilot testing and specialty analysis of groundwater samples.

The TestAmerica Quality Management Plan (QMP) is included as Appendix H. The Microseep Quality Systems Manual is included as Appendix J. Occasionally, due to unique sampling conditions or other considerations (laboratory volume considerations, achieving lower MDLs, forensics, conflict of interest, etc.) another analytical or data validation firm may be selected other than the contractors identified above. When this condition occurs, the substitute firm will be required to achieve the QA/QC standards outlined in this SAP.

3.0 Sampling Program

This SAP dictates how the Facility monitors and samples environmental media. This SAP has been developed to insure that all environmental data collected meets the objectives for the monitoring program and to satisfy the requirements for groundwater monitoring as set forth in New York Statutes, Chapter X - Division of Water Resources, Part 703: Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations.

The investigations conducted to date have largely determined that the primary COCs are VOCs, and this SAP is directed to these compounds, but also includes references to other compounds (SVOCs, metals) as future sampling events may include these other contaminants. The likely future sampling programs consist of site investigative activities for advancing soil borings and collecting soil samples to evaluate the nature and extent of COCs, advancing temporary and permanent groundwater monitoring wells to determine aquifer characteristics, the nature and extent of groundwater impacts, surface water quality in the drainage ditch on the eastern property boundary, air samples collected from indoor air and soil vapor probes, and corrective action activities implemented to address these affected media. Specific sampling programs and methods related to pilot testing, IRM, and final corrective action implementation which lie outside this SAP will be addressed specifically in future Workplans.

The collection and analysis of any data requires that procedures implemented assure the precision, accuracy, completeness and representativeness of the data are known and documented. This SAP presents the data collection objectives, the sampling and analysis procedures, and the Quality Assurance/Quality Control (QA/QC) activities. Specifically, the soil, groundwater, air, and surface water sampling program is designed, operated, and maintained to ensure:

- Delineation of the vertical and horizontal extent of impacts in soil, groundwater and vapor for Facility-related COCs;
- Determination that sample and analytical parameters are representative of concentrations of chemical constituents and/or indicator parameters of impacted soil, groundwater and surface water; and,
- Establishing the effectiveness of groundwater corrective action activities in terms of subsurface removal, destruction, or containment.

3.1 Groundwater Monitoring Wells

The overall objective of this SAP is to obtain sufficient and representative groundwater quality data to adequately monitor the extent and rate of migration of hazardous constituents in groundwater at the Facility. To monitor the groundwater at the Facility, samples are collected from monitoring wells, however, historically the potentiometric data has been collected from inconsistent data points (i.e., wells abandoned and re-installed, inconsistent monitoring intervals). The following issues were considered during the design of the monitoring program to ensure that the data accurately and precisely represent the groundwater conditions at the Facility.

Representativeness of Selected Analytical Parameters -- The analytical parameters selected for monitoring are based on the results of previous investigations which indicate VOCs are the principle COCs for constituents consistently detected above background levels and detection limits.

Representativeness of Selected Media -- The medium selected for sampling is appropriate to determine the pathway for constituent migration from source areas to potential off-site locations.

Number of Location of Sampling Points -- The number and location of monitoring wells selected for groundwater quality analyses is adequate to monitor the Facility.

Environmental Conditions -- Efforts will be made to minimize or reduce the effects of environmental conditions on sample collection efforts. For example, field instruments will be isolated from field contamination so that data accurately reflect sample location conditions.

Modifications to the well purge methods implemented at the Facility are based on NYSDEC requirements which advocated excessive drawdown during purging, particularly in wells that are characterized as low-flow (i.e., wells that are pumped until suction is broken), were not as effective in characterizing the groundwater conditions at the Facility. Further concerns to modify the purge methods also result from the considerable variation in hydraulic properties observed across the Facility due to the complexity of the surficial aquifer (i.e., highly variable sediments screened in the glacial sediments), and the need for greater confidence in sample quality, consistency, and aquifer representation.

Groundwater monitoring wells are divided into two categories; wells with dedicated downhole sampling systems, and those wells where downhole sampling systems have not been installed. As of September 2007, no site wells (Figure 2) have dedicated downhole equipment and this sampling outline includes the procedures for sampling wells and locations without dedicated downhole equipment. ~~The objective of low-flow rate purging and sampling is to purge at a rate with which the velocity of water entering the pump intake is imparted to the formation water and not stagnant water above the well screen. It is not based on the flow at the surface. The best indicator to insure water is being pulled from the formation is by monitoring drawdown during purging. Typical flow rates applied during this type of sampling range 0.1 to 0.3 liters/minute (L/min) (Puls and Barcelona, 1996).~~

~~Temporary or permanent downhole sampling systems will be used to sample each groundwater monitoring well, with the goal to have a permanent low-flow purging method at the Facility for the following reasons:~~

- ~~accurate, representative samples that provide consistent and reliable data;~~
- ~~greatly reduced purge volumes and associated expense with containment and treatment; and~~
- ~~reduction of costs over the life of the project.~~

3.1.1 Sampling Objectives

The objective of the groundwater monitoring program consists of a sufficient number of wells, installed at appropriate depths and locations to yield representative groundwater samples for the aquifer and water bearing formations to determine groundwater impacts related to releases from the identified source areas. Monitoring wells are constructed in a manner that maintains the integrity of the borehole, and screens the selected interval desired for sample collection. The monitoring program includes consistent sampling and analysis procedures that are designed to ensure monitoring results that provide a reliable indication of the groundwater quality below and downgradient of the Facility. The SAP includes the protocol for a consistent program for:

- Sample collection;
- Sample preservation and shipment;
- Analytical procedures, and,
- Chain-of-custody control.

These objectives are critical in determining the long-term groundwater quality conditions at the Facility. With the evolution of the monitoring program changing from delineating the nature and extent of groundwater impacts to a focus on corrective action effectiveness, this SAP provides for modifying the sampling parameters at locations upgradient and downgradient of the Site. The primary compounds of concern at the Facility are VOCs (Figure 4), and to a lesser degree monitoring SVOCs and metals.

3.1.2 Selection of Analytical Parameters

The selection of analytical parameters is designed to meet the requirements of DER-10 Technical Guidance for Site Investigation and Remediation (NYSDEC, December 25, 2002). The groundwater monitoring wells are sampled tri-annually (three times per year) as outlined in Table 2, for VOCs and field parameters (Table 3). The list of analytical parameters to be monitored is listed in Table 4 of the SAP, which has traditionally been VOCs (Appendix A). Although this trend will continue, the analytical protocols for SVOCs are included in Appendix B, metals are included in Appendix C, and inorganic anions are included in Appendix D. Specific changes (additional sample parameters) related to corrective action or pilot testing will be addressed in the corresponding Workplan.

For all wells the field parameters are measured at the time of sampling including pH, temperature, specific conductance, dissolved oxygen, ORP, and bottom depths. A summary of the groundwater monitoring parameters is defined on Table 3. Table 5 specifies the recommended collection and preservation for water samples collected at the Facility. Table 6 outlines the sampling parameters and required volume of water required to accurately achieve the QA/QC requirements.

The Facility monitors groundwater quality conditions at every well once (annually) per year, and groundwater performance sampling is conducted two additional times per year (roughly 3-4 months apart). The sampling events are referred to as Annual Sampling Events and Tri-Annual Sampling Events. The Annual Sampling Event are more comprehensive in terms of sampling parameters and locations, while the Tri-Annual Events monitor performance wells for indicator parameters. The

Annual Groundwater Sampling Events for all monitoring wells analyze for VOCs (8260), and ERD performance parameters (Table 4).

3.1.3 Selection of Monitoring Locations

As of October 12, 2007, the Facility samples 10 (ten) monitoring wells on- and off-site to characterize the hydrogeology at and near the Facility. Future phases of the remedial investigation may result in additional wells advanced on- or off-site. Monitoring wells selected in the SAP were chosen based on location, depth, historical groundwater quality results and hydraulic characteristics. A list of the wells and the known construction details are provided on Table 1.

3.1.4 Monitoring Frequency

All of the groundwater monitoring wells will be sampled annually (once per year), and a select series of wells will be sampled twice (two times) more per year, roughly 3-4 months apart. The groundwater monitoring well sampling schedule is provided in Table 3.

3.1.5 Monitoring Well Inspection Program

The groundwater monitoring well inspection and maintenance program is implemented to ensure the structural integrity for all monitoring wells for the duration of the SAP. The well inspection program will be conducted, at a minimum, at the time of sampling at each well. The well and screen casing monitoring program for each well will be conducted on an annual basis. Wells will be monitored for bottom depths and screen occlusion (percent of screen) and compared to the original depths to determine if siltation, or the potential for DNAPL, has occurred at each well. Additional information will be evaluated for turbidity and specific conductance to evaluate significant changes in these parameters. If evidence suggests (e.g., screen occlusions greater than 10 %, or an order of magnitude increase in turbidity and specific conductance) that the well casing or screen is compromised, an inspection will occur which may consist of caliper logging or downhole videotaping, or the well may be re-developed to remove obstructions or foreign material.

3.2 Surface Water

The objective of the surface water monitoring program is to identify sufficient locations to yield representative samples for evaluating surface water conditions, or potential groundwater discharge from the surficial aquifer into the drainage ditch along the eastern property boundary. The north to south ephemeral stream is located off-site at the base of an adjacent railroad track running along the eastern property boundary. Recent observations suggest that the primary stream flow originates north of the Facility. Stream flow becomes channelized south of the property into a buried culvert pipe connected to the City of Geneva storm water sewer system.

During the SRI (Delta, 2006), it was interpreted that groundwater flow and gradients near the steep embankment likely rendered the channel to capture a small volume of groundwater discharge from the surficial aquifer. Based on prior water quality sampling, the highest concentrations of VOCs were detected at the furthest upstream location.

3.2.1 Sampling Objectives

The surface water monitoring locations are selected to be consistent with the objective of the groundwater monitoring program such that the selected stream reaches are the desired interval for sample collection. The monitoring program includes consistent sampling and analysis procedures that are designed to ensure monitoring results that provide a reliable indication of the surface water quality downgradient of the Facility. The SAP includes the protocol for a consistent program for:

- Sample collection;
- Sample preservation and shipment;
- Analytical procedures, and,
- Chain-of-custody control.

These objectives are critical assisting in the determination of the vertical and horizontal extent of surface and groundwater contamination at the Facility.

3.2.2 Selection of Analytical Parameters

Surface water samples will also be characterized for VOCs (Appendix A), pH, temperature, and specific conductance. Table 5 specifies the recommended collection, volume, and preservation for water samples collected at the Facility. Table 6 outlines the sampling parameters, and QA/QC requirements necessary to meet the objectives for sampling for this SAP.

3.2.3 Selection of Monitoring Locations

The surface water sample locations include the monitoring points established in the RI and SRI, and include SW-1 (upstream location), SW-2, and the southern sampling point SW-3. These locations are identified on Figure 2.

3.2.4 Monitoring Frequency

The surface water monitoring locations will be sampled once (1 Sample) per year, coincident with the Annual Groundwater Monitoring Well Sampling, where all wells and surface water monitoring locations are monitored synoptically. This sampling is outlined in Table 7.

3.3 Soil Sampling

The objective of this SAP is to obtain sufficient and representative soil and aquifer data necessary to properly characterize the Site. Soil borings and rock cores must be sampled continuously to the base of the critical stratigraphic section. For nested wells continuous samples must be collected from the surface to the base of the deepest boring or well. Soil borings must be sampled using the split spoon method, generally through hollow stem augers (HSA), direct push drilling methods (i.e., GeoProbe), and bedrock or boulders must be sampled by coring with standard size NX or larger diameter core bits. Samples must be retained in labeled glass jars or wooden core boxes and stored securely and accessible throughout the life of the investigation or corrective action program.

3.4.1 Sampling Objectives

To determine the nature and extent of COC impacts in the subsurface, a representative number of undisturbed samples may be collected from borings using appropriate methods to identify the characteristics of all cohesive soil units, where testing may include analysis for grain size distribution, soil moisture, permeameter, organic carbon, K_{oc}, F_{oc}, Atterberg limits, visual descriptions, bulk density, or other testing.

Sampling data is enhanced with complete and accurate drilling logs, with detail sufficient to reproduce soil unit maps, cross sections, aquifer delineations, and mineralogy classifications. Boring logs must provide detailed characteristics and classification according to the Unified Soil Classification System (USCS), including a description of matrix and clasts, mineralogy, roundness, color, appearance, odor, and behavior of materials using the appropriate descriptive system. Boring logs must contain drilling information observed in the field including: moisture content, location of the water table during drilling, water loss during drilling, depth to significant changes in material and rock, sample (%) recovery, method of drilling, description of drilling fluids or additives, anomalous features, and relevant information regarding vertical and horizontal variation. Similar data, including mineralogy, degree of cementation, color, grain size, and any other physical characteristics of the rock; percent recovery and the rock quality designation (RQD), other primary and secondary features, and contain all drilling observations and appropriate details are required rock coring logs.

3.4.2 Selection of Analytical Parameters

The selection of analytical parameters in soil to evaluate the impacts of Facility-related COCs is primarily related to VOCs, SVOCs, petroleum hydrocarbons, and metals above background concentrations. Previous investigations have delineated the outline of the soil source areas, but additional investigation may be required to determine the lateral and vertical extent of COCs, primarily VOCs.

The further sampling of on- and off-site soils will be proposed in corresponding Workplans.

3.4.3 Selection of Monitoring Locations

The selection of sampling locations in soil to evaluate the impacts of Facility-related COCs will be proposed in corresponding Workplans.

3.4.4 Monitoring Frequency

The selection of sampling frequency, depth, and locations will be proposed in corresponding Workplans.

3.5 Air Sampling

The collection of air samples, primarily soil vapor and indoor air samples is related to the state SVI program, concerned with the process whereby VOCs from a subsurface source migrates to or into the indoor air of buildings.

On June 12, 2007, the results of the SVI survey were submitted to NYSDEC, which included a pre-sampling building survey, locating air and soil vapor sample locations (Figure 3), collection of indoor air and subsurface soil vapor, and analyzing the data (Delta, 2007). The analytical protocol is included in Appendix E, EPA Test Method TO-15.

3.5.1 Sampling Objectives

Proper delineation of soil vapor sources is directly relevant to completed pathway and exposure analysis. VOCs in the subsurface are mobile, and primarily because of a difference between interior and exterior pressures, soil vapor can enter a building through cracks or perforations in slabs or basement floors and walls, and through openings around sump pumps or where pipes and electrical wires go through the foundation. Heating, ventilation or air-conditioning (HVAC) systems and/or the operation of large mechanical appliances (e.g., exhaust fans, dryers, etc.) may create a negative pressure that can draw soil vapor into the building. Soil vapor and indoor can become contaminated when chemicals evaporate from subsurface VOC sources. Permanent sub-slab sampling (SS) locations have been installed in the manufacturing plant floor, and co-located indoor air sampling locations have been established. If soil vapor is contaminated and enters a building, indoor air quality may be affected. When contaminated vapors are present in the zone directly next to or under the foundation of a building, vapor intrusion is possible. Soil vapor can enter a building whether the building is old or new, or whether it is on a slab or has a crawl space or basement.

3.5.2 Selection of Analytical Parameters

The selection of analytical parameters is designed to meet the requirements of the *Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York* (NYSDOH, October 2006). Typically, a minimum reporting limit of 1 microgram per cubic meter (1 $\mu g/m^3$) or less is sufficient for most analytes, as outlined on Table 8.

3.5.3 Selection of Monitoring Locations

The SVI sampling locations were established in March 2007, with the installation of sub-floor probes and co-located indoor air sampling locations, as noted on Figure 3.

3.5.4 Monitoring Frequency

The monitoring frequency for the SVI is anticipated to be conducted annually. Confirmation sampling conducted during the fall-winter 2007 heating season, and the resulting sampling report will propose the monitoring frequency for SVI.

4.0 Field Methods

This section of the SAP provides a discussion of the groundwater sample collection program at the Facility. This sampling program was developed from a review of the results of previous sampling and testing programs conducted. The practical goal is to have each well with downhole dedicated sampling system for sampling groundwater quality. As monitoring progresses and data is re-evaluated, and additional on-site and off-site investigations are completed, sampling points may be added or deleted from the monitoring network.

4.1 Standard Operating Procedures

This section of the SAP provides a discussion sample collection and data acquisition program at the Facility. The groundwater monitoring wells are divided into two categories; wells with dedicated downhole sampling systems, and those wells where downhole sampling systems have not been installed. As such, this SAP includes the procedures for sampling wells and locations with and without dedicated downhole sampling equipment.

Groundwater Sampling – As of September 2007, no site wells (Figure 2) have dedicated downhole equipment and this sampling outline includes the procedures for sampling wells and locations without dedicated downhole equipment. Numerous studies have been completed on groundwater sampling methods involving low-flow rate purging. Studies by Barcelona, et. al., (1994) and Puls and Barcelona, (1996) discuss and compare conventional versus low-flow purging methods and show that low-flow rate sampling meets high data quality objectives. Low flow sampling techniques will be employed in the groundwater monitoring program for the Site.

Wells sampled without downhole equipment

1. Wells will be sampled in sequence from least contaminated to most contaminated which reduces the potential for cross contamination.
2. Weather conditions such as temperature and general atmospheric conditions will be observed and noted on the *Water Sampling Log* Form (Appendix F).
3. The well and immediate area will be inspected and the integrity of the well noted. Any maintenance requirements and problems will be noted on the *Water Sampling Log* Form (Appendix F).
4. The well will be unlocked and the well cap removed. The top and inside of the casing will be wiped off, if needed.
5. A photoionization detector may be used to check for any organic vapors emanating from the well. The readings will be recorded on the well *Water Sampling Log* Form.
6. The depth to all fluid phases (groundwater, LNAPL, and DNAPL) detected in the wells will be measured to the nearest 0.01 foot using either a steel tape with indicator paste, an acrylic bailer, or an electric or optical probe. All groundwater monitoring wells (Table 7), on-site and off-site, associated with the Facility have permanent datum etched into the top of casing (TOC), identified by a notch and permanent markings on the casing, or the north side of the

well casing. All measurements of total depth, water elevation and immiscible phases will be measured and referenced from this point and recorded on the *Water Sampling Log* Form.

7. The total depth of the well to be sampled will be measured to the nearest 0.01-foot and the measurement recorded on the *Water Sampling Log* Form. The depth will be compared to the total depth recorded from initial construction. Wells will be redeveloped if the comparison of total depth (less well bottom sump) shows that 10 % of the well screen is occluded by sediment.

8. The required volume of water to be purged from each well just prior to sampling will be calculated and noted on the *Water Sampling Log*. The calculated purged volume necessary for representative groundwater samples will be determined just prior to sample collection. Purge volumes are calculated specifically for each well by multiplying the well casing volume determined in each well in gallons per foot of water by the feet of water in the well.

9. The wells without dedicated downhole pumps will be purged with a non-dedicated submersible pump such as a 2-inch Grundfos Redi-Flo^2, a low-flow bladder pump, or a peristaltic pump until at least three well volumes have been removed and the field parameters of pH, conductivity, and temperature of the water have stabilized within 10 percent of the last two measurements or the well is pumped dry in which case only one group of readings will be taken. Instances where the saturated thickness is insufficient to purge with a pump a dedicated bailer will be used to collect the sample. Field measurements will be measured according to equipment manufacturer's instructions and referenced EPA methods. Three readings will be taken unless the well goes dry in which case only one reading will be recorded. The readings will be recorded on the *Water Sampling Log* Form. All instruments will be calibrated before each days use according to manufacturer's instructions and referenced EPA methods. The manufacturer and model number of the instruments used at each sample location will be logged in the *Calibration Log Book* for that instrument.

10. The final measurements collected are the most representative of the water sampled and will be recorded on the *Water Sampling Log* Form. All purge volumes and calculations will be recorded on the *Water Sampling Log* Form.

11. The non-dedicated submersible or bladder pump will be fitted with polyethylene tubing for discharge. The pump and tubing will be lowered by hand to 5-feet below the water level or to bottom of the well minus 5 feet. The pump will be powered with a portable generator, situated down-wind and away from the well to avoid contamination from exhaust fumes. Purge rates will be kept low (50 – 300 ml/min), with the intent of limiting drawdown to less than 0.3' during purging. If recharge can not keep pace with the pump rate and the pump breaks suction, the pump will be shut off immediately and the well will be considered dry. The pump will then be lowered by hand to the bottom of the well screen and all sampling containers placed nearby. The pump will then be turned on and the pumping rate reduced to 100 ml/min or less. When the sampling is complete, the pump and tubing will be withdrawn from the well. The pump and tubing will be discarded, or decontaminated. Disposable equipment will be containerized. Water pumped from the wells will be contained and profiled prior to proper disposal.

12. Samples will be collected directly from the sampling port located at the discharge manifold or tubing. Samples will be preserved as appropriate in sample containers provided by the laboratory and submitted and analyzed before the required holding time has expired. Water samples for dissolved metals analyses will be filtered through a 0.45 micron filter to remove suspended particulate matter prior to filling sample bottles, or shipped next day for filtration at the appropriate analytical laboratory. Information on the sample location, analysis to be performed, date and time of sample collection, name of sampler, and preservatives used will be recorded on the sample container label.
13. The individual sample containers will be identified with the unique well ID, identical to the information recorded on the *Water Sampling Log* Form. Samples requiring refrigeration will be placed in a cooler immediately after sampling and cooled to 4^{o} Celsius. Prior to shipment to the laboratory, the samples will remain chilled using ice packs or an on-site refrigerator and kept under constant chain-of-custody surveillance. When shipped to the laboratory, the shipping containers will include the completed Chain-of-Custody Records and laboratory instructions, and sealed with a custody seal. The shipping containers will be shipped to the appropriate laboratory via overnight or same-day courier service.

Wells with downhole equipment

The Facility may implement a low-flow sampling method in the future. This implementation would increase consistency, accuracy and precision and reduce the potential of cross contamination and that can be associated with conventional non-dedicated sampling methods. This would provide more representative groundwater sample results in addition to reducing sampling time and costs. Properly constructed groundwater monitoring wells will allow flow naturally and constantly to recharge the screened interval and by applying a dedicated, low-flow groundwater sampling method samples can be collected without disturbing stagnant water above the screen. This eliminates excessive purge volumes, improves sample quality, and reduces costs (Barcelona et. al., 1994).

Numerous studies have been completed on groundwater sampling methods involving low-flow rate purging. Studies by Barcelona, et. al., (1994) and Puls and Barcelona, (1996) discuss and compare conventional versus low-flow purging methods and show that low-flow rate sampling meets high data quality objectives. An example of a low-flow sampling system developed by QED Environmental Systems, Inc. (QED - MicoPurge™ System), or a similar system may be installed at the Facility. Under this system, each well uses a dedicated bladder pump for extracting groundwater from the well. The pumping rate and pneumatic source is controlled at the surface with QED MicroPurge™ Controller and either compressed breathing-air or nitrogen. Water levels are collected with the MicroPurge™ Water Level Meter which allows continuous water level measurements to be collected during purging. A flow cell analyzer for measuring groundwater stabilization parameters is also used in the system. The following equipment is used in sampling the groundwater at MicroPurge wells:

- Well Wizard Bladder Pumps
- MicroPurge™ Controller
- Oil-less Compressor/compressed breathing-air or nitrogen
- MicroPurge™ Water Level Meter and dedicated probes

Equipment necessary for using the MicroPurge™ System requires the electronic programmable controller (Model 6400). Water levels are measured manually with a downhole electric line measure (ELM) or MicroPurge™ Water Level Meter (Model 6400). The water level meter will record water levels continuously during low-flow sampling to insure drawdown does not exceed 0.3 feet during purging. The 0.3 foot value is the currently accepted maximum drawdown before stagnant water from the casing begins to influence sample quality (Puls and Barcelona, 1996, p.7).

Water samples collected from the surface water streams, springs, and ponds will be collected by filling the samples bottles directly from the stream (Appendix K - SOP #6). Sample containers will be partially immersed for filling ensuring that the preservative is not lost. The cap is then immersed in water and added to the sample vial until the proper amount of liquid is added to the vial. Field measurements will be taken directly in the stream at the sample location. Samples will be handled and shipped according to the procedures described above for the samples collected from the monitoring wells.

Groundwater samples taken from the operating wells will be collected directly from the discharge pipe. Sample preservation methods and sample containers will be consistent the EPA guidelines provided in the Federal Register (EPA, 1984). The sample containers and preservatives that will be used during this monitoring program are given in Table 5. Sampling and analysis, field methodologies, and the procedures utilized at the Facility will follow the Standard Operating Procedures (SOPs), provided in Appendix K.

4.2 Quality Control Samples

Quality Assurance of data will also involve the collection of field replicates, split samples and the use of trip and equipment blanks. The role of these samples along with the preparation and analysis are identified and discussed in the following.

Replicate samples and if necessary, split samples, will be collected following collection of the actual sample vials for a specific analyte group. The sampling protocol would be as follows:

- VOC vials followed immediately by replicate or split VOC vials;
- SVOC vials followed immediately replicate or split VOC vials;
- Metal containers followed immediately by replicate or split metals containers.

4.2.1 Replicate Samples

Replicates or field duplicates of groundwater samples will be collected and used to assess field sampling procedures and the laboratory's precision. Information on the collection of replicate samples will be noted on a *Water Sampling Log* (Appendix F). A replicate sample is collected at the same time, in the same manner, in the same type of container, preserved in the same way, and analyzed by the same laboratory and for the same constituents as the primary sample. A replicate sample will be analyzed for VOCs in the groundwater sample (Table 5). At least five percent of the

total number of locations sampled (or 1 for every 20 sample locations) will be replicated each sampling event (EPA, 1987). The wells replicated may change each sampling event.

4.2.2 Trip Blanks

To assess possible contamination of the samples during shipment to the laboratory, trip blanks for VOC analyses will be shipped along with field samples and will be analyzed at the same time as the field samples. A trip blank is a container filled with organic constituent free water in the laboratory and travels unopened with the empty VOC sample containers to the Facility and is returned with field samples from the Facility back to the laboratory. The trip blank is analyzed for the same volatile organic constituents as the field samples. A minimum of one trip blank, if required, will be included in each shipping container which contains samples for VOC analysis shipped to the Laboratory (Table 9).

Sample temperature will be measured by the laboratory when samples are received and logged in by the sample custodian.

4.2.3 Field Blanks

Equipment field blanks will be prepared by rinsing the sampling equipment after decontamination and collecting a sample of the rinsate. These samples will be analyzed for the same organic and inorganic parameters as field samples to evaluate equipment decontamination procedures. One equipment field blank will be collected during each sampling event. The equipment and the sampling location prior to and after the procedures will be noted on the *Water Sampling Log Form*.

Equipment field blanks will be prepared by collecting a sample of the rinse water used for decontamination of the sampling equipment. One equipment field blank will be collected for every 20 sampling locations. The information will be recorded on the *Water Sampling Log Form*. Equipment field blanks will only be used when wells need to be bailed and even then disposable bailers would be used for each sampling location.

5.0 Sample Custody and Shipment

The primary purpose of sample custody procedures is to create a written record that documents a sample from the moment of collection through analysis. The resulting information aids in data interpretation and serves as legal evidence of sample handling.

Sample custody procedures will be designed to comply with EPA requirements for sample control. All samples will remain in the custody of sampling personnel from the time of collection until transfer to a representative of the courier service for delivery to the laboratory. Stringent chain-of-custody procedures will be followed to maintain and document sample possession and transfer. An example of the Chain-of-Custody Record that will be used during this monitoring program to document sample custody and transfer is included in Appendix G.

5.1 Field Custody

The Sample Team Leader or other appropriate project staff will be responsible for the care and custody of the collected samples until they are shipped to the laboratory.

- Chain-of-Custody Records will be completed to the fullest extent possible prior to sample shipment. They will include the following information: project number, sample number, date and time collected, type of sample matrix, name of responsible party, analyses to be performed, preservative used, bottle description, and total number of bottles. These forms will be filled out in a legible manner using indelible ink and will be signed by the sampler. Similar information provided on the sample label and the *Water Sampling Log Form* will be compared to that on the Chain-of-Custody Record.

5.2 Transfer of Custody for Shipment

The following procedures will be used when transferring custody of samples:

- When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the Chain-of-Custody Record. This record documents sample custody transfer from the sampler to the laboratory. The Chain-of-Custody Records will be sealed in plastic within shipping containers or coolers.
- Samples will be packaged properly for shipment and dispatched to the laboratory for analysis. A separate Chain-of-Custody Record will accompany each container shipped. Shipping containers will be sealed with a Chain-of-Custody Seal for shipment to the laboratory. Other pertinent information for the laboratory will be entered in the remarks section of the Chain-of-Custody Record.
- The original Chain-of-Custody Record will accompany the shipment and a copy will be retained by the field sampler.
- Proper documentation also will be maintained for shipments by the courier service.

5.3 Sample Shipment Procedures

The following procedures will be followed when shipping samples for laboratory analysis:

- Samples requiring refrigeration will remain chilled to a temperature approaching 4^{o} C and packaged in an insulated shipping container or cooler for transport to the Laboratory.

- Only shipping containers which meet all applicable state and federal Department of Transportation standards for safe shipment will be used.

- The shipping containers will be sealed with a Chain-of-Custody Seal that will allow the laboratory receiver to quickly identify any tampering which may occurred during transport to the laboratory.

- The Chain-of-Custody Record and Laboratory Task Order will be placed inside the shipping container in a sealed plastic envelope.

- Shipment will be by courier service and receipt of shipment will be retained with Chain-of-Custody Record.

5.4 Laboratory Custody

Upon receipt of the samples at the Laboratory, the sample custodian will note the condition of the shipping containers and each sample, record the temperature of the temperature blank, and log in the samples. The accompanying original Chain-of-Custody Record will be signed and dated and returned to the Woodbury, MN office. The laboratory will maintains a tracking record that documents the sample from receipt through analysis and storage or final disposition. The laboratory's sample custody program is discussed in their QAP (Appendix H, Appendix I). Upon check-in at the laboratory, an electronic version (.pdf) of the Chain-of-Custody Record is forwarded via e-mail to the Site Manager and Project Manager, documenting the requested analysis.

5.5 Documentation Responsibilities

It will be the responsibility of the Project Manager or Sample Team Leader to arrange for securing all documents produced in the field at the end of each day's work. In addition all project related documents, information, communications, and reports will be stored and filed in the Woodbury, MN office. Field activities will be recorded on forms designed to ensure clear and complete records are maintained.

Examples of these forms are presented in the Appendices and may include the following:

- *Water Sampling Log* (Appendix F)
- Chain-of-Custody Record(Appendix G)

ECOR has implemented an electronic database to manage the ever increasing volume of analytical laboratory data, remediation operation and monitoring data, and environmental information. Verified laboratory data is down-loaded directly from the laboratory information management system (LIMS) in a standard electronic format onto a floppy diskette. Laboratory reports are supplied in .pdf format directly from the LIMS, which is directly uploaded into the GIS\Key™ Database. GIS\Key™ software is a comprehensive environmental database and information management system that seamlessly integrates with computer visualization systems for ease of regulatory reporting and environmental information management. GIS\Key's™ database platform is Microsoft Visual FoxPro, which uses the dBase III+ or .DBF file format which can be read from, and written to, by almost any database or spreadsheet software program. The drawing management capabilities allows the environmental professional to visualize their own interpretations of site data into any graphic, CAD or GIS platform.

6.0 Laboratory Procedures

TestAmerica is the principle laboratory conducting soil and groundwater analysis under the SAP. A copy of the TestAmerica Quality Management Plan (QMP) is included in Appendix H. The primary laboratory contact is:

TestAmerica Buffalo
10 Hazelwood Drive
Amherst, NY 14228
Tel 716.691.2600
Fax 716.691.7991
http://www.testamericainc.com/

All other laboratories utilized at the Facility will meet the laboratory criteria outlined in Appendix I (TestAmerica) and Appendix J (Microseep), at a minimum. The primary laboratory will be the TestAmerica – Buffalo Facility, however, samples may also be analyzed at the TestAmerica – North Canton facility (Appendix L). Operating procedures will be those outlined under the Statements of Work for the EPA Contract Laboratory Program (CLP). Any other laboratory conducting work at the Facility will supply the individual laboratory QAP, which will be kept on file at the Woodbury, MN office.

6.1 Calibration Procedures and Frequency

Appendix I specifically addresses instrument calibration procedures and frequency. Additional instrument preventative maintenance are discussed in the Laboratory Quality Manual (Appendix I). Each method has its own requirement for calibration checks using a variety of QA/QC samples. In general all methods require that if a calibration check is not acceptable, all samples are re-analyzed after the last acceptable calibration check is re-analyzed. Initial and continuing calibration procedures will be those required by the EPA.

6.2 Analytical Procedures

The acceptance criteria and general procedures for the analytical methods used by TestAmerica are outlined in Tables 10 of Appendix I. Each of these references cite specific procedures that include precision and accuracy of the methodology to achieve the required method detection limits, and corrective actions if the test does not meet the QC criteria. The methods of interest are for water analysis and predominately are those specified by the EPA. The specific analytical methods to be used for project samples are presented in Table 4 of this SAP.

6.3 Internal Quality Control

TestAmerica quality control checks are described in Section 5.8 of their QAM included in Appendix I. The main elements of their program are:

- Proficiency Testing
- Control Samples
- Calibration
- Permitting Departures from Documented Procedure
- Development of QC Criteria, Non-Specified in Method/Regulation.

The QAM also describes the frequency that quality control samples are analyzed. The QAM also describes reagent quality control blanks and reference calibration standards used for various analytical methods.

6.4 Performance and Systems Audits

TestAmerica audit schedule and the operation of system and performance audits is discussed in Section 4.13 of Appendix I. These audits are based on EPA requirements. Performance audits specifically utilize blind check samples. TestAmerica uses an independent firm to provide these blind QC check samples on a quarterly basis. Government proficiency samples are analyzed annually to maintain certification. Control charts are maintained for each instrument to evaluate, on a quarterly basis, the instrument's performance. Two laboratory control samples are used in this QC check.

6.5 Preventative Maintenance Procedures and Schedules

TestAmerica preventative maintenance program for analytical equipment is described in Section 4.11 of Appendix H. Routine maintenance is performed by laboratory personnel. Repairs are performed by trained service engineers of the instrument manufacturer under service contracts. Detailed log books documenting maintenance and repairs are maintained for each analytical instrument.

6.6 Corrective Action For Internal Laboratory Problems

TestAmerica has implemented a close-loop corrective action system that provide steps for identifying and correcting variances in laboratory operations, including calibration, maintenance, and analytical methods in an effective and timely manner. These are discussed in Section 4.10 of Appendix H. Full documentation of the corrective action is filed with the QA department.

7.0 Data Validation

The overall QA objective is to ensure that monitoring data of known and acceptable quality are obtained. To achieve this objective Vickers will implement sample collection and chain-of-custody procedures to ensure that representative samples are collected for analysis. A subset of the analytical data may be validated by an independent 3rd party validator identified in Section 2 of the SAP to ensure it meets precision, accuracy, completeness, representativeness, and comparability requirements (EPA, 1987). If sufficient data irregularities are identified and the data validation program recommends, the full data package will be validated. The following is a summary of these data quality requirements.

Precision -- The QA objective in testing precision is to demonstrate the reproducibility of the data. The precision analytical measurements will be evaluated and reported along with the method reference number in a manner consistent with previously published data on precision. Also, precision will be assessed by the use of replicate field samples. Moreover, precision measurements will be done with high purity materials, knowledgeable laboratory personnel, internal quality control, and consistent scientific practice.

Accuracy -- Accuracy is the relationship of the reported data to the "true" value and will be (1) reported with the data, (2) attained by using standards that are difference from those used during routing operations, and (3) consistent with any previously published accuracy data from the applicable literature, and federal and state regulations and guidelines. Accuracy will also be assessed by the use of matrix spike and matrix spike duplicate samples.

Completeness -- Completeness is a measure of the amount of valid data obtained from a measurement program compared to the amount that would be expected to be obtained under normal conditions. The database will be routinely assessed on the basis of expected versus actual data.

Representativeness -- All data should be representative of the actual conditions at the sampling location. Considerations in evaluating the representativeness of the data include, but are not limited to, the location being sampled, the methods used to obtain samples at the Facility, and the appropriateness of the analytical method to the type of sample obtained.

Comparability -- All data will be reported in units consistent with both federal and state regulations, methods, and guidelines. All data will be compared with previously reported data that addresses the same concerns through a similar approach. Comparability between databases will also be achieved by citing standardized sampling and analysis methods and data report formats and use of the previous laboratory. Any deviation from the standard operating procedures will be noted and if necessary, data will be qualified for comparative purposes.

7.1 Objectives

Validation of the analytical data will primarily be the responsibility of the laboratory. Evaluation of the data validation and reporting will be performed by an independent 3rd party. For each groundwater monitoring event the laboratory will be required to submit reports that are supported

by sufficient QA backup information and data to enable the data reviewer to evaluate the quality of the data. The results of these data validations will be reported as required. Data reduction will be performed by ECOR and the results presented in chemical data tables organized by monitoring well location and sample date.

The data validation procedures will include an evaluation of field data and an evaluation of the laboratory analytical data. The data validation checklists presented in Appendix I will be used as guides in evaluating sample collection, field records, and analytical performance; these checklists will aid in identifying valid data and classifying (usability determinations) the data into one of three categories: unusable data (Class U), qualitative data (Class A) and quantitative data (Class B).

The assessment of environmental data occurs in two phases. The first phase consists of reviewing and determining the validity of the analytical data. The second phase consists of interpreting the data to determine its usability. The information from the assessment is included in the data quality assessment report with the results from each sampling event. Only the routine sampling event can be validated by the laboratory performing the analysis. All other validation activities must be performed by a validator independent of the laboratory performing the analyses.

7.2 Validation of Field Data

The field data will be reviewed for completeness and accuracy. The field data includes all of the field records and measurements developed by the sampling team personnel. It is compiled from all of the field activity forms. Failure in any of these areas may result in the data being invalidated.

The field data validation procedure will consist of:

- A review of field data contained in the *Water Sampling Log* for completeness.
- A verification that equipment, field and temperature blanks, and trip blanks were properly prepared, identified and analyzed.
- A check on the field calibration and condition of instruments used for recording stabilization data.
- A review of Chain-of-Custody Records for proper completion, signatures of field personnel and laboratory sample custodian, and dates.

7.3 Validation of Analytical Data

If validation of the field or analytical data is deemed necessary, validation of the analytical data will be performed. The validation steps will be performed by the validator following data validation procedures. Data validation is the process by which the quality of the data is determined with respect to data quality criteria that are defined in facility and laboratory quality control programs and in the referenced analytical methods. Data validation must be performed on a minimum of one in 20 samples (five percent) for every Baseline and Expanded Parameter sampling event.

The process consists of data editing, screening, checking, auditing, certification, reviewing and interpretation. The goal of the process is to define and document data quality and determine if it is sufficient for the intended use. Ideally, data validation evaluates the data quality in terms of the

DQOs. The validator should have experience evaluating data packages for NYSDEC ASP or USEPA Contract Laboratory Program (CLP) compliance. These procedures, where applicable, utilize most current EPA *Laboratory Data Validation Functional Guidelines for Evaluating Organics and Inorganic Analyses*, and the EPA *Precision and Accuracy Statements* (DQOs specified) for the analytical methods employed (Table 6). The following is a description of the tasks that should be performed during data validation:

Task I: Completeness-the validator should review the data package to determine completeness. A complete data package should contain the following components:

- sample chain-of-custody forms;
- case narratives including sample analysis summaries;
- AQA/AQC summary forms and supporting documentation;
- relevant calibration data and supporting documentation;
- instrument and method performance data;
- documentation showing the laboratory's ability to attain the method detection limits for analytes in the required matrices;
- data report forms, including sample preparation logs, analytical run logs and examples of the calculations used in determining final concentrations; and
- raw data used in the identification and quantification of the contract-specified analytes

Deficiencies in completeness should be reported to the AQA officer. The laboratory should be contacted by the AQA officer and should be given a reasonable time (ten calendar days) to produce the documentation necessary to remove the deficiencies.

Task II: Compliance-the validator should review the submitted data package to determine compliance with those portions of the SAP that pertain to the production of laboratory data. Compliance is defined by, but not limited to, the following criteria:

- the data package is complete as defined in Task 1 above;
- the data has been produced and reported in a manner consistent with the requirements of the SAP;
- SAP-defined AQA/AQC criteria have been met;
- instrument calibration requirements have been met for the time frame during which the analyses were completed;
- SAP-defined initial and continuing calibration data is present and documented;
- data reporting forms are complete for all samples submitted, including sample dilution and concentration factors and re-measurement sample clean-up procedures; and
- problems encountered during the analytical process have been reported in the case narrative.

The data validation task requires that the validator conduct a detailed comparison of the reported data with the raw data submitted as part of the supporting documentation package. It is the responsibility of the validator to determine if the reported data can be substantiated by applying protocol-defined procedures for the identification and quantification of the individual analytes.

Task III: Report Submittal-the validator must submit a final report to the Department. The final report should discuss the results of the data review process that includes:

- a general assessment of the data package as determined by the accomplishment of Tasks I and II above;
- detailed descriptions of any deviations from the required protocols. These descriptions should include references to the portions of the protocols involved in the alleged deviations. Accuracy and defensibility issues should also be addressed;
- failures in the validator's attempt to reconcile the reported data with the raw data from which it was derived. Documentation of pertinent telephone conversations and meeting minutes;
- a detailed assessment by the validator of the degree to which the data may have been compromised by any deviations from protocol, AQA/AQC breakdowns, lack of analytical control, etc., that occurred during the sampling acquisition and analytical processes;
- a copy of the laboratory's case narrative and field notes, if applicable;
- data summary forms with validator qualifiers; and
- a chart, consisting of the site name, sample numbers, date submitted to the laboratory, methodologies, matrix, and fractions analyzed (i.e., volatiles, semi-volatiles, pesticides, metals).

Data usability analysis review for usability is a process that evaluates the validated data in context to the original DQOs. The formal process of usability determination involves a complex series of editing, screening, auditing, verifying and reviewing the validated data. The analysis should evaluate the following items:

- determination of whether or not the DQOs were met;
- consistency of the data with the site history;
- consistency of the data with regard to previous data rounds;
- evaluation of blanks for potential field or laboratory contamination;
- evaluation of duplicates for representativeness;
- evaluation of the matrix effect for high or low bias tracking;
- evaluation of the data characteristics (i.e., precision, accuracy, representativeness, comparability, completeness and defensibility) and justification for the use of both compliant and non-compliant data; and

- integration of field and laboratory data with hydrogeological, geological, and meteorological data to provide information for the intended use of the data.

7.4 Data Usability Summary Reports

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.

The DUSR and the data deliverables package will be reviewed by the DER Quality Assurance Unit. 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 quality control verification forms?
6. Have the correct data qualifiers been used?

Evaluation of NYSDEC ASP Matrix Spike Blank (MSB) data - If the MSB recovery is less that the ASP criteria, the positive results should be qualified as J, estimated biased low. If the MSB recovery is less than the ASP criteria, but greater than 10%, the non-detects should be qualified J, biased low. If the MSB recovery is less than 10%, the non-detect data must be rejected.

Any Quality Control exceedances must be numerically specified in the DUSR and the corresponding QC summary sheet from the data package should be attached to the DUSR. All data that would be rejected by the EPA Region 2 Data Validation Guidelines must also be rejected in the DUSR. 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 re-sampling/reanalysis. All data qualifications must be documented following the NYSDEC ASP 1995 Rev. guidelines.

Unless otherwise approved in advance by the DER, laboratory data deliverables should be as follows .

i. Category B laboratory data deliverables as defined in the analytical services protocol (ASP) should be submitted for confirmatory (post remediation) samples and final delineation samples for all sites except those noted for exception. In addition, a Data Usability Summary Report should be prepared by a party independent from the laboratory performing the analysis.

ii. Category A (as defined in the ASP) or Category Spills laboratory data deliverables should be submitted for all other analyses; and,

iii. Analytical results without all quality control documentation and raw data may be provided for all intermediate sampling events and for all long-term groundwater monitoring samples where the site has DER oversight, provided the following information is submitted:

(1) A cover page, including facility name and address, laboratory name and address, laboratory certification number, if applicable, date of analytical report preparation and signature of laboratory director;

(2) A listing of all field sample identification numbers and corresponding laboratory sample identification numbers;

(3) A listing of all analytical methods used, including matrix cleanup method;

(4) The method detection limit and practical quantitation level for each analyte for each sample analysis;

(5) All sample results including date of analysis;

(6) All method blank results; and

(7) All chain of custody (Appendix G) documentation.

iv. Upon written request, the DER may require that deliverables package be upgraded to a "Category B" data deliverables package for any sample analysis. If the backup documentation is not available to generate “Category B” deliverables or that the lab is not qualified to generate “Category B” deliverables (not ELAP-CLP lab), re-analysis or re-sampling and analysis is an option.

7.5 Holding Times

The guidelines set forth in Table II of the Federal register (EPA, 1984) will be utilized to evaluate the effects of exceeded holding times upon the validity of the data. Any data that exceed the maximum established holding time(s) will be evaluated on a case-by-case basis to determine whether such data should be assigned a validation level of A (qualitative) or U (unusable). This determination will be founded on (1) the professional judgment of the validator and an advising

expert chemist, (2) an appreciation for the biases resulting from exceeding holding times, and (3) the comparison of the given data with historical data.

To ensure holding times are met, all samples will be extracted for analysis within the holding time allocations specified in their appropriate test methods. The results for any analyses performed on samples that exceed the extraction holding time requirements may be validated as level U (unusable). The corrective actions to rectify the problem of exceeded holding times may be re-sampling and analysis of the samples, if needed.

7.6 Spike Recoveries and Relative Percent Differences

Matrix spike recoveries are conducted by the analytical laboratories and are reported as a percent of the amount of the known spiked target compounds that are recovered during sample analysis. Surrogate spike recoveries are also conducted by the analytical laboratories and are reported as a percent of the amount of the known spiked non-target compounds that are recovered during sample analysis. The RPD is the difference between the primary and secondary spike values divided by the average of the primary and secondary spike recovery values.

The spike recovery and RPD limits prescribed by the EPA in their *Contract Laboratory Program Statement of Work For Organic Analysis* (EPA, 1986) and Inorganic Analysis (EPA, 1987) will be utilized as a measure of data validity. Data related to average spike recoveries that fall within the prescribed spike recovery limits will be considered B (quantitative). Average spike recoveries outside the prescribed limits dictate that related sample data be considered A (qualitative) or U (unusable). A decision of whether a validation level of A or U be assigned to poor average spike recovery data will be made by the validator on a case-by-case basis. A significant departure of the average spike recoveries from the established limits will result in a significant reduction in the validity of the related data.

7.7 Corrective Measures for Invalid Data

Actions to correct problems associated with the quality assurance requirements for a sample collection, sample custody, and sample analysis will be implemented as needed. Problems in sample collection will be corrected and documented by Vickers. Depending on the nature of the problem and the corrective action taken, appropriate personnel may be notified. Problems in sample analysis encountered by the laboratory that affect the quality of data will be corrected by the laboratory and properly reported to the Woodbury, MN office and documented.

If the validation review reveals unacceptable reporting or data, the Project Director, Project Manager and/or Project QA/QC Coordinator will implement corrective action. Corrective action may include:

- Re-analyzing the samples if the holding time criteria permit
- Re-sampling and analyzing if the holding time is exceeded
- Amending data reports and results
- Evaluating and amending analytical procedures with regulatory approval
- Designating data as qualitative.

8.0 Reporting

Data collection activities to be completed during the monitoring program include groundwater and surface water quality sampling, the SVI monitoring, implementation of corrective action and IRM, and subsequent investigative activities. Monitoring events will be scheduled and notification will be submitted to NYSDEC. Groundwater monitoring reports will contain analytical results, field parameter measurements, copies of field sampling and well inspection log sheets, well repair documentation, QA/QC data, statistical analyses of groundwater data, field investigation results, and other relevant groundwater related information collected during the reporting period.

Reporting of monitoring data will primarily occur in PSR, Annual Groundwater Monitoring Reports, IRM Completion Reports, and corrective action completion reports. A summary of the information for the groundwater monitoring reports will include the following:

Description of field activities

- status and integrity of the monitoring well network;
- surface and subsurface well integrity problems noted, or repaired;
- discussion of any variations in sampling protocol.

Summary of groundwater quality data

- contaminant transport rate and direction;
- horizontal and vertical extent of contamination;
- analytical data summary tables;
- tables, maps, and charts which depict time changes in water quality;
- discussion of data validation results;
- evaluation of variations in upgradient and/or downgradient groundwater quality.

Summary of water level data

- water level data summary tables;
- various hydrographs to depict groundwater conditions during reporting period.

Groundwater level data generated from monitoring will be tabulated and used to generate groundwater potentiometric contour maps for selected monitoring. Contouring and evaluation of the data will be performed by or under the direction of a qualified and experienced geologist. Groundwater quality data will be tabulated for each sampling event. Isoconcentration contour maps may be prepared for selected constituents, and data tables will be used to summarize the constituents analyzed, concentrations detected, units of the concentrations, detection limits, sample location and data of sampling. A separate table may be prepared for the QA/QC samples and will include similar information. The quality assurance information for the groundwater program will be presented as required and will include the following:

- Field and laboratory quality assurance activities;
- Data validation results;
- Precision and accuracy of data;
- Completeness of data;
- Usability of data.

In the *Annual Groundwater Monitoring Reports*, a brief summary of the adequacy of the monitoring program will be compiled at the end of each reporting. If inadequate, recommendations on changes to the monitoring program will be made. Reporting will principally be in electronic format, provided via CD-ROM. Limited paper copies will be prepared, but they will contain the same data as the electronic copies. Distribution of Workplan submittals and corrective action reports, at a minimum, will consist of:

Bart Putzig, P.E.
New York State Dept of Environmental Conservation
Division of Environmental Remediation
6274 East Avon-Lima Road
Avon, New York 14414-9519
(585) 226-5349

Debbie McNaughton
New York State Department of Health
335 E. Main Street
Rochester, NY 14604-2127

Gary Litwin
Bureau of Environmental Exposure Investigation
New York State Department of Health
Flanigan Square
547 River Street
Troy, New York 12180-2216

Joseph White
NYSDEC
Division of Environmental Remediation
6274 East Avon-Lima Road
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Andrew J. English, P.E.
New York State Dept of Environmental Conservation
Division of Environmental Remediation
625 Broadway Avenue
Albany, New York 12233-7010

James Craft
NYSDEC
Division of Environmental Remediation
6274 East Avon-Lima Road
Avon, New York 14414-9519

Glen R. Bailey, Esq.
Division of Environmental Enforcement
New York State Dept of Environmental Conservation
270 Michigan Avenue
Buffalo, New York 14203-2999

Kristin Colberg
H.B. Fuller Company
1200 Willow Lake Blvd
St. Paul, MN 55110-5101

9.0 Health and Safety

A copy of the Site *Health and Safety Plan* (SHSP) will be made available to all employees prior to their Site work. They will be required to review this plan before the start of any site activities. All other personnel involved with remediation activities will have received a pre-entry briefing.

Chemical Hazards

A summary of the exposure limits and physical properties of the organic chemicals of concern are outlined in the SHSP, consisting primarily of chlorinated solvents and petroleum hydrocarbons in soil, vapor, groundwater and surface water. The potential pathways of chemical exposure may be through inhalation, ingestion, injection, and skin contact. This information was used to develop action levels for on-site activities. Although the chemicals identified are a potential health concern, measures will be taken to minimize potential for exposure. Because these contaminants were identified as potential constituents in groundwater, employees performing intrusive activities, such as drilling, subsurface injections involving soil and groundwater, and miscellaneous maintenance work, excavations, and equipment installations, should follow the specific health and safety procedures provided in the SHSP. The potential acute and chronic affects of chlorinated solvents and VOC exposures are described in the SHSP, as well as action levels to mitigate these risks.

Physical Hazards

The SHSP contains a summary of potential work hazards that may be encountered during remediation activities. Potential hazards are described according to each specific job task. The two main physical tasks performed at this Site include drilling and injection activities into the shallow subsurface, groundwater monitoring and soil sampling, and risks associated with large construction equipment related to conducting site investigation and sampling (drilling rigs, forklifts, trucking, maintenance equipment, vehicle traffic). Other physical activities performed at this Site, such as regular maintenance work and equipment installations, exposure of employees to potential hazards , and standard operating safety procedures are described in detail in the SHSP.

Health and Safety Program Manager

ECOR has designated an employee as its Health and Safety Program Manager (HSPM). This person has the authority to command sufficient resources to safely perform the Work. Mike Wisnewski, P.E., has been designated as the HSPM.

Project Manager

ECOR has designated an employee as its Project Manager (PM), who oversees all aspects of the implementation of this work. This person has the authority to command sufficient resources to perform all work associated with this IRM Workplan. Keith Rapp has been designated as the PM.

Health and Safety Coordinator

ECOR has designated a Site Health and Safety Coordinator (SHSC) for this project. Day-to-day industrial hygiene support, including air monitoring, training, site safety inspections, shall be provided by the SHSC. The SHSC has the authority to stop on-site operations whenever conditions threaten the health or safety of employees. The SHSC or his\her designee will remain on-site during all project operations.

ECOR has designated Jennifer Good as the SHSC. Ms. Good has a sound working knowledge of occupational safety and health regulations; experience on the type of project described in this SHSP; and training in air monitoring practices and techniques.

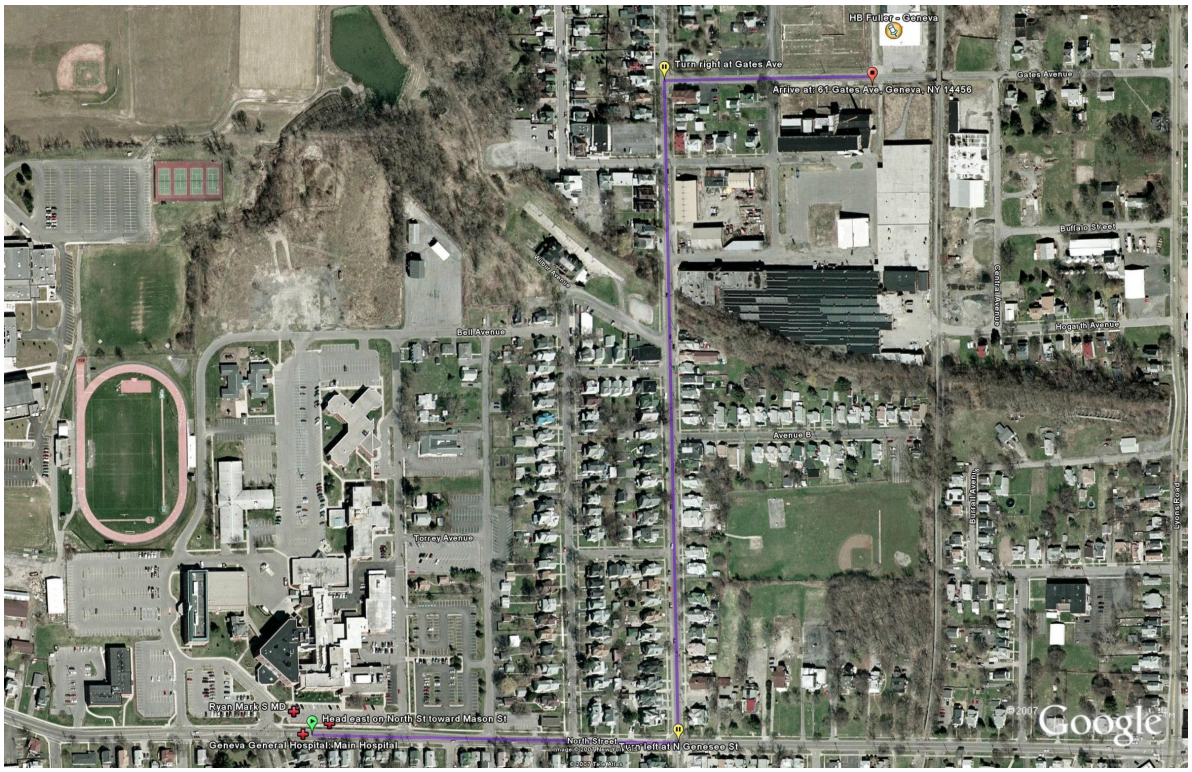
Subcontractors

ECOR may be supported in the remediation activities by subcontractors. All subcontractors involved with on-site operations will be provided a copy of this SHSP. Subcontractors involved with intrusive remediation activities will be informed of the health and safety concerns and will be responsible, at a minimum, for maintaining the health and safety requirements presented in this plan. Information, such as air monitoring and analytical results, will be shared with subcontractors to assist them in addressing the health and safety recommendations. ECOR will ensure that subcontractors have met the training, respirator certification and medical examination requirements of the OSHA Hazardous Waste Operations and Emergency Response Standard (29 CFR 1910.120). Subcontractors involved with intrusive activities will be required to provide to ECOR completed certification forms for this purpose.

The nearest emergency room to the Site is the Geneva General Hospital. The directions are as follows.

Route to Hospital

From:	Former Monarch Chemical Facility	**Arrive:**	Geneva General Hospital
	61 Gates Ave		196 North St
	Geneva, NY 14456		Geneva, NY 14456
Drive time: 0.7 mi – about 2 mins			(315) 787-4000



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Tables

Table 1
Groundwater Monitoring Well Completion Details
Former Monarch Chemicals Division Facility
61 Gates Avenue
Geneva, New York

	Top of Casing Elevation (ft AMSL)	Depth of Borehole (ft below TOC)	Well Diameter (in)	Casing Material	Screen Material	Screen Slot Size (in)	Screen Length (ft)	Total Well Depth (ft below TOC)	Height of TOC (ft. above surface)	Bottom Sump Depth (ft)	Depth to Top of Screen (ft below TOC)	Top of Screen Elevation (ft AMSL)	Depth to Bottom of Screen (ft below TOC)	Bottom of Screen Elevation (ft AMSL)	Date Installed
MW-114	462.03	16	2	PVC	unk	0.01	10	14.0	0	0	4.0	458.03	14.0	448.03	7/14/2005
MW-115	462.25	16	2	PVC	unk	0.01	10	14.0	0	0	4.0	458.25	14.0	448.25	7/12/2005
MW-116	462.44	16	2	PVC	unk	0.01	10	13.9	0	0	3.9	458.54	13.9	448.54	7/12/2005
MW-117	463.44	16	2	PVC	unk	0.01	10	13.7	unk	0	3.7	459.74	13.7	449.74	7/12/2005
MW-118	462.40	16	2	PVC	unk	0.01	10	14.0	0	0	4.0	458.40	14.0	448.40	7/12/2005
MW-119	462.68	16	2	PVC	unk	0.01	10	14.0	0	0	4.0	458.68	14.0	448.68	7/11/2005
MW-120	462.12	16	2	PVC	unk	0.01	10	14.0	0	0	4.0	458.12	14.0	448.12	7/11/2005
MW-121	461.77	16	2	PVC	unk	0.01	10	14.0	0	0	4.0	457.77	14.0	447.77	7/13/2005
MW-122	461.65	16	2	PVC	unk	0.01	10	14.0	0	0	4.0	457.65	14.0	447.65	7/13/2005
MW-123	456.42	16	2	PVC	unk	0.01	10	16.5	0.5	0	6.5	449.92	16.5	439.92	7/14/2005

Table 2
Groundwater Monitoring Schedule
Former Monarch Chemicals Division Facility
61 Gates Avenue
Geneva, New York

Sample Location	Annual Sampling Event [1]	Tri-Annual Sampling Events [2]
MW-114	X	X
MW-115	X	X
MW-116	X	X
MW-117	X	X
MW-118	X	X
MW-119	X	X
MW-120	X	X
MW-121	X	X
MW-122	X	X
MW-123	X	X
SW-1	X	
SW-2	X	
SW-3	X	

1 Annual Perimeter Groundwater Sampling Events for all wells for VOCs, performance monitoring parameters and field parameters for all wells and surface water sampled (see Table 3)

2 Tri-Annual Sampling Events consists of VOCs + field parameters. All wells will be sampled 3 times\year, as field conditions permit.

Annual sampling event will be conducted Mar-Apr yearly.
Triannual sampling will be conducted in July and October.

April 27, 2026

Table 2 has been replaced by Table I, Groundwater Monitoring Schedule, which is included in H & A of New York Engineering and Geology LLP's letter dated April 27, 2026.

Table 3
Annual Groundwater Sampling Program
Former Monarch Chemicals Division Facility
61 Gates Avenue
Geneva, New York

Annual Sampling Parameters								
Well	**Water Level**	**Temp [°C]**	**ORP [mV]**	**pH [SU]**	**DO [mg/L]**	**Specific Cond. [umhos/cm]**	**VOCs (8260)**	**TOC (9060)**
MW-114	X	X	X	X	X	X	X	X
MW-115	X	X	X	X	X	X	X	X
MW-116	X	X	X	X	X	X	X	X
MW-117	X	X	X	X	X	X	X	X
MW-118	X	X	X	X	X	X	X	X
MW-119	X	X	X	X	X	X	X	X
MW-120	X	X	X	X	X	X	X	X
MW-121	X	X	X	X	X	X	X	X
MW-122	X	X	X	X	X	X	X	X
MW-123	X	X	X	X	X	X	X	X

Table 4
Groundwater Monitoring Parameters
Former Monarch Chemicals Division Facility
61 Gates Avenue
Geneva, New York

Parameters	Sampling Method	Frequency
VOCs	EPA 8260	Annually all wells. Tri-Annual groundwater Locations listed in Table 3.
VOCs (air)	TO-14\TO-15	As necessary for SVI.
SVOCs	EPA 8270	As necessary for RFI.
Metals	EPA 6010	As necessary for RFI.
Cyanide	EPA 335.2	As necessary for RFI.
Inorganics	EPA 300 Series	As necessary for RFI.
Groundwater elevation	field measurement	Measured Tri-annually, at a minumum for all locations in Table 1.
NAPL Thickness	field measurement	NAPL not expected, ELM suitable for measurements.
Well Depths	field measurement	Measured at time of sampling or well repairs\maintenance.
pH	field measurement	Measured during purging.
Temperature	field measurement	Measured during purging.
Specific conductance	field measurement	Measured during purging.
Dissolved Oxygen	field measurement	Measured during purging.
ORP	field measurement	Measured during purging.

Surface Water and

Volatile Organic Compounds (EPA 8260)	Metals (Dissolved - EPA Method 6010)
(All Sampling Events)	**(As necessary for performance monitoring)**
1,1,1-Trichloroethane	Barium
1,1,2,2-Tetrachloroethane	Cadmium
1,1,2-Trichloroethane	Chromium
1,1-Dichloroethane	Lead
1,1-Dichloroethene	Zinc
1,2-Dichloroethane	
1,2-Dichloropropane	
2-Butanone	**Inorganics - Non-Metals (Total - EPA Method 335.2)**
2-Chloroethyl vinyl ether	**(As necessary for performance monitoring)**
2-Hexanone	Cyanide
4-Methyl 2-pentanone	
Acetone	**Pilot Testing Programs**
Benzene	**(As necessary for performance monitoring)**
Bromodichloromethane	Fluoride (EPA 300)
Bromoform	Sulfate (EPA 300)
Bromomethane	Chloride (EPA 300)
Carbon disulfide	Iron (EPA Method 6010)
Carbon tetrachloride	Bromide, Chloride Nitrogen, Sulfate (ion chromotograph EPA 300.0)
Chlorobenzene	Ethane, Ethene (Test Method AM18 or AM20GAX)
Chloroethane	Methane (Test Method AM18 or AM20GAX)
Chloroform	Carbon Dioxide (Method RSK114)
Chloromethane	Total Organic Carbon (EPA 415.1)
Dibromochloromethane	Alkalinity (as $CaCO_3$ [Method 310.1])
Ethyl benzene	
Methylene chloride	**Physical Parameters (Field)**
Styrene	Temperature
Tetrachloroethene	pH
Toluene	Conductivity
Trichloroethene	Dissolved Oxygen
Vinyl acetate	Oxidation Reduction Potential
Vinyl chloride	
Xylenes - Total	
cis-1,2-Dichloroethene	
cis-1,3-Dichloropropene	
trans-1,2-Dichloroethene	
trans-1,3-Dichloropropene	

Table 5
Collection and Preservation of Groundwater and Surface Water Samples
Former Monarch Chemicals Division Facility
61 Gates Avenue
Geneva, New York

Parameter	Volume Required	Container[1]	Preservative[2]	Holding Time[3]
VOCs				
VOCs	40 ml	G	4° C, HCL	14 Days
SVOCs				
SVOCs	1000 ml	G	4° C, HCL	7 Days
Inorganics – Metals				
All Metals	1000 ml	P	HNO_3	6 months
Inorganics -- Non-Metals				
Cyanide	500 ml	P, G	4° C, NaOH	14 Days
Physical				
pH	25 ml	flowcell	None	Immediately
Temperature	25 ml	flowcell	None	Immediately
Conductivity	25 ml	flowcell	None	Immediately
Diss. Oxygen	25 ml	flowcell	None	Immediately
ORP	25 ml	flowcell	None	Immediately

Notes:

[1] Plastic (P), or Glass (G). For metals, polyethylene with a polypropylene cap. Physical parameters measured in QED MicroPurge flowcell.

[2] When any sample is to be shipped by common carrier or sent through the United States Mail, it Person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of environmental samples, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials regulations do not apply to the following materials: Nitric Acid (HN03) in water solution at concentrations of 0.15% by weight or less (pH about 1.62 or greater).

[3] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held for analysis and the results considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for a longer time, and has received a variance from the USEPA Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the samples for a shorter period of time, if knowledge exists to show this is necessary to maintain sample stability.

Table 6
Sampling Parameters, Detection Limits, and QA/QC Summary for Water Samples
Former Monarch Chemicals Division Facility
61 Gates Avenue
Geneva, New York

Parameters	USEPA Method	Detection Limit	Precision	Accuracy	Completeness
Organics[1]					
VOCs	8260	2-10.0 ug/l	30 % RPD	55-145 %	100%
Semi-Volatile Organics[1]					
SVOCs	8270	3-5.0 ug/l	30 % RPD	55-145 %	100%
Inorganics -- Metals[2,3]					
Barium	6010	5.0 ug/l	20% RPD	75-125%	100%
Cadmium	6010	3.0 ug/l	20% RPD	75-125%	100%
Chromium	6010	5.0 ug/l	20% RPD	75-125%	100%
Lead	6010	3.0 ug/l	20% RPD	75-125%	100%
Zinc	6010	2.0 ug/l	20% RPD	75-125%	100%
Air					
VOCs	TO-14\TO-15	0.006-0.02 ug/l	20% RPD	75-125%	100%
ethene, ethane	AM20GAX	5.0 ng/l	20% RPD	75-125%	100%
methane	AM20GAX	0.015 ug/l	20% RPD	75-125%	100%
Inorganics -- Non-Metals [2,4]					
Chloride	300	1,000 ug/l	20% RPD	75-125%	100%
Cyanide	335.3	5.0 ug/l	20% RPD	75-125%	100%
Fluoride	300	100 ug/l	20% RPD	75-125%	100%
Sulfate	300	1,000 ug/l	20% RPD	75-125%	100%
Physical Parameters[5]					
pH	--	--	0.1 SU	0.2 SU	100%
Temperature	--	--	0.1° C	0.2° C	100%
Conductivity	--	--	50 umhos/cm	2 umhos/cm	100%
Diss. Oxygen	--	--	0.1 mg/l	0.2 mg/l	100%
ORP	--	--	1 eV	1 eV	100%

RPD = Relative Percent Difference

[1]Test Methods for Evaluating Solid Wastes, USEPA SW-846, December 1996; Methods for Organic Chemical Analysi Municipal and Industrial Waste-Water, USEPA 600/4-82-057, July 1982; Guidelines Establishing Test Procedures f Analysis or Organic Pollutants Under the Clean Water Act, Federal Register, V. 49, no. 209; and USEPA Contract Laboratory Program (CLP) Statement of Work for Organics Analysis, October 1986 (revised December 1996).

[2]Test Methods for Evaluating Solid Wastes, USEPA SW-846, November 1986; Methods for Chemical Analysis of Wate and Wastes, USEPA-600/4-79-020 1979 (revised December 1996).

[3]Test Methods for Evaluating Solid Wastes, USEPA SW-846, November 1986; USEPA Contract Laboratory Program (Statement of Work for Inorganic Analysis, July 1987 (revised December 1996).

[4]Standard Methods for the Examination of Water and Wastewater, 17th Ed., 1989.

[5]Field parameters during purging measured with portable meter.

Table 7
Groundwater, Surface Water and Air Monitoring Locations
Former Monarch Chemicals Division Facility
61 Gates Avenue
Geneva, New York

Annual Surface Water Perimeter Sample Locations (1 Sampling/year)	Tri-Annual On-Site Groundwater Sample Locations (3 Sampling/year)	SVI Sample Locations (1 Sampling/year)
SW-1	MW-114	SS-1
SW-2	MW-115	SS-2
SW-3	MW-116	SS-3
	MW-117	SS-4
	MW-118	IA-1
	MW-119	IA-2
	MW-120	IA-3
	MW-121	IA-4
	MW-122	
	MW-123	

annual sampling conducted Mar-Apr each year
SVI sampling per respective Workplans

April 27, 2026

Table 7 has been replaced by Table II, Groundwater Monitoring Location, which is included in Sections H & A of New York Engineering and Geology LLP's letter dated April 27, 2026.

Table 8
TO-15 Analyte List for Air Monitoring Locations
Former Monarch Chemicals Division Facility
61 Gates Avenue
Geneva, New York

Compound	CAS No.
1,1-Dimethylhydrazine; C2H8N2	57-14-7
1,1,2-Trichloroethane; C2H3Cl3	79-00-5
1,2-Propyleneimine (2-methylaziridine); C3H7N	75-55-8
1,2,4-Trichlorobenzene; C6H3Cl3	120-82-1
1,2-Dibromo-3-chloropropane; C3H5Br2Cl	96-12-8
1,2-Epoxybutane (1,2-butylene oxide); C4H8O	106-88-7
1,3-Butadiene ; C4H6	106-99-0
1,3-Dichloropropene; C3H4Cl2 (cis)	542-75-6
1,3-Propane sultone; C3H6O3S	1120-71-4
1,4-Dioxane (1,4-diethylene oxide); C4H8O2	123-91-1
1,4-Dichlorobenzene (p); C6H4Cl2	106-46-7
2,2,4-Trimethyl pentane C8H18	540-84-1
2-Nitropropane; C3H7NO2	79-46-9
Acetaldehyde (ethanal); C2H4O	75-07-0
Acetonitrile (cyanomethane); C2H3N	75-05-8
Acetophenone; C8H8O	98-86-2
Acrolein (2-propenal); C3H4O	107-02-8
Acrylamide; C3H5NO	79-06-1
Acrylic acid; C3H4O2	79-10-7
Acrylonitrile (2-propenenitrile); C3H3N	107-13-1
Allyl chloride (3-chloropropene); C3H5Cl	107-05-1
Aniline (aminobenzene); C6H7N	62-53-3
Benzene; C6H6	71-43-2
Benzyl chloride (a-chlorotoluene); C7H7Cl	100-44-7
Beta-Propiolactone; C3H4O2	57-57-8
Bis(2-Chloroethyl)ether; C4H8Cl2O	111-44-4
Bis(chloromethyl) ether; C2H4Cl2O	542-88-1
Bromoform (tribromomethane); CHBr3	75-25-2
Carbon disulfide; CS2	75-15-0
Carbon tetrachloride; CCl4	56-23-5
Carbonyl sulfide; COS	463-58-1
Catechol (o-hydroxyphenol); C6H6O2	120-80-9
Catechol (o-hydroxyphenol); C6H6O2	120-80-9
Chloroacetic acid; C2H3ClO2	79-11-8
Chlorobenzene; C6H5Cl	108-90-7
Chloroform; CHCl3	67-66-3
Chloromethyl methyl ether; C2H5ClO	107-30-2
Chloroprene (2-chloro-1,3-butadiene); C4H5Cl	126-99-8
Cresylic acid (cresol isomer mixture);C7H8O	1319-77-3
Cumene (isopropylbenzene); C9Hl2	98-82-8
Diazomethane; CH2N2	334-88-3
Diethyl sulfate; C4H10O4S	64-67-5
Dimethyl sulfate; C2H6O4S	77-78-1
Dimethylcarbamyl chloride; C3H6ClNO	79-44-7
Epichlorohydrin (1-chloro-2,3-epoxy propane); C3H5ClO	106-89-8
Ethyl acrylate; C5H8O2	140-88-5
Ethyl carbamate (urethane); C3H7NO2	51-79-6
Ethyl chloride (chloroethane); C2H5Cl	75-00-3
Ethylbenzene; C8H10	100-41-4
Ethylene dibromide (1,2-dibromoethane); C2H4Br2	106-93-4
Ethylene dichloride (1,2-dichloroethane); C2H4Cl2	107-06-2
Ethylene oxide; C2H4O	75-21-8
Ethyleneimine (aziridine); C2H5N	151-56-4
Ethylidene dichloride (1,1-dichloroethane); C2H4Cl2	75-34-3

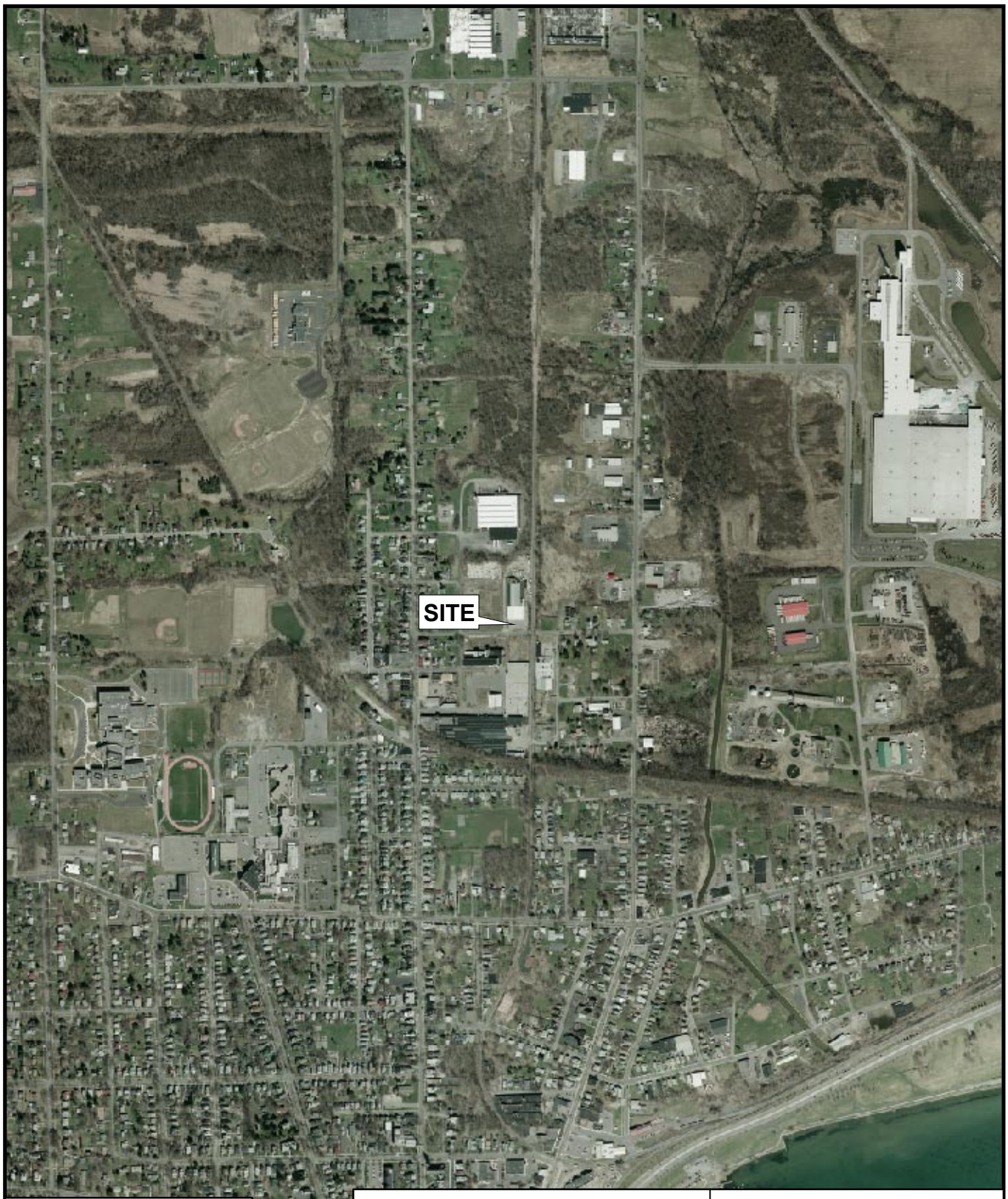
Table 8
TO-15 Analyte List for Air Monitoring Locations
Former Monarch Chemicals Division Facility
61 Gates Avenue
Geneva, New York

Compound	CAS No.
Formaldehyde ; CH2O	50-00-0
Hexachlorobutadiene; C4Cl6	87-68-3
Hexachloroethane; C2Cl6	67-72-1
Hexane; C6H14	110-54-3
Isophorone; C9H14O	78-59-1
l,l,2,2-Tetrachloroethane; C2H2Cl4	79-34-5
Methanol; CH4O	67-56-1
Methyl bromide (bromomethane); CH3Br	74-83-9
Methyl chloride (chloromethane); CH3Cl	74-87-3
Methyl chloroform (1,1,1-trichloroethane); C2H3Cl3	71-55-6
Methyl ethyl ketone (2-butanone); C4H8O	78-93-3
Methyl iodide (iodomethane); CH3I	74-88-4
Methyl isobutyl ketone (hexone); C6H12O	108-10-1
Methyl isocyanate; C2H3NO	624-83-9
Methyl methacrylate; C5H8O2	80-62-6
Methyl tert-butyl ether; C5H12O	1634-04-4
Methylene chloride; CH2Cl2	75-09-2
Methylhydrazine; CH6N2	60-34-4
m-Xylene; C8H10	108-38-3
N,N-Dimethylaniline; C8H11N	121-69-7
N,N-Dimethylformamide; C3H7NO	68-12-2
Nitrobenzene; C6H5NO2	98-95-3
N-Nitrosodimethylamine; C2H6N2O	62-75-9
N-Nitrosomorpholine; C4H8N2O2	59-89-2
N-Nitroso-N-methylurea; C2H5N3O2	684-93-5
o-Cresol; C7H8O	95-48-7
o-Xylene; C8H10	95-47-6
Phenol; C6H6O	108-95-2
Phosgene; CCl2O	75-44-5
Propionaldehyde; C2H5CHO	123-38-6
Propylene dichloride (1,2-dichloropropane); C3H6Cl2	78-87-5
Propylene oxide; C3H6O	75-56-9
p-Xylene; C8H10	106-42-3
Styrene oxide; C8H8O	96-09-3
Styrene; C8H8	100-42-5
Tetrachloroethylene; C2Cl4	127-18-4
Toluene; C7H8	108-88-3
Trichloroethylene; C2HCl3	79-01-6
Triethylamine; C6H15N	121-44-8
Vinyl acetate; C4H6O2	108-05-4
Vinyl bromide (bromoethene); C2H3Br	593-60-2
Vinyl chloride (chloroethene); C2H3Cl	75-01-4
Vinylidene chloride (1,1-dichloroethylene); C2H2Cl2	75-35-4
Xylenes (isomer & mixtures); C8H10	1330-20-7

Table 9
QA\QC Outline
Former Monarch Chemicals Division Facility
61 Gates Avenue
Geneva, New York

QA\QC Sample	Frequency	Comment
Field Duplicate	one in 20	Data shows precision of analytical scheme from sampling through analysis when compared with results of sample. This represents a blind QC sample to the laboratory. Collect an additional amount of sample.
Laboratory Duplicate	one in 20	Data shows precision of the analytical scheme within the laboratory. The difference between this precision and that of the field duplicate represents the precision of the analytical method.
Laboratory Spike	one in 20	Data shows how well the analysis of interest can be performed, and recovered from the sample matrix. Such information is useful when reported value is near an action level, but the sample exhibits poor recovery.
Matrix Spike	one in 20	Data shows precision of laboratory analysis when compared with results of sample. Collect an additional amount of sample for each analysis. Analyzed as unspiked sample.
Matrix Duplicate (inorganic)	one in 20	
Matrix Spike (inorganic)	one in 20	Data shows matrix effects from recovery of spiked analysis. Collect an additional amount for each analysis. Analyzed as a spike sample.
Matrix Spike/Matrix Spike Duplicate	one in 20	Data shows precision of analysis when compared with matrix spike duplicate and matrix effects from recovery of spiked analysis. Collect an additional amount for each analysis. Analyzed as a spike.
Field Blank/Equipment Blank	Once per event	Data demonstrates that sampling equipment was clean prior to use. Pass a sample of reagent water through collection device. Submit for analysis of analytes of concern.
Trip Blank	1 per cooler	Data demonstrates that sample was not contaminated with volatile organics by other samples in shipping container, laboratory or outside influences.
Background or Reference Sample	As required by the DQOs	Data provides baseline information to evaluate environmental impact.
Split Samples	Frequency dependant on State Sampling	Compare the quality of laboratory procedures of the permittee with State contracted laboratory procedures. Collect an additional amount of sample for each analysis.

Figures



Basemap Source: USGS Digital Orthoimagery of Ontario County, New York, April 2005.

0 300 600 Feet

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Date: 09/20/07

Figure 1
Site Location Map

Former Monarch Chemicals Facility
61 Gates Avenue
Geneva, New York

CCN International
Culvert Pipe
SW-1/SED-1
Drainage Ditch
SB-11
SB-12
Drainage Ditch
MW-115
MW-114
MW-123
MW-116
MW-117
Blacktop
MW-118
MW-122
SB-13
SW-2/SED-2
MW-119
Concrete Pad
MW-121
MW-120
Driveway
Building
SW-3/SED-3
Buried Culvert Pipe
Blacktop Parking Lot
Gates Avenue
Legend
Monitoring Well Location
Soil Boring Location
Surface Water and Sediment Sampling Location
Fence
Railroad
AST Containment Area
N
S
E
W
0
60
120
Feet
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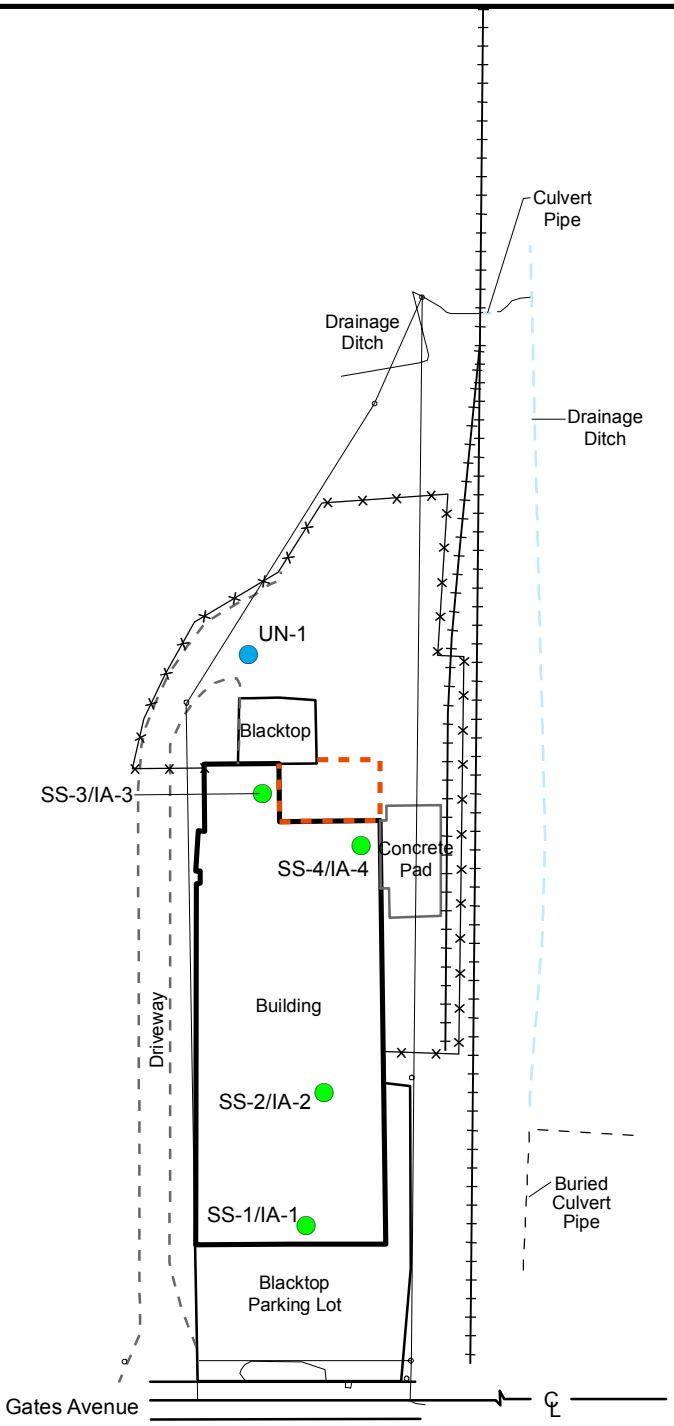
Date: 11/12/07
Figure 2
Groundwater Monitoring Well Network
Former Monarch Chemicals Facility
61 Gates Avenue
Geneva, New York



Legend

- Indoor and Sub-Slab SVI Sample
  SS = Subfloor Soil Vapor Sample
  IA = Indoor Air Sample
- Upwind Sample
- Fence
- Railroad
- AST Containment Area

0 60 120 Feet

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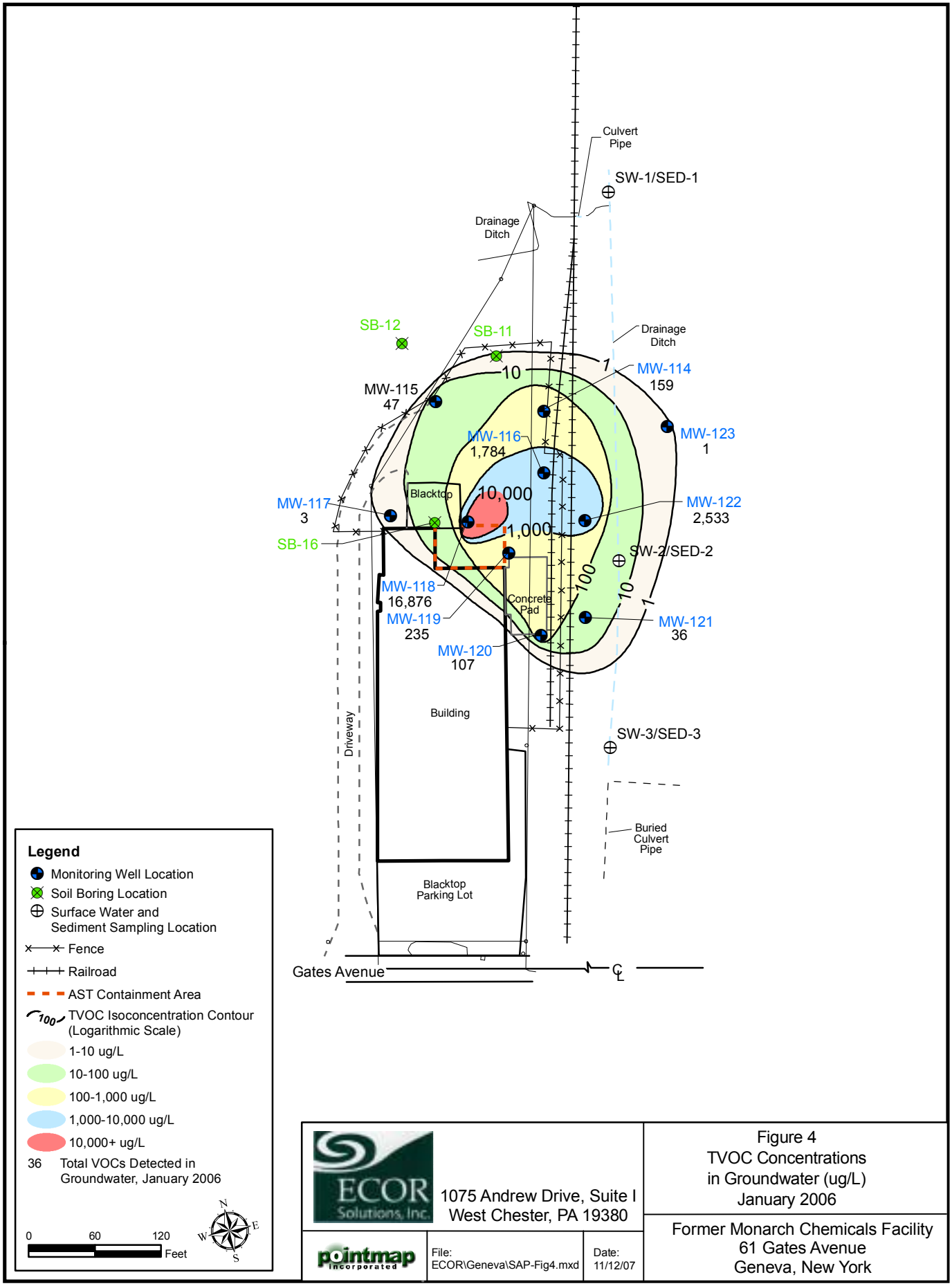
Date: 11/12/07

Figure 3
SVI Sampling Locations

Former Monarch Chemicals Facility
61 Gates Avenue
Geneva, New York



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Date: 11/12/07

Figure 4
TVOC Concentrations in Groundwater (ug/L)
January 2006

Former Monarch Chemicals Facility
61 Gates Avenue
Geneva, New York

Appendix A
EPA Test Method 8260 B

METHOD 8260B
VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/
MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

		Appropriate Preparation Technique[a]					
Compound	CAS No.[b]	5030/ 5035	5031	5032	5021	5041	Direct Inject.
Acetone	67-64-1	pp	c	c	nd	c	c
Acetonitrile	75-05-8	pp	c	nd	nd	nd	c
Acrolein (Propenal)	107-02-8	pp	c	c	nd	nd	c
Acrylonitrile	107-13-1	pp	c	c	nd	c	c
Allyl alcohol	107-18-6	ht	c	nd	nd	nd	c
Allyl chloride	107-05-1	c	nd	nd	nd	nd	c
Benzene	71-43-2	c	nd	c	c	c	c
Benzyl chloride	100-44-7	c	nd	nd	nd	nd	c
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd	nd	nd	nd	c
Bromoacetone	598-31-2	pp	nd	nd	nd	nd	c
Bromochloromethane	74-97-5	c	nd	c	c	c	c
Bromodichloromethane	75-27-4	c	nd	c	c	c	c
4-Bromofluorobenzene (surr)	460-00-4	c	nd	c	c	c	c
Bromoform	75-25-2	c	nd	c	c	c	c
Bromomethane	74-83-9	c	nd	c	c	c	c
n-Butanol	71-36-3	ht	c	nd	nd	nd	c
2-Butanone (MEK)	78-93-3	pp	c	c	nd	nd	c
t-Butyl alcohol	75-65-0	pp	c	nd	nd	nd	c
Carbon disulfide	75-15-0	pp	nd	c	nd	c	c
Carbon tetrachloride	56-23-5	c	nd	c	c	c	c
Chloral hydrate	302-17-0	pp	nd	nd	nd	nd	c
Chlorobenzene	108-90-7	c	nd	c	c	c	c
Chlorobenzene-d_5 (IS)		c	nd	c	c	c	c
Chlorodibromomethane	124-48-1	c	nd	c	nd	c	c
Chloroethane	75-00-3	c	nd	c	c	c	c
2-Chloroethanol	107-07-3	pp	nd	nd	nd	nd	c
2-Chloroethyl vinyl ether	110-75-8	c	nd	c	nd	nd	c
Chloroform	67-66-3	c	nd	c	c	c	c
Chloromethane	74-87-3	c	nd	c	c	c	c
Chloroprene	126-99-8	c	nd	nd	nd	nd	c
3-Chloropropionitrile	542-76-7	l	nd	nd	nd	nd	pc

(continued)

Compound	CAS No.[b]	Appropriate Preparation Technique[a] 5030/ 5035	5031	5032	5021	5041	Direct Inject.
Crotonaldehyde	4170-30-3	pp	c	nd	nd	nd	c
1,2-Dibromo-3-chloropropane	96-12-8	pp	nd	nd	c	nd	c
1,2-Dibromoethane	106-93-4	c	nd	nd	c	nd	c
Dibromomethane	74-95-3	c	nd	c	c	c	c
1,2-Dichlorobenzene	95-50-1	c	nd	nd	c	nd	c
1,3-Dichlorobenzene	541-73-1	c	nd	nd	c	nd	c
1,4-Dichlorobenzene	106-46-7	c	nd	nd	c	nd	c
1,4-Dichlorobenzene-d_4 (IS)		c	nd	nd	c	nd	c
cis-1,4-Dichloro-2-butene	1476-11-5	c	nd	c	nd	nd	c
trans-1,4-Dichloro-2-butene	110-57-6	pp	nd	c	nd	nd	c
Dichlorodifluoromethane	75-71-8	c	nd	c	c	nd	c
1,1-Dichloroethane	75-34-3	c	nd	c	c	c	c
1,2-Dichloroethane	107-06-2	c	nd	c	c	c	c
1,2-Dichloroethane-d_4 (surr)		c	nd	c	c	c	c
1,1-Dichloroethene	75-35-4	c	nd	c	c	c	c
trans-1,2-Dichloroethene	156-60-5	c	nd	c	c	c	c
1,2-Dichloropropane	78-87-5	c	nd	c	c	c	c
1,3-Dichloro-2-propanol	96-23-1	pp	nd	nd	nd	nd	c
cis-1,3-Dichloropropene	10061-01-5	c	nd	c	nd	c	c
trans-1,3-Dichloropropene	10061-02-6	c	nd	c	nd	c	c
1,2,3,4-Diepoxybutane	1464-53-5	c	nd	nd	nd	nd	c
Diethyl ether	60-29-7	c	nd	nd	nd	nd	c
1,4-Difluorobenzene (IS)	540-36-3	nd	nd	nd	nd	c	nd
1,4-Dioxane	123-91-1	pp	c	c	nd	nd	c
Epichlorohydrin	106-89-8	l	nd	nd	nd	nd	c
Ethanol	64-17-5	l	c	c	nd	nd	c
Ethyl acetate	141-78-6	l	c	nd	nd	nd	c
Ethylbenzene	100-41-4	c	nd	c	c	c	c
Ethylene oxide	75-21-8	pp	c	nd	nd	nd	c
Ethyl methacrylate	97-63-2	c	nd	c	nd	nd	c
Fluorobenzene (IS)	462-06-6	c	nd	nd	nd	nd	nd
Hexachlorobutadiene	87-68-3	c	nd	nd	c	nd	c
Hexachloroethane	67-72-1	l	nd	nd	nd	nd	c
2-Hexanone	591-78-6	pp	nd	c	nd	nd	c
2-Hydroxypropionitrile	78-97-7	l	nd	nd	nd	nd	pc
Iodomethane	74-88-4	c	nd	c	nd	c	c
Isobutyl alcohol	78-83-1	pp	c	nd	nd	nd	c
Isopropylbenzene	98-82-8	c	nd	nd	c	nd	c
Malononitrile	109-77-3	pp	nd	nd	nd	nd	c
Methacrylonitrile	126-98-7	pp	l	nd	nd	nd	c
Methanol	67-56-1	l	c	nd	nd	nd	c
Methylene chloride	75-09-2	c	nd	c	c	c	c
Methyl methacrylate	80-62-6	c	nd	nd	nd	nd	c
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	c	c	nd	nd	c
Naphthalene	91-20-3	c	nd	nd	c	nd	c

(continued)

Compound	CAS No.[b]	Appropriate Preparation Technique[a] 5030/ 5035	5031	5032	5021	5041	Direct Inject.
Nitrobenzene	98-95-3	c	nd	nd	nd	nd	c
2-Nitropropane	79-46-9	c	nd	nd	nd	nd	c
N-Nitroso-di-n-butylamine	924-16-3	pp	c	nd	nd	nd	c
Paraldehyde	123-63-7	pp	c	nd	nd	nd	c
Pentachloroethane	76-01-7	I	nd	nd	nd	nd	c
2-Pentanone	107-87-9	pp	c	nd	nd	nd	c
2-Picoline	109-06-8	pp	c	nd	nd	nd	c
1-Propanol	71-23-8	pp	c	nd	nd	nd	c
2-Propanol	67-63-0	pp	c	nd	nd	nd	c
Propargyl alcohol	107-19-7	pp	I	nd	nd	nd	c
β-Propiolactone	57-57-8	pp	nd	nd	nd	nd	c
Propionitrile (ethyl cyanide)	107-12-0	ht	c	nd	nd	nd	pc
n-Propylamine	107-10-8	c	nd	nd	nd	nd	c
Pyridine	110-86-1	I	c	nd	nd	nd	c
Styrene	100-42-5	c	nd	c	c	c	c
1,1,1,2-Tetrachloroethane	630-20-6	c	nd	nd	c	c	c
1,1,2,2-Tetrachloroethane	79-34-5	c	nd	c	c	c	c
Tetrachloroethene	127-18-4	c	nd	c	c	c	c
Toluene	108-88-3	c	nd	c	c	c	c
Toluene-d_8 (surr)	2037-26-5	c	nd	c	c	c	c
o-Toluidine	95-53-4	pp	c	nd	nd	nd	c
1,2,4-Trichlorobenzene	120-82-1	c	nd	nd	c	nd	c
1,1,1-Trichloroethane	71-55-6	c	nd	c	c	c	c
1,1,2-Trichloroethane	79-00-5	c	nd	c	c	c	c
Trichloroethene	79-01-6	c	nd	c	c	c	c
Trichlorofluoromethane	75-69-4	c	nd	c	c	c	c
1,2,3-Trichloropropane	96-18-4	c	nd	c	c	c	c
Vinyl acetate	108-05-4	c	nd	c	nd	nd	c
Vinyl chloride	75-01-4	c	nd	c	c	c	c
o-Xylene	95-47-6	c	nd	c	c	c	c
m-Xylene	108-38-3	c	nd	c	c	c	c
p-Xylene	106-42-3	c	nd	c	c	c	c

[a] See Sec. 1.2 for other appropriate sample preparation techniques
[b] Chemical Abstract Service Registry Number

c = Adequate response by this technique
ht = Method analyte only when purged at 80°C
nd = Not determined
I = Inappropriate technique for this analyte
pc = Poor chromatographic behavior
pp = Poor purging efficiency resulting in high Estimated Quantitation Limits
surr = Surrogate
IS = Internal Standard

1.2 There are various techniques by which these compounds may be introduced into the GC/MS system. The more common techniques are listed in the table above. Purge-and-trap, by Methods 5030 (aqueous samples) and 5035 (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. These include direct injection following dilution with hexadecane (Method 3585) for waste oil samples; automated static headspace by Method 5021 for solid samples; direct injection of an aqueous sample (concentration permitting) or injection of a sample concentrated by azeotropic distillation (Method 5031); and closed system vacuum distillation (Method 5032) for aqueous, solid, oil and tissue samples. For air samples, Method 5041 provides methodology for desorbing volatile organics from trapping media (Methods 0010, 0030, and 0031). In addition, direct analysis utilizing a sample loop is used for sub-sampling from Tedlar® bags (Method 0040). Method 5000 provides more general information on the selection of the appropriate introduction method.

1.3 Method 8260 can be used to quantitate most volatile organic compounds that have boiling points below 200°C. Volatile, water soluble compounds can be included in this analytical technique by the use of azeotropic distillation or closed-system vacuum distillation. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. See Tables 1 and 2 for analytes and retention times that have been evaluated on a purge-and-trap GC/MS system. Also, the method detection limits for 25-mL sample volumes are presented. The following compounds are also amenable to analysis by Method 8260:

Bromobenzene
n-Butylbenzene
sec-Butylbenzene
tert-Butylbenzene
Chloroacetonitrile
1-Chlorobutane
1-Chlorohexane
2-Chlorotoluene
4-Chlorotoluene
Dibromofluoromethane
cis-1,2-Dichloroethene
1,3-Dichloropropane
2,2-Dichloropropane
1,1-Dichloropropene
p-Isopropyltoluene
Methyl acrylate
Methyl-t-butyl ether
Pentafluorobenzene
n-Propylbenzene
1,2,3-Trichlorobenzene
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene

1.4 The estimated quantitation limit (EQL) of Method 8260 for an individual compound is somewhat instrument dependent and also dependent on the choice of sample preparation/introduction method. Using standard quadrapole instrumentation and the purge-and-trap technique, limits should be approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 µg/L for ground water (see Table 3). Somewhat lower limits may be achieved using an ion trap mass spectrometer or other instrumentation of improved design. No matter which instrument is used, EQLs will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.

1.5 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by other methods (see Sec. 1.2). The analytes are introduced directly to a wide-bore capillary column or cryofocussed on a capillary pre-column before being flash evaporated to a narrow-bore capillary for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC).

2.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. (Wide-bore capillary columns normally require a jet separator, whereas narrow-bore capillary columns may be directly interfaced to the ion source). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.3 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values without correcting for the blank results in what the laboratory feels is a false positive result for a sample, the laboratory should fully explained this in text accompanying the uncorrected data.

3.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

3.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique (Method 5021) or by Method 3820 (Hexadecane Extraction and Screening of Purgeable Organics).

3.4 Many analytes exhibit low purging efficiencies from a 25-mL sample. This often results in significant amounts of these analytes remaining in the sample purge vessel after analysis. After removal of the sample aliquot that was purged, and rinsing the purge vessel three times with organic-free water, the empty vessel should be subjected to a heated purge cycle prior to the analysis of another sample in the same purge vessel. This will reduce sample-to-sample carryover.

3.5 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

3.6 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

3.7 Use of sensitive mass spectrometers to achieve lower detection level will increase the potential to detect laboratory contaminants as interferences.

3.8 Direct injection - Some contamination may be eliminated by baking out the column between analyses. Changing the injector liner will reduce the potential for cross-contamination. A portion of the analytical column may need to be removed in the case of extreme contamination. The use of direct injection will result in the need for more frequent instrument maintenance.

3.9 If hexadecane is added to waste samples or petroleum samples that are analyzed, some chromatographic peaks will elute after the target analytes. The oven temperature program must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are volatilized.

4.0 APPARATUS AND MATERIALS

4.1 Purge-and-trap device for aqueous samples - Described in Method 5030.

4.2 Purge-and-trap device for solid samples - Described in Method 5035.

4.3 Automated static headspace device for solid samples - Described in Method 5021.

4.4 Azeotropic distillation apparatus for aqueous and solid samples - Described in Method 5031.

4.5 Vacuum distillation apparatus for aqueous, solid and tissue samples - Described in Method 5032.

4.6 Desorption device for air trapping media for air samples - Described in Method 5041.

4.7 Air sampling loop for sampling from Tedlar® bags for air samples - Described in Method 0040.

4.8 Injection port liners (HP Catalog #18740-80200, or equivalent) - modified for direct injection analysis by placing a 1-cm plug of glass wool approximately 50-60 mm down the length of the injection port towards the oven (see illustration below). A 0.53-mm ID column is mounted 1 cm into the liner from the oven side of the injection port, according to manufacturer's specifications.

4.9 Gas chromatography/mass spectrometer/data system

4.9.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

4.9.1.1 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

4.9.1.2 For some column configurations, the column oven must be cooled to less than 30°C, therefore, a subambient oven controller may be necessary.

4.9.1.3 The capillary column is either directly coupled to the source or interfaced through a jet separator, depending on the size of the capillary and the requirements of the GC/MS system.

4.9.1.4 Capillary pre-column interface - This device is the interface between the sample introduction device and the capillary gas chromatograph, and is necessary when using cryogenic cooling. The interface condenses the desorbed sample components and focuses them into a narrow band on an uncoated fused-silica capillary pre-column. When the interface is flash heated, the sample is transferred to the analytical capillary column.

4.9.1.5 During the cryofocussing step, the temperature of the fused-silica in the interface is maintained at -150°C under a stream of liquid nitrogen. After the desorption period, the interface must be capable of rapid heating to 250°C in 15 seconds or less to complete the transfer of analytes.

4.9.2 Gas chromatographic columns

4.9.2.1 Column 1 - 60 m x 0.75 mm ID capillary column coated with VOCOL (Supelco), 1.5-µm film thickness, or equivalent.

4.9.2.2 Column 2 - 30 - 75 m x 0.53 mm ID capillary column coated with DB-624 (J&W Scientific), Rt_x-502.2 (RESTEK), or VOCOL (Supelco), 3-µm film thickness, or equivalent.

4.9.2.3 Column 3 - 30 m x 0.25 - 0.32 mm ID capillary column coated with 95% dimethyl - 5% diphenyl polysiloxane (DB-5, Rt_x-5, SPB-5, or equivalent), 1-µm film thickness.

4.9.2.4 Column 4 - 60 m x 0.32 mm ID capillary column coated with DB-624 (J&W Scientific), 1.8-µm film thickness, or equivalent.

4.9.3 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 4 when 5-50 ng of the GC/MS tuning standard (BFB) are injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. Because ion-molecule reactions with water and methanol in an ion trap mass spectrometer may produce interferences that coelute with chloromethane and chloroethane, the base peak for both of these analytes will be at m/z 49. This ion should be used as the quantitation ion in this case. The mass spectrometer must be capable of producing a mass spectrum for BFB which meets all of the criteria in Table 3 when 5 or 50 ng are introduced.

4.9.4 GC/MS interface - Two alternatives may be used to interface the GC to the mass spectrometer.

4.9.4.1 Direct coupling, by inserting the column into the mass spectrometer, is generally used for 0.25 - 0.32 mm ID columns.

4.9.4.2 A jet separator, including an all-glass transfer line and glass enrichment device or split interface, is used with a 0.53 mm column.

4.9.4.3 Any enrichment device or transfer line may be used, if all of the performance specifications described in Sec. 8.0 (including acceptable calibration at 50 ng or less) can be achieved. GC/MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass may be deactivated by silanizing with dichlorodimethylsilane.

4.9.5 Data system - A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.10 Microsyringes - 10-, 25-, 100-, 250-, 500-, and 1,000-µL.

4.11 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.12 Syringes - 5-, 10-, or 25-mL, gas-tight with shutoff valve.

4.13 Balance - Analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1 g.

4.14 Glass scintillation vials - 20-mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps.

4.15 Vials - 2-mL, for GC autosampler.

4.16 Disposable pipets - Pasteur.

4.17 Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers.

4.18 Spatula - Stainless steel.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Methanol, CH_3OH - Pesticide quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents.

5.4 Reagent Hexadecane - Reagent hexadecane is defined as hexadecane in which interference is not observed at the method detection limit of compounds of interest. Hexadecane quality is demonstrated through the analysis of a solvent blank injected directly into the GC/MS. The results of such a blank analysis must demonstrate that all interfering volatiles have been removed from the hexadecane.

5.5 Polyethylene glycol, $H(OCH_2CH_2)_nOH$ - Free of interferences at the detection limit of the target analytes.

5.6 Hydrochloric acid (1:1 v/v), HCl - Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

5.7 Stock solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate.

5.7.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.0001 g.

5.7.2 Add the assayed reference material, as described below.

5.7.2.1 Liquids - Using a 100-µL syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.7.2.2 Gases - To prepare standards for any compounds that boil below 30°C (e.g., bromomethane, chloroethane, chloromethane, or vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0 mL mark. Lower the needle to

5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas will rapidly dissolve in the methanol. Standards may also be prepared by using a lecture bottle equipped with a septum. Attach PTFE tubing to the side arm relief valve and direct a gentle stream of gas into the methanol meniscus.

5.7.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in milligrams per liter (mg/L) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.7.4 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at -10°C or less or as recommended by the standard manufacturer. Standards should be returned to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

5.7.5 Frequency of Standard Preparation

5.7.5.1 Standards for the permanent gases should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for gases usually need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Dichlorodifluoromethane and dichloromethane will usually be the first compounds to evaporate from the standard and should, therefore, be monitored very closely when standards are held beyond one week.

5.7.5.2 Standards for the non-gases should be monitored frequently by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases usually need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether and styrene may need to be prepared more frequently.

5.7.6 Preparation of Calibration Standards From a Gas Mixture

An optional calibration procedure involves using a certified gaseous mixture daily, utilizing a commercially-available gaseous analyte mixture of bromomethane, chloromethane, chloroethane, vinyl chloride, dichloro-difluoromethane and trichlorofluoromethane in nitrogen. Mixtures of documented quality are stable for as long as six months without refrigeration. (VOA-CYL III, RESTEK Corporation, Cat. #20194 or equivalent).

5.7.6.1 Before removing the cylinder shipping cap, be sure the valve is completely closed (turn clockwise). The contents are under pressure and should be used in a well-ventilated area.

5.7.6.2 Wrap the pipe thread end of the Luer fitting with PTFE tape. Remove the shipping cap from the cylinder and replace it with the Luer fitting.

5.7.6.3 Transfer half the working standard containing other analytes, internal standards, and surrogates to the purge apparatus.

5.7.6.4 Purge the Luer fitting and stem on the gas cylinder prior to sample removal using the following sequence:

a) Connect either the 100-µL or 500-µL Luer syringe to the inlet fitting of the cylinder.

b) Make sure the on/off valve on the syringe is in the open position.

c) Slowly open the valve on the cylinder and withdraw a full syringe volume.

d) Be sure to close the valve on the cylinder before you withdraw the syringe from the Luer fitting.

e) Expel the gas from the syringe into a well-ventilated area.

f) Repeat steps a through e one more time to fully purge the fitting.

5.7.6.5 Once the fitting and stem have been purged, quickly withdraw the volume of gas you require using steps 5.6.6.1.4(a) through (d). Be sure to close the valve on the cylinder and syringe before you withdraw the syringe from the Luer fitting.

5.7.6.6 Open the syringe on/off valve for 5 seconds to reduce the syringe pressure to atmospheric pressure. The pressure in the cylinder is ~30 psi.

5.7.6.7 The gas mixture should be quickly transferred into the reagent water through the female Luer fitting located above the purging vessel.

NOTE: Make sure the arrow on the 4-way valve is pointing toward the female Luer fitting when transferring the sample from the syringe. Be sure to switch the 4-way valve back to the closed position before removing the syringe from the Luer fitting.

5.7.6.8 Transfer the remaining half of the working standard into the purging vessel. This procedure insures that the total volume of gas mix is flushed into the purging vessel, with none remaining in the valve or lines.

5.7.6.9 The concentration of each compound in the cylinder is typically 0.0025 µg/µL.

5.7.6.10 The following are the recommended gas volumes spiked into 5 mL of water to produce a typical 5-point calibration:

Gas Volume	Calibration Concentration
40 µL	20 µg/L
100 µL	50 µg/L
200 µL	100 µg/L
300 µL	150 µg/L
400 µL	200 µg/L

5.7.6.11 The following are the recommended gas volumes spiked into 25 mL of water to produce a typical 5-point calibration:

Gas Volume	Calibration Concentration
10 µL	1 µg/L
20 µL	2 µg/L
50 µL	5 µg/L
100 µL	10 µg/L
250 µL	25 µg/L

5.8 Secondary dilution standards - Using stock standard solutions, prepare secondary dilution standards in methanol containing the compounds of interest, either singly or mixed together. Secondary dilution standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Store in a vial with no headspace. Replace after one week. Secondary standards for gases should be replaced after one week unless the acceptability of the standard can be documented. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations. The analyst should also handle and store standards as stated in Sec. 5.7.4 and return them to the freezer as soon as standard mixing or diluting is completed to prevent the evaporation of volatile target compounds.

5.9 Surrogate standards - The recommended surrogates are toluene-d_8, 4-bromofluorobenzene, 1,2-dichloroethane-d_4, and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. A stock surrogate solution in methanol should be prepared as described above, and a surrogate standard spiking solution should be prepared from the stock at a concentration of 50-250 µg/10 mL, in methanol. Each sample undergoing GC/MS analysis must be spiked with 10 µL of the surrogate spiking solution prior to analysis. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute surrogate solutions may be required.

5.10 Internal standards - The recommended internal standards are fluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Prepare internal standard stock and secondary dilution standards in methanol using the procedures described in Secs. 5.7 and 5.8. It is recommended that the secondary dilution standard be prepared at a concentration of 25 mg/L of each internal standard compound. Addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be the equivalent of 50 µg/L. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute internal standard solutions may be required. Area counts of the internal standard peaks should be between 50-200% of the areas of the target analytes in the mid-point calibration analysis.

5.11 4-Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/µL of BFB in methanol should be prepared. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then a more dilute BFB standard solution may be required.

5.12 Calibration standards -There are two types of calibration standards used for this method: initial calibration standards and calibration verification standards. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.12.1 Initial calibration standards should be prepared at a minimum of five different concentrations from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in typical samples but should not exceed the working range of the GC/MS system. Initial calibration standards should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

5.12.2 Calibration verification standards should be prepared at a concentration near the mid-point of the initial calibration range from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. See Sec. 7.4 for guidance on calibration verification.

5.12.3 It is the intent of EPA that all target analytes for a particular analysis be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.12.4 The calibration standards must also contain the internal standards chosen for the analysis.

5.13 Matrix spiking and laboratory control sample (LCS) standards - Matrix spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigated. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The matrix spiking solution should contain compounds that are expected to be found in the types of samples to be analyzed.

5.13.1 Some permits may require the spiking of specific compounds of interest, especially if polar compounds are a concern, since the spiking compounds listed above would not be representative of such compounds. The standard should be prepared in methanol, with each compound present at a concentration of 250 µg/10.0 mL.

5.13.2 The spiking solutions should not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

5.13.3 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

5.14 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C or less, in amber bottles with PTFE-lined screw-caps.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

7.0 PROCEDURE

7.1 Various alternative methods are provided for sample introduction. All internal standards, surrogates, and matrix spiking compounds (when applicable) must be added to the samples before introduction into the GC/MS system. Consult the sample introduction method for the procedures by which to add such standards.

7.1.1 Direct injection - This includes: injection of an aqueous sample containing a very high concentration of analytes; injection of aqueous concentrates from Method 5031 (azeotropic distillation); and injection of a waste oil diluted 1:1 with hexadecane (Method 3585). Direct injection of aqueous samples (non-concentrated) has very limited applications. It is only used for the determination of volatiles at the toxicity characteristic (TC) regulatory limits or at concentrations in excess of 10,000 µg/L. It may also be used in conjunction with the test for ignitability in aqueous samples (along with Methods 1010 and 1020), to determine if alcohol is present at greater than 24%.

7.1.2 Purge-and-trap - This includes purge-and-trap for aqueous samples (Method 5030) and purge-and-trap for solid samples (Method 5035). Method 5035 also provides techniques for extraction of high concentration solid and oily waste samples by methanol (and other water-miscible solvents) with subsequent purge-and-trap from an aqueous matrix using Method 5030.

7.1.2.1 Traditionally, the purge-and-trap of aqueous samples is performed at ambient temperature, while purging of soil/solid samples is performed at 40°C, to improve purging efficiency.

7.1.2.2 Aqueous and soil/solid samples may also be purged at temperatures above those being recommended as long as all calibration standards, samples, and QC samples are purged at the same temperature, appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Purging of aqueous samples at elevated temperatures (e.g., 40°C) may improve the purging performance of many of the water soluble compounds which have poor purging efficiencies at ambient temperatures.

7.1.3 Vacuum distillation - this technique may be used for the introduction of volatile organics from aqueous, solid, or tissue samples (Method 5032) into the GC/MS system.

7.1.4 Automated static headspace - this technique may be used for the introduction of volatile organics from solid samples (Method 5021) into the GC/MS system.

7.1.5 Cartridge desorption - this technique may be for the introduction of volatile organics from sorbent cartridges (Method 5041) used in the sampling of air. The sorbent cartridges are from the volatile organics sampling train (VOST) or SMVOC (Method 0031).

7.2 Recommended chromatographic conditions

7.2.1 General conditions

Injector temperature:	200 - 225°C
Transfer line temperature:	250 - 300°C

7.2.2 Column 1 and Column 2 with cryogenic cooling (example chromatograms are presented in Figures 1 and 2)

Carrier gas (He) flow rate:	15 mL/min
Initial temperature:	10°C, hold for 5 minutes
Temperature program:	6°C/min to 70°C, then 15°C/min to 145°C
Final temperature:	145°C, hold until all expected compounds have eluted.

7.2.5 Direct injection - Column 2

Carrier gas (He) flow rate:	4 mL/min
Column:	J&W DB-624, 70m x 0.53 mm
Initial temperature:	40°C, hold for 3 minutes
Temperature program:	8°C/min
Final temperature:	260°C, hold until all expected compounds have eluted.
Column Bake out:	75 minutes
Injector temperature:	200-225°C
Transfer line temperature:	250-300°C

7.2.6 Direct split interface - Column 4

Carrier gas (He) flow rate:	1.5 mL/min
Initial temperature:	35°C, hold for 2 minutes
Temperature program:	4°C/min to 50°C 10°C/min to 220°C
Final temperature:	220°C, hold until all expected compounds have eluted
Split ratio:	100:1
Injector temperature:	125°C

7.3 Initial calibration

Establish the GC/MS operating conditions, using the following as guidance:

Mass range:	35 - 260 amu
Scan time:	0.6 - 2 sec/scan
Source temperature:	According to manufacturer's specifications
Ion trap only:	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

7.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 4 for a 5-50 ng injection or purging of 4-bromofluorobenzene (2-µL injection of the BFB standard). Analyses must not begin until these criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of BFB from the instrument manufacturer, the following approach has been shown to be useful: The mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of

BFB. Do not background subtract part of the BFB peak. Alternatively, the analyst may use other documented approaches suggested by the instrument manufacturer.

7.3.1.2 Use the BFB mass intensity criteria in Table 4 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g., CLP, Method 524.2, or manufacturer's instructions), provided that method performance is not adversely affected.

NOTE: All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

7.3.2 Set up the sample introduction system as outlined in the method of choice (see Sec. 7.1). A different calibration curve is necessary for each method because of the differences in conditions and equipment. A set of at least five different calibration standards is necessary (see Sec. 5.12 and Method 8000). Calibration must be performed using the sample introduction technique that will be used for samples. For Method 5030, the purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, develop the standard curve with whichever volume of sample that will be analyzed.

7.3.2.1 To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be prepared daily. Transfer 5.0 mL (or 25 mL if lower detection limits are required) of each standard to a gas tight syringe along with 10 µL of internal standard. Then transfer the contents to the appropriate device or syringe. Some of the introduction methods may have specific guidance on the volume of calibration standard and the way the standards are transferred to the device.

7.3.2.2 The internal standards selected in Sec. 5.10 should permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion.

7.3.2.3 To prepare a calibration standard for direct injection analysis of waste oil, dilute standards in hexadecane.

7.3.3 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. For direct injection, inject 1 - 2 µL into the GC/MS system. The injection volume will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water.

7.3.4 Tabulate the area response of the characteristic ions (see Table 5) against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured (Sec. 7.6.2).

The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate.
C_{is} = Concentration of the internal standard.

7.3.5 System performance check compounds (SPCCs) - Calculate the mean RF for each target analyte using the five RF values calculated from the initial (5-point) calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; chlorobenzene; and 1,1,2,2-tetrachloroethane. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Example problems include:

7.3.5.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

7.3.5.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

7.3.5.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.3.5.4 The minimum mean response factors for the volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

7.3.6 Calibration check compounds (CCCs)

7.3.6.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Sec. 7.0 of Method 8000.

7.3.6.2 Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration, as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

RF_i = RF for each of the calibration standards
$\overline{RF}$ = mean RF for each compound from the initial calibration
n = Number of calibration standards, e.g., 5

7.3.6.3 The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual Calibration Check Compound (CCC) must be equal or less than 30%. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

7.3.6.4 If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before reattempting calibration.

7.3.7 Evaluation of retention times - The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

7.3.8 Linearity of target analytes

7.3.8.1 If the RSD of any target analyte is 15% or less, then the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation (Sec. 7.7.2).

7.3.8.2 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 of Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

NOTE: Method 8000 specifies a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.8.3 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

NOTE: The 20% RSD criteria in Method 8000 pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject or introduce 5-50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 4 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration curve (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The BFB and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000 for method blank performance criteria.

7.4.4 System Performance Check Compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard must meet its minimum response factor (see Sec. 7.3.5.4). This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

7.4.5 Calibration Check Compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Sec. 7.3.6 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater

than 20% difference or drift), for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.5 GC/MS analysis of samples

7.5.1 It is highly recommended that the sample be screened to minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. Some of the screening options available utilizing SW-846 methods are automated headspace-GC/FID (Methods 5021/8015), automated headspace-GC/PID/ELCD (Methods 5021/8021), or waste dilution-GC/PID/ELCD (Methods 3585/8021) using the same type of capillary column. When used only for screening purposes, the quality control requirements in the methods above may be reduced as appropriate. Sample screening is particularly important when Method 8260 is used to achieve low detection levels.

7.5.2 BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

7.5.3 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice.

7.5.4 The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future analysis. Therefore, if only one VOA vial is provided to the laboratory, the analyst should prepare two aliquots for analysis at this time, to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. For aqueous samples, one 20-mL syringe could be used to hold two 5-mL aliquots. If the second aliquot is to be taken from the syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

7.5.5 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. If lower detection limits are required, use a 25-mL syringe, and adjust the final volume to 25.0 mL.

7.5.6 The following procedure may be used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delays, until the diluted sample is in a gas-tight syringe.

7.5.6.1 Dilutions may be made in volumetric flasks (10- to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.

7.5.6.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.

7.5.6.3 Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

7.5.6.4 Fill a 5-mL syringe with the diluted sample, as described in Sec. 7.5.5.

7.5.7 Compositing aqueous samples prior to GC/MS analysis

7.5.7.1 Add 5 mL of each sample (up to 5 samples are allowed) to a 25-mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be used, provided that equal volumes of each sample are composited.

7.5.7.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

7.5.7.3 Mix each vial well and draw out a 5-mL aliquot with the 25-mL syringe.

7.5.7.4 Once all the aliquots have been combined on the syringe, invert the syringe several times to mix the aliquots. Introduce the composited sample into the instrument, using the method of choice (see Sec. 7.1).

7.5.7.5 If less than five samples are used for compositing, a proportionately smaller syringe may be used, unless a 25-mL sample is to be purged.

7.5.8 Add 10 µL of the surrogate spiking solution and 10 µL of the internal standard spiking solution to each sample either manually or by autosampler. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 µL of the surrogate spiking solution to 5 mL of aqueous sample will yield a concentration of 50 µg/L of each surrogate standard. The addition of 10 µL of the surrogate spiking solution to 5 g of a non-aqueous sample will yield a concentration of 50 µg/kg of each standard.

If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute surrogate and internal standard solutions may be required.

7.5.9 Add 10 µL of the matrix spike solution (Sec. 5.13) to a 5-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each matrix spike standard.

7.5.9.1 Follow the same procedure in preparing the laboratory control sample (LCS), except the spike is added to a clean matrix. See Sec. 8.4 and Method 5000 for more guidance on the selection and preparation of the matrix spike and the LCS.

7.5.9.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking and LCS solutions may be required.

7.5.10 Analyze the sample following the procedure in the introduction method of choice.

7.5.10.1 For direct injection, inject 1 to 2 µL into the GC/MS system. The volume limitation will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water (if an aqueous sample is being analyzed).

7.5.10.2 The concentration of the internal standards, surrogates, and matrix spiking standards (if any) added to the injection aliquot must be adjusted to provide the same concentration in the 1-2 µL injection as would be introduced into the GC/MS by purging a 5-mL aliquot.

NOTE: It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

7.5.11 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion.

7.5.11.1 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

7.5.11.2 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.5.12 The use of selected ion monitoring (SIM) is acceptable in situations requiring detection limits below the normal range of full EI spectra. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

7.6 Qualitative analysis

7.6.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

7.6.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.6.1.2 The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

7.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library

searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:

(1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of that of a given analyte.

7.7.2 If the RSD of a compound's response factors is 15% or less, then the concentration in the extract may be determined using the average response factor ($\overline{RF}$) from initial calibration data (7.3.6). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Methods 3500 and 5000. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8260:

8.2.1 The GC/MS system must be tuned to meet the BFB specifications in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the SPCC criteria described in Sec. 7.4.4 and the CCC criteria in Sec. 7.4.5, each 12 hours.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

9.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

9.2 This method has been tested using purge-and-trap (Method 5030) in a single laboratory using spiked water. Using a wide-bore capillary column, water was spiked at concentrations between 0.5 and 10 µg/L. Single laboratory accuracy and precision data are presented for the method analytes in Table 6. Calculated MDLs are presented in Table 1.

9.3 The method was tested using purge-and-trap (Method 5030) with water spiked at 0.1 to 0.5 µg/L and analyzed on a cryofocussed narrow-bore column. The accuracy and precision data for these compounds are presented in Table 7. MDL values were also calculated from these data and are presented in Table 2.

9.4 Direct injection (Method 3585) has been used for the analysis of waste motor oil samples using a wide-bore column. Single laboratory precision and accuracy data are presented in Tables 10 and 11 for TCLP volatiles in oil. The performance data were developed by spiking and analyzing seven replicates each of new and used oil. The oils were spiked at the TCLP regulatory concentrations for most analytes, except for the alcohols, ketones, ethyl acetate and chlorobenzene which are spiked at 5 ppm, well below the regulatory concentrations. Prior to spiking, the new oil (an SAE 30-weight motor oil) was heated at 80°C overnight to remove volatiles. The used oil (a mixture of used oil drained from passenger automobiles) was not heated and was contaminated with 20 - 300 ppm of BTEX compounds and isobutanol. These contaminants contributed to the extremely high recoveries of the BTEX compounds in the used oil. Therefore, the data from the deuterated analogs of these analytes represent more typical recovery values.

9.5 Single laboratory accuracy and precision data were obtained for the Method 5035 analytes in three soil matrices: sand; a soil collected 10 feet below the surface of a hazardous landfill, called C-Horizon; and a surface garden soil. Sample preparation was by Method 5035. Each

sample was fortified with the analytes at a concentration of 4 µg/kg. These data are listed in Tables 17, 18, and 19. All data were calculated using fluorobenzene as the internal standard added to the soil sample prior to extraction. This causes some of the results to be greater than 100% recovery because the precision of results is sometimes as great as 28%.

9.5.1 In general, the recoveries of the analytes from the sand matrix are the highest, the C-Horizon soil results are somewhat less, and the surface garden soil recoveries are the lowest. This is due to the greater adsorptive capacity of the garden soil. This illustrates the necessity of analyzing matrix spike samples to assess the degree of matrix effects.

9.5.2 The recoveries of some of the gases, or very volatile compounds, such as vinyl chloride, trichlorofluoromethane, and 1,1-dichloroethene, are somewhat greater than 100%. This is due to the difficulty encountered in fortifying the soil with these compounds, allowing an equilibration period, then extracting them with a high degree of precision. Also, the garden soil results in Table 19 include some extraordinarily high recoveries for some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection, and to the fact that no background was subtracted.

9.6 Performance data for nonpurgeable volatiles using azeotropic distillation (Method 5031) are included in Tables 12 to 16.

9.7 Performance data for volatiles prepared using vacuum distillation (Method 5032) in soil, water, oil and fish tissue matrices are included in Tables 20 to 27.

9.8 Single laboratory accuracy and precision data were obtained for the Method 5021 analytes in two soil matrices: sand and a surface garden soil. Replicate samples were fortified with the analytes at concentrations of 10 µg/kg. These data are listed in Table 30. All data were calculated using the internal standards listed for each analyte in Table 28. The recommended internal standards were selected because they generated the best accuracy and precision data for the analyte in both types of soil.

9.8.1 If a detector other than an MS is used for analysis, consideration must be given to the choice of internal standards and surrogates. They must not coelute with any other analyte and must have similar properties to the analytes. The recoveries of the analytes are 50% or higher for each matrix studied. The recoveries of the gases or very volatile compounds are greater than 100% in some cases. Also, results include high recoveries of some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection.

9.8.2 The method detection limits using Method 5021 listed in Table 29 were calculated from results of seven replicate analyses of the sand matrix. Sand was chosen because it demonstrated the least degree of matrix effect of the soils studied. These MDLs were calculated utilizing the procedure described in Chapter One and are intended to be a general indication of the capabilities of the method.

9.9 The MDL concentrations listed in Table 31 were determined using Method 5041 in conjunction with Method 8260. They were obtained using cleaned blank VOST tubes and reagent water. Similar results have been achieved with field samples. The MDL actually achieved in a given analysis will vary depending upon instrument sensitivity and the effects of the matrix. Preliminary spiking studies indicate that under the test conditions, the MDLs for spiked compounds in extremely complex matrices may be larger by a factor of 500 - 1000.

9.10 The EQL of sample taken by Method 0040 and analyzed by Method 8260 is estimated to be in the range of 0.03 to 0.9 ppm (See Table 33). Matrix effects may cause the individual compound detection limits to be higher.

10.0 REFERENCES

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TABLE 1

CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL)
FOR VOLATILE ORGANIC COMPOUNDS ON WIDE-BORE CAPILLARY COLUMNS

Compound	Retention Time (minutes)			MDL[d]
	Column 1[a]	Column 2[b]	Column 2'[c]	(µg/L)
Dichlorodifluoromethane	1.35	0.70	3.13	0.10
Chloromethane	1.49	0.73	3.40	0.13
Vinyl Chloride	1.56	0.79	3.93	0.17
Bromomethane	2.19	0.96	4.80	0.11
Chloroethane	2.21	1.02	--	0.10
Trichlorofluoromethane	2.42	1.19	6.20	0.08
Acrolein	3.19			
Iodomethane	3.56			
Acetonitrile	4.11			
Carbon disulfide	4.11			
Allyl chloride	4.11			
Methylene chloride	4.40	2.06	9.27	0.03
1,1-Dichloroethene	4.57	1.57	7.83	0.12
Acetone	4.57			
trans-1,2-Dichloroethene	4.57	2.36	9.90	0.06
Acrylonitrile	5.00			
1,1-Dichloroethane	6.14	2.93	10.80	0.04
Vinyl acetate	6.43			
2,2-Dichloropropane	8.10	3.80	11.87	0.35
2-Butanone	--			
cis-1,2-Dichloroethene	8.25	3.90	11.93	0.12
Propionitrile	8.51			
Chloroform	9.01	4.80	12.60	0.03
Bromochloromethane	--	4.38	12.37	0.04
Methacrylonitrile	9.19			
1,1,1-Trichloroethane	10.18	4.84	12.83	0.08
Carbon tetrachloride	11.02	5.26	13.17	0.21
1,1-Dichloropropene	--	5.29	13.10	0.10
Benzene	11.50	5.67	13.50	0.04
1,2-Dichloroethane	12.09	5.83	13.63	0.06
Trichloroethene	14.03	7.27	14.80	0.19
1,2-Dichloropropane	14.51	7.66	15.20	0.04
Bromodichloromethane	15.39	8.49	15.80	0.08
Dibromomethane	15.43	7.93	5.43	0.24
Methyl methacrylate	15.50			
1,4-Dioxane	16.17			
2-Chloroethyl vinyl ether	--			
4-Methyl-2-pentanone	17.32			
trans-1,3-Dichloropropene	17.47	--	16.70	--
Toluene	18.29	10.00	17.40	0.11
cis-1,3-Dichloropropene	19.38	--	17.90	--

TABLE 1 (cont.)

Compound	Retention Time (minutes)			MDL[d]
	Column 1[a]	Column 2[b]	Column 2"[c]	(µg/L)
1,1,2-Trichloroethane	19.59	11.05	18.30	0.10
Ethyl methacrylate	20.01			
2-Hexanone	20.30			
Tetrachloroethene	20.26	11.15	18.60	0.14
1,3-Dichloropropane	20.51	11.31	18.70	0.04
Dibromochloromethane	21.19	11.85	19.20	0.05
1,2-Dibromoethane	21.52	11.83	19.40	0.06
1-Chlorohexane	--	13.29	--	0.05
Chlorobenzene	23.17	13.01	20.67	0.04
1,1,1,2-Tetrachloroethane	23.36	13.33	20.87	0.05
Ethylbenzene	23.38	13.39	21.00	0.06
p-Xylene	23.54	13.69	21.30	0.13
m-Xylene	23.54	13.68	21.37	0.05
o-Xylene	25.16	14.52	22.27	0.11
Styrene	25.30	14.60	22.40	0.04
Bromoform	26.23	14.88	22.77	0.12
Isopropylbenzene (Cumene)	26.37	15.46	23.30	0.15
cis-1,4-Dichloro-2-butene	27.12			
1,1,2,2-Tetrachloroethane	27.29	16.35	24.07	0.04
Bromobenzene	27.46	15.86	24.00	0.03
1,2,3-Trichloropropane	27.55	16.23	24.13	0.32
n-Propylbenzene	27.58	16.41	24.33	0.04
2-Chlorotoluene	28.19	16.42	24.53	0.04
trans-1,4-Dichloro-2-butene	28.26			
1,3,5-Trimethylbenzene	28.31	16.90	24.83	0.05
4-Chlorotoluene	28.33	16.72	24.77	0.06
Pentachloroethane	29.41			
1,2,4-Trimethylbenzene	29.47	17.70	31.50	0.13
sec-Butylbenzene	30.25	18.09	26.13	0.13
tert-Butylbenzene	30.59	17.57	26.60	0.14
p-Isopropyltoluene	30.59	18.52	26.50	0.12
1,3-Dichlorobenzene	30.56	18.14	26.37	0.12
1,4-Dichlorobenzene	31.22	18.39	26.60	0.03
Benzyl chloride	32.00			
n-Butylbenzene	32.23	19.49	27.32	0.11
1,2-Dichlorobenzene	32.31	19.17	27.43	0.03
1,2-Dibromo-3-chloropropane	35.30	21.08	--	0.26
1,2,4-Trichlorobenzene	38.19	23.08	31.50	0.04
Hexachlorobutadiene	38.57	23.68	32.07	0.11
Naphthalene	39.05	23.52	32.20	0.04
1,2,3-Trichlorobenzene	40.01	24.18	32.97	0.03

TABLE 1 (cont.)

Compound	Retention Time (minutes) Column 1[a]	Column 2[b]	Column 2"[c]	MDL[d] (µg/L)
INTERNAL STANDARDS/SURROGATES				
1,4-Difluorobenzene	13.26			
Chlorobenzene-d_5	23.10			
1,4-Dichlorobenzene-d_4	31.16			
4-Bromofluorobenzene	27.83	15.71	23.63	
1,2-Dichlorobenzene-d_4	32.30	19.08	27.25	
Dichloroethane-d_4	12.08			
Dibromofluoromethane	--			
Toluene-d_8	18.27			
Pentafluorobenzene	--			
Fluorobenzene	13.00	6.27	14.06	

[a] Column 1 - 60 meter x 0.75 mm ID VOCOL capillary. Hold at 10°C for 8 minutes, then program to 180°C at 4°C/min.

[b] Column 2 - 30 meter x 0.53 mm ID DB-624 wide-bore capillary using cryogenic oven. Hold at 10°C for 5 minutes, then program to 160°C at 6°C/min.

[c] Column 2" - 30 meter x 0.53 mm ID DB-624 wide-bore capillary, cooling GC oven to ambient temperatures. Hold at 10°C for 6 minutes, program to 70°C at 10 °C/min, program to 120°C at 5°C/min, then program to 180°C at 8°C/min.

[d] MDL based on a 25-mL sample volume.

TABLE 2

CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL)
FOR VOLATILE ORGANIC COMPOUNDS ON NARROW-BORE CAPILLARY COLUMNS

Compound	Retention Time (minutes) Column 3[a]	MDL[b] (µg/L)
Dichlorodifluoromethane	0.88	0.11
Chloromethane	0.97	0.05
Vinyl chloride	1.04	0.04
Bromomethane	1.29	0.03
1,1-Dichloroethane	4.03	0.03
cis-1,2-Dichloroethene	5.07	0.06
2,2-Dichloropropane	5.31	0.08
Chloroform	5.55	0.04
Bromochloromethane	5.63	0.09
1,1,1-Trichloroethane	6.76	0.04
1,2-Dichloroethane	7.00	0.02
1,1-Dichloropropene	7.16	0.12
Carbon tetrachloride	7.41	0.02
Benzene	7.41	0.03
1,2-Dichloropropane	8.94	0.02
Trichloroethene	9.02	0.02
Dibromomethane	9.09	0.01
Bromodichloromethane	9.34	0.03
Toluene	11.51	0.08
1,1,2-Trichloroethane	11.99	0.08
1,3-Dichloropropane	12.48	0.08
Dibromochloromethane	12.80	0.07
Tetrachloroethene	13.20	0.05
1,2-Dibromoethane	13.60	0.10
Chlorobenzene	14.33	0.03
1,1,1,2-Tetrachloroethane	14.73	0.07
Ethylbenzene	14.73	0.03
p-Xylene	15.30	0.06
m-Xylene	15.30	0.03
Bromoform	15.70	0.20
o-Xylene	15.78	0.06
Styrene	15.78	0.27
1,1,2,2-Tetrachloroethane	15.78	0.20
1,2,3-Trichloropropane	16.26	0.09
Isopropylbenzene	16.42	0.10
Bromobenzene	16.42	0.11
2-Chlorotoluene	16.74	0.08
n-Propylbenzene	16.82	0.10
4-Chlorotoluene	16.82	0.06

TABLE 2 (cont.)

Compound	Retention Time (minutes) Column 3[a]	MDL[b] (µg/L)
1,3,5-Trimethylbenzene	16.99	0.06
tert-Butylbenzene	17.31	0.33
1,2,4-Trimethylbenzene	17.31	0.09
sec-Butylbenzene	17.47	0.12
1,3-Dichlorobenzene	17.47	0.05
p-Isopropyltoluene	17.63	0.26
1,4-Dichlorobenzene	17.63	0.04
1,2-Dichlorobenzene	17.79	0.05
n-Butylbenzene	17.95	0.10
1,2-Dibromo-3-chloropropane	18.03	0.50
1,2,4-Trichlorobenzene	18.84	0.20
Naphthalene	19.07	0.10
Hexachlorobutadiene	19.24	0.10
1,2,3-Trichlorobenzene	19.24	0.14

[a] Column 3 - 30 meter x 0.32 mm ID DB-5 capillary with 1 µm film thickness.

[b] MDL based on a 25-mL sample volume.

TABLE 3

ESTIMATED QUANTITATION LIMITS FOR VOLATILE ANALYTES[a]

Estimated Quantitation Limits		
5-mL Ground Water Purge (µg/L)	25-mL Ground water Purge (µg/L)	Low Soil/Sediment[b] µg/kg
5	1	5

[a] Estimated Quantitation Limit (EQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following footnote for further guidance on matrix-dependent EQLs.

[b] EQLs listed for soil/sediment are based on wet weight. Normally data are reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

Other Matrices	Factor[c]
Water miscible liquid waste	50
High concentration soil and sludge	125
Non-water miscible waste	500

[c] EQL = [EQL for low soil sediment (Table 3)] x [Factor].

For non-aqueous samples, the factor is on a wet-weight basis.

TABLE 4

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA[a]

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

[a] Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers' instructions), provided that method performance is not adversely affected.

TABLE 5

CHARACTERISTIC MASSES (m/z) FOR PURGEABLE ORGANIC COMPOUNDS

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,2-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene-d_4	152	115, 150
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53
β-Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Internal Standards/Surrogates:		
Benzene-d_6	84	83
Bromobenzene-d_5	82	162
Bromochloromethane-d_2	51	131
1,4-Difluorobenzene	114	
Chlorobenzene-d_5	117	
1,4-Dichlorobenzene-d_4	152	115, 150
1,1,2-Trichloroethane-d_3	100	
4-Bromofluorobenzene	95	174, 176
Chloroform-d_1	84	
Dibromofluoromethane	113	

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Internal Standards/Surrogates		
Dichloroethane-d_4	102	
Toluene-d_8	98	
Pentafluorobenzene	168	
Fluorobenzene	96	77

* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).

TABLE 6

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A WIDE-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Benzene	0.1 - 10	31	97	6.5	5.7
Bromobenzene	0.1 - 10	30	100	5.5	5.5
Bromochloromethane	0.5 - 10	24	90	5.7	6.4
Bromodichloromethane	0.1 - 10	30	95	5.7	6.1
Bromoform	0.5 - 10	18	101	6.4	6.3
Bromomethane	0.5 - 10	18	95	7.8	8.2
n-Butylbenzene	0.5 - 10	18	100	7.6	7.6
sec-Butylbenzene	0.5 - 10	16	100	7.6	7.6
tert-Butylbenzene	0.5 - 10	18	102	7.4	7.3
Carbon tetrachloride	0.5 - 10	24	84	7.4	8.8
Chlorobenzene	0.1 - 10	31	98	5.8	5.9
Chloroethane	0.5 - 10	24	89	8.0	9.0
Chloroform	0.5 - 10	24	90	5.5	6.1
Chloromethane	0.5 - 10	23	93	8.3	8.9
2-Chlorotoluene	0.1 - 10	31	90	5.6	6.2
4-Chlorotoluene	0.1 - 10	31	99	8.2	8.3
1,2-Dibromo-3-Chloropropane	0.5 - 10	24	83	16.6	19.9
Dibromochloromethane	0.1 - 10	31	92	6.5	7.0
1,2-Dibromoethane	0.5 - 10	24	102	4.0	3.9
Dibromomethane	0.5 - 10	24	100	5.6	5.6
1,2-Dichlorobenzene	0.1 - 10	31	93	5.8	6.2
1,3-Dichlorobenzene	0.5 - 10	24	99	6.8	6.9
1,4-Dichlorobenzene	0.2 - 20	31	103	6.6	6.4
Dichlorodifluoromethane	0.5 - 10	18	90	6.9	7.7
1,1-Dichlorobenzene	0.5 - 10	24	96	5.1	5.3
1,2-Dichlorobenzene	0.1 - 10	31	95	5.1	5.4
1,1-Dichloroethene	0.1 - 10	34	94	6.3	6.7
cis-1,2-Dichloroethene	0.5 - 10	18	101	6.7	6.7
trans-1,2-Dichloroethene	0.1 - 10	30	93	5.2	5.6
1,2-Dichloropropane	0.1 - 10	30	97	5.9	6.1
1,3-Dichloropropane	0.1 - 10	31	96	5.7	6.0
2,2-Dichloropropane	0.5 - 10	12	86	14.6	16.9
1,1-Dichloropropene	0.5 - 10	18	98	8.7	8.9
Ethylbenzene	0.1 - 10	31	99	8.4	8.6
Hexachlorobutadiene	0.5 - 10	18	100	6.8	6.8
Isopropylbenzene	0.5 - 10	16	101	7.7	7.6
p-Isopropyltoluene	0.1 - 10	23	99	6.7	6.7
Methylene chloride	0.1 - 10	30	95	5.0	5.3

TABLE 6 (cont.)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Naphthalene	0.1 -100	31	104	8.6	8.2
n-Propylbenzene	0.1 - 10	31	100	5.8	5.8
Styrene	0.1 -100	39	102	7.3	7.2
1,1,1,2-Tetrachloroethane	0.5 - 10	24	90	6.1	6.8
1,1,2,2-Tetrachloroethane	0.1 - 10	30	91	5.7	6.3
Tetrachloroethene	0.5 - 10	24	89	6.0	6.8
Toluene	0.5 - 10	18	102	8.1	8.0
1,2,3-Trichlorobenzene	0.5 - 10	18	109	9.4	8.6
1,2,4-Trichlorobenzene	0.5 - 10	18	108	9.0	8.3
1,1,1-Trichloroethane	0.5 - 10	18	98	7.9	8.1
1,1,2-Trichloroethane	0.5 - 10	18	104	7.6	7.3
Trichloroethene	0.5 - 10	24	90	6.5	7.3
Trichlorofluoromethane	0.5 - 10	24	89	7.2	8.1
1,2,3-Trichloropropane	0.5 - 10	16	108	15.6	14.4
1,2,4-Trimethylbenzene	0.5 - 10	18	99	8.0	8.1
1,3,5-Trimethylbenzene	0.5 - 10	23	92	6.8	7.4
Vinyl chloride	0.5 - 10	18	98	6.5	6.7
o-Xylene	0.1 - 31	18	103	7.4	7.2
m-Xylene	0.1 - 10	31	97	6.3	6.5
p-Xylene	0.5 - 10	18	104	8.0	7.7

[a] Recoveries were calculated using internal standard method. The internal standard was fluorobenzene.

[b] Standard deviation was calculated by pooling data from three concentrations.

TABLE 7

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Benzene	0.1	7	99	6.2	6.3
Bromobenzene	0.5	7	97	7.4	7.6
Bromochloromethane	0.5	7	97	5.8	6.0
Bromodichloromethane	0.1	7	100	4.6	4.6
Bromoform	0.5	7	101	5.4	5.3
Bromomethane	0.5	7	99	7.1	7.2
n-Butylbenzene	0.5	7	94	6.0	6.4
sec-Butylbenzene	0.5	7	110	7.1	6.5
tert-Butylbenzene	0.5	7	110	2.5	2.3
Carbon tetrachloride	0.1	7	108	6.8	6.3
Chlorobenzene	0.1	7	91	5.8	6.4
Chloroethane	0.1	7	100	5.8	5.8
Chloroform	0.1	7	105	3.2	3.0
Chloromethane	0.5	7	101	4.7	4.7
2-Chlorotoluene	0.5	7	99	4.6	4.6
4-Chlorotoluene	0.5	7	96	7.0	7.3
1,2-Dibromo-3-chloropropane	0.5	7	92	10.0	10.9
Dibromochloromethane	0.1	7	99	5.6	5.7
1,2-Dibromoethane	0.5	7	97	5.6	5.8
Dibromomethane	0.5	7	93	5.6	6.0
1,2-Dichlorobenzene	0.1	7	97	3.5	3.6
1,3-Dichlorobenzene	0.1	7	101	6.0	5.9
1,4-Dichlorobenzene	0.1	7	106	6.5	6.1
Dichlorodifluoromethane	0.1	7	99	8.8	8.9
1,1-Dichloroethane	0.5	7	98	6.2	6.3
1,2-Dichloroethane	0.1	7	100	6.3	6.3
1,1-Dichloroethene	0.1	7	95	9.0	9.5
cis-1,2-Dichloroethene	0.1	7	100	3.5	3.7
trans-1,2-Dichloroethene	0.1	7	98	7.2	7.3
1,2-Dichloropropane	0.5	7	96	6.0	6.3
1,3-Dichloropropane	0.5	7	99	5.8	5.9
2,2-Dichloropropane	0.5	7	99	4.9	4.9
1,1-Dichloropropene	0.5	7	102	7.4	7.3
Ethylbenzene	0.5	7	99	5.2	5.3
Hexachlorobutadiene	0.5	7	100	6.7	6.7
Isopropylbenzene	0.5	7	102	6.4	6.3
p-Isopropyltoluene	0.5	7	113	13.0	11.5
Methylene chloride	0.5	7	97	13.0	13.4
Naphthalene	0.5	7	98	7.2	7.3

TABLE 7 (cont.)

Compound	Conc. (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
n-Propylbenzene	0.5	7	99	6.6	6.7
Styrene	0.5	7	96	19.0	19.8
1,1,1,2-Tetrachloroethane	0.5	7	100	4.7	4.7
1,1,2,2-Tetrachloroethane	0.5	7	100	12.0	12.0
Tetrachloroethene	0.1	7	96	5.0	5.2
Toluene	0.5	7	100	5.9	5.9
1,2,3-Trichlorobenzene	0.5	7	102	8.9	8.7
1,2,4-Trichlorobenzene	0.5	7	91	16.0	17.6
1,1,1-Trichloroethane	0.5	7	100	4.0	4.0
1,1,2-Trichloroethane	0.5	7	102	4.9	4.8
Trichloroethene	0.1	7	104	2.0	1.9
Trichlorofluoromethane	0.1	7	97	4.6	4.7
1,2,3-Trichloropropane	0.5	7	96	6.5	6.8
1,2,4-Trimethylbenzene	0.5	7	96	6.5	6.8
1,3,5-Trimethylbenzene	0.5	7	101	4.2	4.2
Vinyl chloride	0.1	7	104	0.2	0.2
o-Xylene	0.5	7	106	7.5	7.1
m-Xylene	0.5	7	106	4.6	4.3
p-Xylene	0.5	7	97	6.1	6.3

[a] Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

TABLE 8

SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Water	Soil/Sediment
4-Bromofluorobenzene[a]	86-115	74-121
Dibromofluoromethane[a]	86-118	80-120
Toluene-d_8[a]	88-110	81-117
Dichloroethane-d_4[a]	80-120	80-120

[a] Single laboratory data, for guidance only.

TABLE 9

QUANTITY OF EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SAMPLES

Approximate Concentration Range (µg/kg)	Volume of Extract[a]
500 - 10,000	100 µL
1,000 - 20,000	50 µL
5,000 - 100,000	10 µL
25,000 - 500,000	100 µL of 1/50 dilution[b]

Calculate appropriate dilution factor for concentrations exceeding this table.

[a] The volume of solvent added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of solvent is necessary to maintain a volume of 100 µL added to the syringe.

[b] Dilute an aliquot of the solvent extract and then take 100 µL for analysis.

TABLE 10

DIRECT INJECTION ANALYSIS OF NEW OIL AT 5 PPM (METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone	91	14.8	1.9	5.0
Benzene	86	21.3	0.1	0.5
n-Butanol*,**	107	27.8	0.5	5.0
iso-Butanol*,**	95	19.5	0.9	5.0
Carbon tetrachloride	86	44.7	0.0	0.5
Carbon disulfide**	53	22.3	0.0	5.0
Chlorobenzene	81	29.3	0.0	5.0
Chloroform	84	29.3	0.0	6.0
1,4-Dichlorobenzene	98	24.9	0.0	7.5
1,2-Dichloroethane	101	23.1	0.0	0.5
1,1-Dichloroethene	97	45.3	0.0	0.7
Diethyl ether	76	24.3	0.0	5.0
Ethyl acetate	113	27.4	0.0	5.0
Ethylbenzene	83	30.1	0.2	5.0
Hexachloroethane	71	30.3	0.0	3.0
Methylene chloride	98	45.3	0.0	5.0
Methyl ethyl ketone	79	24.6	0.4	5.0
MIBK	93	31.4	0.0	5.0
Nitrobenzene	89	30.3	0.0	2.0
Pyridine	31	35.9	0.0	5.0
Tetrachloroethene	82	27.1	0.0	0.7
Trichlorofluoromethane	76	27.6	0.0	5.0
1,1,2-Trichlorotrifluoroethane	69	29.2	0.0	5.0
Toluene	73	21.9	0.6	5.0
Trichloroethene	66	28.0	0.0	0.5
Vinyl chloride	63	35.2	0.0	0.2
o-Xylene	83	29.5	0.4	5.0
m/p-Xylene	84	29.5	0.6	10.0

* Alternate mass employed
** IS quantitation

Data are taken from Reference 9.

TABLE 11

SINGLE LABORATORY PERFORMANCE
DATA FOR THE DIRECT INJECTION METHOD - USED OIL (METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone**	105	54	2.0	5.0
Benzene	3135	44	14	0.5
Benzene-d_6	56	44	2.9	0.5
n-Butanol**	100	71	12	5.0
iso-Butanol*,**	132	27	0	5.0
Carbon tetrachloride	143	68	0	0.5
Carbon tetrachloride-^{13}C	99	44	5.1	0.5
Carbon disulfide**	95	63	0	5.0
Chlorobenzene	148	71	0	5.0
Chlorobenzene-d_5	60	44	3.6	5.0
Chloroform	149	74	0	6.0
Chloroform-d_1	51	44	2.6	6.0
1,4-Dichlorobenzene	142	72	0	7.5
1,4-Dichlorobenzene-d_4	53	44	3.4	7.5
1,2-Dichloroethane**	191	54	0	0.5
1,1-Dichloroethene*	155	51	0	0.7
1,1-Dichloroethene-d_2	68	44	3.4	0.7
Diethyl ether**	95	66	0	5.0
Ethyl acetate*,**	126	39	0	5.0
Ethylbenzene	1298	44	54	5.0
Ethylbenzene-d_{10}	63	44	3.6	5.0
Hexachloroethane	132	72	0	3.0
Hexachloroethane-^{13}C	54	45	3.5	3.0
Methylene chloride**	86	65	0.3	5.0
Methyl ethyl ketone**	107	64	0	5.0
4-Methyl-2-pentanone (MIBK)**	100	74	0.1	5.0
Nitrobenzene	111	80	0	2.0
Nitrobenzene-d_5	65	53	4.0	2.0
Pyridine**	68	85	0	5.0
Pyridine-d_5	ND	--	0	5.0
Tetrachloroethene**	101	73	0	0.7
Trichlorofluoromethane**	91	70	0	5.0
1,1,2-Cl_3F_3ethane**	81	70	0	5.0
Toluene	2881	44	128	5.0
Toluene-d_8	63	44	3.6	5.0
Trichloroethene	152	57	0	0.5
Trichloroethene-d_1	55	44	2.8	0.5

TABLE 11 (cont.)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Vinyl chloride**	100	69	0	0.2
o-Xylene	2292	44	105	5.0
o-Xylene-d_{10}	76	44	4.2	5.0
m-/p-Xylene	2583	44	253	10.0
p-Xylene-d_{10}	67	44	3.7	10.0

* Alternate mass employed
** IS quantitation
ND = Not Detected

Data are based on seven measurements and are taken from Reference 9.

TABLE 12

METHOD DETECTION LIMITS (METHOD 5031)

Compound	MDL (µg/L) Macro[a]	Concentration Factor Macro	Micro
Acetone	31	25-500	-
Acetonitrile	57	25-500	200
Acrolein	-	-	100
Acrylonitrile	16	25-500	100
Allyl Alcohol	7	25-500	-
1-Butanol	-	-	250
Crotonaldehyde	12	25-500	-
1,4-Dioxane	12	25-500	150
Ethyl Acetate	-	-	100
Isobutyl alcohol	7	25-500	-
Methanol	38	25-500	140
Methyl Ethyl Ketone	16	25-500	-
2-Methyl-1-propanol	-	-	250
n-Nitroso-di-n-butylamine	14	25-500	-
Paraldehyde	10	25-500	-
2-Picoline	7	25-500	-
1-Propanol	-	-	240
Propionitrile	11	25-500	200
Pyridine	4	25-500	-
o-Toluidine	13	25-500	-

[a] Produced by analysis of seven aliquots of reagent water spiked at 25 ppb at the listed compounds; calculations based on internal standard technique and use of the following equation:

MDL = 3.134 x Std. Dev. of low concentration spike (ppb).

[b] When a 40-mL sample is used, and the first 100 µL of distillate are collected.

TABLE 13

TARGET COMPOUNDS, SURROGATES, AND INTERNAL STANDARDS (METHOD 5031)

Target Compound	Surrogate	Internal Standard
Acetone	d_6-Acetone	d_8-Isopropyl alcohol
Acetonitrile	d_3-Acetonitrile	d_8-Isopropyl alcohol
Acrylonitrile	d_8-Isopropyl alcohol	
Allyl alcohol	d_7-Dimethyl formamide	
Crotonaldehyde	d_8-Isopropyl alcohol	
1,4-Dioxane	d_8-1,4-Dioxane	d_7-Dimethyl formamide
Isobutyl alcohol	d_7-Dimethyl formamide	
Methanol	d_3-Methanol	d_8-Isopropyl alcohol
Methyl ethyl ketone	d_8-Isopropyl alcohol	
N-Nitroso-di-n-butylamine	d_7-Dimethyl formamide	
Paraldehyde	d_7-Dimethyl formamide	
2-Picoline	d_7-Dimethyl formamide	
Propionitrile	d_8-Isopropyl alcohol	
Pyridine	d_5-Pyridine	d_7-Dimethyl formamide
o-Toluidine	d_7-Dimethyl formamide	

TABLE 14

RECOMMENDED CONCENTRATIONS FOR CALIBRATION SOLUTIONS (METHOD 5031)

Compound	Concentration(s) (ng/µL)
Internal Standards	
d_5-benzyl alcohol	10.0
d_{14}-Diglyme	10.0
d_7-Dimethyl formamide	10.0
d_8-Isopropyl alcohol	10.0
Surrogates	
d_6-Acetone	10.0
d_3-Acetonitrile	10.0
d_8-1,4-Dioxane	10.0
d_3-Methanol	10.0
d_5-Pyridine	10.0
Target Compounds	
Acetone	1.0, 5.0, 10.0, 25.0, 100.0
Acetonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Acrylonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Allyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Crotonaldehyde	1.0, 5.0, 10.0, 25.0, 100.0
1,4-Dioxane	1.0, 5.0, 10.0, 25.0, 100.0
Isobutyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Methanol	1.0, 5.0, 10.0, 25.0, 100.0
Methyl ethyl ketone	1.0, 5.0, 10.0, 25.0, 100.0
N-Nitroso-di-n-butylamine	1.0, 5.0, 10.0, 25.0, 100.0
Paraldehyde	1.0, 5.0, 10.0, 25.0, 100.0
2-Picoline	1.0, 5.0, 10.0, 25.0, 100.0
Propionitrile	1.0, 5.0, 10.0, 25.0, 100.0
Pyridine	1.0, 5.0, 10.0, 25.0, 100.0
o-Toluidine	1.0, 5.0, 10.0, 25.0, 100.0

TABLE 15

CHARACTERISTIC IONS AND RETENTION TIMES FOR VOCs (METHOD 5031)

Compound	Quantitation Ion[a]	Secondary Ions	Retention Time (min)[b]
Internal Standards			
d_8-Isopropyl alcohol	49		1.75
d_{14}-Diglyme	66	98,64	9.07
d_7-Dimethyl formamide	50	80	9.20
Surrogates			
d_6-Acetone	46	64,42	1.03
d_3-Methanol	33	35,30	1.75
d_3-Acetonitrile	44	42	2.63
d_8-1,4-Dioxane	96	64,34	3.97
d_5-Pyridine	84	56,79	6.73
d_5-Phenol[c]	99	71	15.43
Target Compounds			
Acetone	43	58	1.05
Methanol	31	29	1.52
Methyl ethyl ketone	43	72,57	1.53
Methacrylonitrile[c]	67	41	2.38
Acrylonitrile	53	52,51	2.53
Acetonitrile	41	40,39	2.73
Methyl isobutyl ketone[c]	85	100,58	2.78
Propionitrile	54	52,55	3.13
Crotonaldehyde	41	70	3.43
1,4-Dioxane	58	88,57	4.00
Paraldehyde	45	89	4.75
Isobutyl alcohol	43	33,42	5.05
Allyl alcohol	57	39	5.63
Pyridine	79	50,52	6.70
2-Picoline	93	66	7.27
N-Nitroso-di-n-butylamine	84	116	12.82
Aniline[c]	93	66,92	13.23
o-Toluidine	106	107	13.68
Phenol[c]	94	66,65	15.43

[a] These ions were used for quantitation in selected ion monitoring.
[b] GC column: DB-Wax, 30 meter x 0.53 mm, 1 µm film thickness.
Oven program: 45°C for 4 min, increased to 220°C at 12°C/min.
[c] Compound removed from target analyte list due to poor accuracy and precision.

TABLE 16

METHOD ACCURACY AND PRECISION BY MEAN PERCENT RECOVERY AND PERCENT RELATIVE STANDARD DEVIATION[a] (METHOD 5031 - MACRODISTILLATION TECHNIQUE)
(Single Laboratory and Single Operator)

Compound	25 ppb Spike		100 ppb Spike		500 ppb Spike	
	Mean %R	%RSD	Mean %R	%RSD	Mean %R	%RSD
d_6-Acetone	66	24	69	14	65	16
d_3-Acetonitrile	89	18	80	18	70	10
d_8-1,4-Dioxane	56	34	58	11	61	18
d_3-Methanol	43	29	48	19	56	14
d_5-Pyridine	83	6.3	84	7.8	85	9.0
Acetone	67	45	63	14	60	14
Acetonitrile	44	35	52	15	56	15
Acrylonitrile	49	42	47	27	45	27
Allyl alcohol	69	13	70	9.7	73	10
Crotonaldehyde	68	22	68	13	69	13
1,4-Dioxane	63	25	55	16	54	13
Isobutyl alcohol	66	14	66	5.7	65	7.9
Methanol	50	36	46	22	49	18
Methyl ethyl ketone	55	37	56	20	52	19
N-Nitroso-di-n-butylamine	57	21	61	15	72	18
Paraldehyde	65	20	66	11	60	8.9
Picoline	81	12	81	6.8	84	8.0
Propionitrile	67	22	69	13	68	13
Pyridine	74	7.4	72	6.7	74	7.3
o-Toluidine	52	31	54	15	58	12

[a] Data from analysis of seven aliquots of reagent water spiked at each concentration, using a quadrapole mass spectrometer in the selected ion monitoring mode.

TABLE 17

RECOVERIES IN SAND SAMPLES FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

Compound	Recovery per Replicate (ng) 1	2	3	4	5	Mean	RSD	Mean Rec
Vinyl chloride	8.0	7.5	6.7	5.4	6.6	6.8	13.0	34.2
Trichlorofluoromethane	13.3	16.5	14.9	13.0	10.3	13.6	15.2	68.0
1,1-Dichloroethene	17.1	16.7	15.1	14.8	15.6	15.9	5.7	79.2
Methylene chloride	24.5	22.7	19.7	19.4	20.6	21.4	9.1	107
trans-1,2-Dichloroethene	22.7	23.6	19.4	18.3	20.1	20.8	0.7	104
1,2-Dichloroethane	18.3	18.0	16.7	15.6	15.9	16.9	6.4	84.4
cis-1,2-Dichloroethene	26.1	23.1	22.6	20.3	20.8	22.6	9.0	113
Bromochloromethane	24.5	25.4	20.9	20.1	20.1	22.2	10.2	111
Chloroform	26.5	26.0	22.1	18.9	22.1	23.1	12.2	116
1,1,1-Trichloroethane	21.5	23.0	23.9	16.7	31.2	23.4	21.2	117
Carbon tetrachloride	23.6	24.2	22.6	18.3	23.3	22.4	9.4	112
Benzene	22.4	23.9	20.4	17.4	19.2	20.7	11.2	103
Trichloroethene	21.5	20.5	19.2	14.4	19.1	18.9	12.7	94.6
1,2-Dichloropropane	24.9	26.3	23.1	19.0	23.3	23.3	10.5	117
Dibromomethane	25.4	26.4	21.6	20.4	23.6	23.5	9.6	117
Bromodichloromethane	25.7	26.7	24.1	17.9	23.0	23.5	13.1	117
Toluene	28.3	25.0	24.8	16.3	23.6	23.6	16.9	118
1,1,2-Trichloroethane	25.4	24.5	21.6	17.7	22.1	22.2	12.1	111
1,3-Dichloropropane	25.4	24.2	22.7	17.0	22.2	22.3	12.8	112
Dibromochloromethane	26.3	26.2	23.7	18.2	23.2	23.5	12.5	118
Chlorobenzene	22.9	22.5	19.8	14.6	19.4	19.9	15.0	99.3
1,1,1,2-Tetrachloroethane	22.4	27.7	25.1	19.4	22.6	23.4	12.0	117
Ethylbenzene	25.6	25.0	22.1	14.9	24.0	22.3	17.5	112
p-Xylene	22.5	22.0	19.8	13.9	20.3	19.7	15.7	98.5
o-Xylene	24.2	23.1	21.6	14.0	20.4	20.7	17.3	103
Styrene	23.9	21.5	20.9	14.3	20.5	20.2	15.7	101
Bromoform	26.8	25.6	26.0	20.1	23.5	24.4	9.9	122
iso-Propylbenzene	25.3	25.1	24.2	15.4	24.6	22.9	16.6	114
Bromobenzene	19.9	21.8	20.0	15.5	19.1	19.3	10.7	96.3
1,2,3-Trichloropropane	25.9	23.0	25.6	15.9	21.4	22.2	15.8	111
n-Propylbenzene	26.0	23.8	22.6	13.9	21.9	21.6	19.0	106
2-Chlorotoluene	23.6	23.8	21.3	13.0	21.5	20.6	19.2	103
4-Chlorotoluene	21.0	19.7	18.4	12.1	18.3	17.9	17.1	89.5
1,3,5-Trimethylbenzene	24.0	22.1	22.5	13.8	22.9	21.1	17.6	105
sec-Butylbenzene	25.9	25.3	27.8	16.1	28.6	24.7	18.1	124
1,2,4-Trimethylbenzene	30.6	39.2	22.4	18.0	22.7	26.6	28.2	133
1,3-Dichlorobenzene	20.3	20.6	18.2	13.0	17.6	17.9	15.2	89.7
p-iso-Propyltoluene	21.6	22.1	21.6	16.0	22.8	20.8	11.8	104
1,4-Dichlorobenzene	18.1	21.2	20.0	13.2	17.4	18.0	15.3	90.0
1,2-Dichlorobenzene	18.4	22.5	22.5	15.2	19.9	19.7	13.9	96.6
n-Butylbenzene	13.1	20.3	19.5	10.8	18.7	16.5	23.1	82.4
1,2,4-Trichlorobenzene	14.5	14.9	15.7	8.8	12.3	13.3	18.8	66.2
Hexachlorobutadiene	17.6	22.5	21.6	13.2	21.6	19.3	18.2	96.3
1,2,3-Trichlorobenzene	14.9	15.9	16.5	11.9	13.9	14.6	11.3	73.1

Data in Tables 17, 18, and 19 are from Reference 15.

TABLE 18
RECOVERIES IN C-HORIZON SOILS FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

Compound	Recovery per Replicate (ng) 1	2	3	4	5	Mean	RSD	Mean Rec
Vinyl chloride	33.4	31.0	30.9	29.7	28.6	30.8	5.2	154
Trichlorofluoromethane	37.7	20.8	20.0	21.8	20.5	24.1	28.2	121
1,1-Dichloroethene	21.7	33.5	39.8	30.2	32.5	31.6	18.5	158
Methylene chloride	20.9	19.4	18.7	18.3	18.4	19.1	5.1	95.7
trans-1,2-Dichloroethene	21.8	18.9	20.4	17.9	17.8	19.4	7.9	96.8
1,1-Dichloroethane	23.8	21.9	21.3	21.3	20.5	21.8	5.2	109
cis-1,2-Dichloroethene	21.6	18.8	18.5	18.2	18.2	19.0	6.7	95.2
Bromochloromethane	22.3	19.5	19.3	19.0	19.2	20.0	6.0	100
Chloroform	20.5	17.1	17.3	16.5	15.9	17.5	9.2	87.3
1,1,1-Trichloroethane	16.4	11.9	10.7	9.5	9.4	11.6	22.4	57.8
Carbon tetrachloride	13.1	11.3	13.0	11.8	11.2	12.1	6.7	60.5
Benzene	21.1	19.3	18.7	18.2	16.9	18.8	7.4	94.1
Trichloroethene	19.6	16.4	16.5	16.5	15.5	16.9	8.3	84.5
1,2-Dichloropropane	21.8	19.0	18.3	18.8	16.5	18.9	9.0	94.4
Dibromomethane	20.9	17.9	17.9	17.2	18.3	18.4	6.9	92.1
Bromodichloromethane	20.9	18.0	18.9	18.2	17.3	18.6	6.6	93.2
Toluene	22.2	17.3	18.8	17.0	15.9	18.2	12.0	91.2
1,1,2-Trichloroethane	21.0	16.5	17.2	17.2	16.5	17.7	9.6	88.4
1,3-Dichloropropane	21.4	17.3	18.7	18.6	16.7	18.5	8.8	92.6
Dibromochloromethane	20.9	18.1	19.0	18.8	16.6	18.7	7.5	93.3
Chlorobenzene	20.8	18.4	17.6	16.8	14.8	17.7	11.2	88.4
1,1,1,2-Tetrachloroethane	19.5	19.0	17.8	17.2	16.5	18.0	6.2	90.0
Ethylbenzene	21.1	18.3	18.5	16.9	15.3	18.0	10.6	90.0
p-Xylene	20.0	17.4	18.2	16.3	14.4	17.3	10.9	86.3
o-Xylene	20.7	17.2	16.8	16.2	14.8	17.1	11.4	85.7
Styrene	18.3	15.9	16.2	15.3	13.7	15.9	9.3	79.3
Bromoform	20.1	15.9	17.1	17.5	16.1	17.3	8.6	86.7
iso-Propylbenzene	21.0	18.1	19.2	18.4	15.6	18.4	9.6	92.2
Bromobenzene	20.4	16.2	17.2	16.7	15.4	17.2	10.1	85.9
1,1,2,2-Tetrachloroethane	23.3	17.9	21.2	18.8	16.8	19.6	12.1	96.0
1,2,3-Trichloropropane	18.4	14.6	15.6	16.1	15.6	16.1	8.0	80.3
n-Propylbenzene	20.4	18.9	17.9	17.0	14.3	17.7	11.6	88.4
2-Chlorotoluene	19.1	17.3	16.1	16.0	14.4	16.7	9.2	83.6
4-Chlorotoluene	19.0	15.5	16.8	15.9	13.6	16.4	10.6	81.8
1,3,5-Trimethylbenzene	20.8	18.0	17.4	16.1	14.7	17.4	11.7	86.9
sec-Butylbenzene	21.4	18.3	18.9	17.0	14.9	18.1	11.8	90.5
1,2,4-Trimethylbenzene	20.5	18.6	16.8	15.3	13.7	17.0	14.1	85.0
1,3-Dichlorobenzene	17.6	15.9	15.6	14.2	14.4	15.6	7.9	77.8
p-iso-Propyltoluene	20.5	17.0	17.1	15.6	13.4	16.7	13.9	83.6
1,4-Dichlorobenzene	18.5	13.8	14.8	16.7	14.9	15.7	10.5	78.7
1,2-Dichlorobenzene	18.4	15.0	15.4	15.3	13.5	15.5	10.5	77.6
n-Butylbenzene	19.6	15.9	15.9	14.4	18.9	16.9	11.7	84.6
1,2,4-Trichlorobenzene	15.2	17.2	17.4	13.6	12.1	15.1	13.5	75.4
Hexachlorobutadiene	18.7	16.2	15.5	13.8	16.6	16.1	10.0	80.7
Naphthalene	13.9	11.1	10.2	10.8	11.4	11.5	11.0	57.4
1,2,3-Trichlorobenzene	14.9	15.2	16.8	13.7	12.7	14.7	9.5	73.2

TABLE 19
RECOVERIES IN GARDEN SOIL FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

Compound	Recovery per Replicate (ng) 1	2	3	4	5	Mean	RSD	Mean Rec
Vinyl chloride	12.7	10.9	9.8	8.1	7.2	9.7	20.2	48.7
Trichlorofluoromethane	33.7	6.4	30.3	27.8	22.9	24.2	39.6	121
1,1-Dichloroethene	27.7	20.5	24.1	15.1	13.2	20.1	26.9	101
Methylene chloride	25.4	23.9	24.7	22.2	24.2	24.1	4.4	120
trans-1,2-Dichloroethene	2.8	3.0	3.3	2.2	2.4	2.7	15.0	13.6
1,1-Dichloroethane	24.1	26.3	27.0	20.5	21.2	23.8	11.0	119
cis-1,2-Dichloroethene	8.3	10.2	8.7	5.8	6.4	7.9	20.1	39.4
Bromochloromethane	11.1	11.8	10.2	8.8	9.0	10.2	11.2	50.9
Chloroform	16.7	16.9	17.0	13.8	15.0	15.9	7.9	79.3
1,1,1-Trichloroethane	24.6	22.8	22.1	16.2	20.9	21.3	13.4	107
Carbon tetrachloride	19.4	20.3	22.2	20.0	20.2	20.4	4.6	102
Benzene	21.4	22.0	22.4	19.6	20.4	21.2	4.9	106
Trichloroethene	12.4	16.5	14.9	9.0	9.9	12.5	22.9	62.7
1,2-Dichloropropane	19.0	18.8	19.7	16.0	17.6	18.2	7.1	91.0
Dibromomethane	7.3	8.0	6.9	5.6	6.8	6.9	11.3	34.6
Bromodichloromethane	14.9	15.9	15.9	12.8	13.9	14.7	8.3	73.3
Toluene	42.6	39.3	45.1	39.9	45.3	42.4	5.9	212
1,1,2-Trichloroethane	13.9	15.2	1.4	21.3	14.9	15.9	17.0	79.6
1,3-Dichloropropane	13.3	16.7	11.3	10.9	9.5	12.3	20.3	61.7
Dibromochloromethane	14.5	13.1	14.5	11.9	14.4	13.7	7.6	68.3
Chlorobenzene	8.4	10.0	8.3	6.9	7.8	8.3	12.1	41.3
1,1,1,2-Tetrachloroethane	16.7	16.7	15.6	15.8	15.7	16.1	3.2	80.4
Ethylbenzene	22.1	21.4	23.1	20.1	22.6	21.9	4.8	109
p-Xylene	41.4	38.4	43.8	38.3	44.0	41.2	6.1	206
o-Xylene	31.7	30.8	34.3	30.4	33.2	32.1	4.6	160
Styrene	0	0	0	0	0	0	0	0
Bromoform	8.6	8.9	9.1	7.0	7.7	8.3	9.4	41.4
iso-Propylbenzene	18.1	18.8	9.7	18.3	19.6	18.9	3.5	94.4
Bromobenzene	5.1	5.4	5.3	4.4	4.0	4.8	11.6	24.1
1,1,2,2-Tetrachloroethane	14.0	13.5	14.7	15.3	17.1	14.9	8.5	74.5
1,2,3-Trichloropropane	11.0	12.7	11.7	11.7	11.9	11.8	4.5	59.0
n-Propylbenzene	13.4	13.3	14.7	12.8	13.9	13.6	4.7	68.1
2-Chlorotoluene	8.3	9.0	11.7	8.7	7.9	9.1	14.8	45.6
4-Chlorotoluene	5.1	5.4	5.5	4.8	4.5	5.0	7.9	25.2
1,3,5-Trimethylbenzene	31.3	27.5	33.0	31.1	33.6	31.3	6.8	157
sec-Butylbenzene	13.5	13.4	16.4	13.8	15.4	14.5	8.3	72.5
1,2,4-Trimethylbenzene	38.7	32.4	40.8	34.1	40.3	37.3	9.1	186
1,3-Dichlorobenzene	3.6	3.6	3.7	3.0	3.2	3.4	8.0	17.2
p-iso-Propyltoluene	14.7	14.1	16.1	13.9	15.1	14.8	5.2	73.8
1,4-Dichlorobenzene	3.0	3.5	3.3	2.6	2.8	3.0	10.2	15.0
1,2-Dichlorobenzene	3.6	4.3	4.0	3.5	3.6	3.8	8.3	19.0
n-Butylbenzene	17.4	13.8	14.0	18.9	24.0	17.6	21.2	88.0
1,2,4-Trichlorobenzene	2.8	2.9	3.3	2.6	3.2	3.0	8.5	15.0
Hexachlorobutadiene	4.8	4.0	6.1	5.6	6.0	5.3	15.1	26.4
Naphthalene	5.5	5.1	5.5	4.7	5.6	5.3	6.2	26.5
1,2,3-Trichlorobenzene	2.2	2.3	2.4	2.2	2.3	2.3	3.5	11.4

Data in Table 19 are from Reference 15.

TABLE 20

VOLATILE ORGANIC ANALYTE RECOVERY FROM SOIL
USING VACUUM DISTILLATION (METHOD 5032)[a]

Compound	Soil/H_2O[b] Recovery Mean	Soil/H_2O[b] Recovery RSD	Soil/Oil[c] Recovery Mean	Soil/Oil[c] Recovery RSD	Soil/Oil/H_2O Recovery Mean	Soil/Oil/H_2O Recovery RSD
Chloromethane	61	20	40	18	108	68
Bromomethane	58	20	47	13	74	13
Vinyl chloride	54	12	46	11	72	20
Chloroethane	46	10	41	8	52	14
Methylene chloride	60	2	65	8	76	11
Acetone	INT[e]	INT	44	8		
Carbon disulfide	47	13	53	10	47	4
1,1-Dichloroethene	48	9	47	5	58	3
1,1-Dichloroethane	61	6	58	9	61	6
trans-1,2-Trichloroethane	54	7	60	7	56	5
cis-1,2-Dichloroethene	60	4	72	6	63	8
Chloroform	104	11	93	6	114	15
1,2-Dichloroethane	177	50	117	8	151	22
2-Butanone	INT	36	38	INT		
1,1,1-Trichloroethane	124	13	72	16	134	26
Carbon tetrachloride	172	122	INT	INT		
Vinyl acetate	88	11	INT			
Bromodichloromethane	93	4	91	23	104	23
1,1,2,2-Tetrachloroethane	96	13	50	12	104	7
1,2-Dichloropropane	105	8	102	6	111	6
trans-1,3-Dichloropropene	134	10	84	16	107	8
Trichloroethene	98	9	99	10	100	5
Dibromochloromethane	119	8	125	31	142	16
1,1,2-Trichloroethane	126	10	72	16	97	4
Benzene	99	7	CONT[f]	CONT		
cis-1,3-Dichloropropene	123	12	94	13	112	9
Bromoform	131	13	58	18	102	9
2-Hexanone	155	18	164	19	173	29
4-Methyl-2-pentanone	152	20	185	20	169	18
Tetrachloroethene	90	9	123	14	128	7
Toluene	94	3	CONT	CONT		
Chlorobenzene	98	7	93	18	112	5
Ethylbenzene	114	13	CONT	CONT		
Styrene	106	8	93	18	112	5
p-Xylene	97	9	CONT	CONT		
o-Xylene	105	8	112	12	144	13

TABLE 20 (cont.)

Compound	Soil/H_2O[b] Recovery Mean	RSD	Soil/Oil[c] Recovery Mean	RSD	Soil/Oil/H_2O Recovery Mean	RSD
Surrogates						
1,2-Dichloroethane	177	50	117	8	151	22
Toluene-d_8	96	6	79	12	82	6
Bromofluorobenzene	139	13	37	13	62	5

[a] Results are for 10 min. distillations times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision value reflects the propagated errors. Each analyte was spiked at 50 ppb. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may introduce bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

[b] Soil samples spiked with 0.2 mL water containing analytes and then 5 mL water added to make slurry.

[c] Soil sample + 1 g cod liver oil, spiked with 0.2 mL water containing analytes.

[d] Soil samples + 1 g cod liver oil, spiked as above with 5 mL of water added to make slurry.

[e] Interference by co-eluting compounds prevented accurate measurement of analyte.

[f] Contamination of sample matrix by analyte prevented assessment of efficiency.

TABLE 21

VACUUM DISTILLATION EFFICIENCIES FOR VOLATILE ORGANIC ANALYTES IN FISH TISSUE (METHOD 5032)[a]

Compound	Efficiency Mean (%)	RSD (%)
Chloromethane	N/A[b]	
Bromomethane	N/A[b]	
Vinyl chloride	N/A[b]	
Chloroethane	N/A[b]	
Methylene chloride	CONT[c]	
Acetone	CONT[c]	
Carbon disulfide	79	36
1,1-Dichloroethene	122	39
1,1-Dichloroethane	126	35
trans-1,2-Trichloroethene	109	46
cis-1,2-Dichloroethene	106	22
Chloroform	111	32
1,2-Dichloroethane	117	27
2-Butanone	INT[d]	
1,1,1-Trichloroethane	106	30
Carbon tetrachloride	83	34
Vinyl acetate	INT[d]	
Bromodichloromethane	97	22
1,1,2,2-Tetrachloroethane	67	20
1,2-Dichloropropane	117	23
trans-1,3-Dichloropropene	92	22
Trichloroethene	98	31
Dibromochloromethane	71	19
1,1,2-Trichloroethane	92	20
Benzene	129	35
cis-1,3-Dichloropropene	102	24
Bromoform	58	19
2-Hexanone	INT[d]	
4-Methyl-2-pentanone	113	37
Tetrachloroethene	66	20
Toluene	CONT[c]	
Chlorobenzene	65	19
Ethylbenzene	74	19
Styrene	57	14
p-Xylene	46	13
o-Xylene	83	20

TABLE 21 (cont.)

Compound	Efficiency Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane	115	27
Toluene-d_8	88	24
Bromofluorobenzene	52	15

[a] Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicate 10-g aliquots of fish spiked at 25 ppb were analyzed using GC/MS external standard quantitation. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards were replicated and results reflect 1 sigma propagated standard deviation.

[b] No analyses.

[c] Contamination of sample matrix by analyte prevented accurate assessment of analyte efficiency.

[d] Interfering by co-eluting compounds prevented accurate measurement of analyte.

TABLE 22

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES IN FISH TISSUE (METHOD 5032)[a]

Compound	Method Detection Limit (ppb) External Standard Method	Internal Standard Method
Chloromethane	7.8	7.3
Bromomethane	9.7	9.8
Vinyl chloride	9.5	9.4
Chloroethane	9.2	10.0
Methylene chloride	CONT[b]	CONT[b]
Acetone	CONT[b]	CONT[b]
Carbon disulfide	5.4	4.9
1,1-Dichloroethene	4.0	5.7
1,1-Dichloroethane	4.0	3.5
trans-1,2-Dichloroethene	4.4	4.0
cis-1,2-Dichloroethene	4.7	4.1
Chloroform	5.6	5.0
1,2-Dichloroethane	3.3	3.2
2-Butanone	INT[c]	INT[c]
1,1,1-Trichloroethane	1.1	4.2
Carbon tetrachloride	3.2	3.5
Vinyl acetate	INT[c]	INT[c]
Bromodichloromethane	3.2	2.8
1,1,2,2-Tetrachloroethane	4.4	3.8
1,2-Dichloropropane	3.8	3.7
trans-1,3-Dichloropropene	3.4	3.0
Trichloroethene	3.1	4.0
Dibromochloromethane	3.5	3.2
1,1,2-Trichloroethane	4.4	3.3
Benzene	3.6	3.2
cis-1,3-Dichloropropene	3.5	3.0
Bromoform	4.9	4.0
2-Hexanone	7.7	8.0
4-Methyl-2-pentanone	7.5	8.0
Tetrachloroethene	4.3	4.0
Toluene	3.0	2.5
Chlorobenzene	3.3	2.8
Ethylbenzene	3.6	3.5
Styrene	3.5	3.3
p-Xylene	3.7	3.5
o-Xylene	3.3	4.7

Footnotes are on the following page.

[a] Values shown are the average MDLs for studies on three non-consecutive days, involving seven replicate analyses of 10 g of fish tissue spiked a 5 ppb. Daily MDLs were calculated as three times the standard deviation. Quantitation was performed by GC/MS Method 8260 and separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[b] Contamination of sample by analyte prevented determination.

[c] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 23

VOLATILE ORGANIC ANALYTES RECOVERY FOR WATER
USING VACUUM DISTILLATION (METHOD 5032)[a]

Compound	5 mL H_2O[b] Recovery Mean	RSD	20 mL H_2O[c] Recovery Mean	RSD	20 mL H_2O/Oil Recovery Mean	RSD
Chloromethane	114	27	116	29	176	67
Bromomethane	131	14	121	14	113	21
Vinyl chloride	131	13	120	16	116	23
Chloroethane	110	15	99	8	96	16
Methylene chloride	87	16	105	15	77	6
Acetone	83	22	65	34	119	68
Carbon disulfide	138	17	133	23	99	47
1,1-Dichloroethene	105	11	89	4	96	18
1,1-Dichloroethane	118	10	119	11	103	25
trans-1,2-Dichloroethene	105	11	107	14	96	18
cis-1,2-Dichloroethene	106	7	99	5	104	23
Chloroform	114	6	104	8	107	21
1,2-Dichloroethane	104	6	109	8	144	19
2-Butanone	83	50	106	31	INT[c]	
1,1,1-Trichloroethane	118	9	109	9	113	23
Carbon tetrachloride	102	6	108	12	109	27
Vinyl acetate	90	16	99	7	72	36
Bromodichloromethane	104	3	110	5	99	5
1,1,2,2-Tetrachloroethane	85	17	81	7	111	43
1,2-Dichloropropane	100	6	103	2	104	7
trans-1,3-Dichloropropene	105	8	105	4	92	4
Trichloroethene	98	4	99	2	95	5
Dibromochloroethane	99	8	99	6	90	25
1,1,2-Trichloroethane	98	7	100	4	76	12
Benzene	97	4	100	5	112	10
cis-1,3-Dichloropropene	106	5	105	4	98	3
Bromoform	93	16	94	8	57	21
2-Hexanone	60	17	63	16	78	23
4-Methyl-2-pentanone	79	24	63	14	68	15
Tetrachloroethene	101	3	97	7	77	14
Toluene	100	6	97	8	85	5
Chlorobenzene	98	6	98	4	88	16
Ethylbenzene	100	3	92	8	73	13
Styrene	98	4	97	9	88	16
p-Xylene	96	4	94	8	60	12
o-Xylene	96	7	95	6	72	14

TABLE 23 (cont.)

Compound	5 mL H_2O[b] Recovery Mean	RSD	20 mL H_2O[c] Recovery Mean	RSD	20 mL H_2O/Oil Recovery Mean	RSD
Surrogates						
1,2-Dichloroethane	104	6	109	6	144	19
Toluene-d_8	104	5	102	2	76	7
Bromofluorobenzene	106	6	106	9	40	8

[a] Results are for 10 min. distillation times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision values reflect the propagated errors. Concentrations of analytes were 50 ppb for 5-mL samples and 25 ppb for 20-mL samples. Recovery data generated with comparison to analyses of standards without the water matrix.

[b] Sample contained 1 gram cod liver oil and 20 mL water. An emulsion was created by adding 0.2 mL of water saturated with lecithin.

[c] Interference by co-eluting compounds prevented accurate assessment of recovery.

TABLE 24

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES
USING VACUUM DISTILLATION (METHOD 5032) (INTERNAL STANDARD METHOD)[a]

Compound	Water[b] (µg/L)	Soil[c] (µg/kg)	Tissue[d] (µg/kg)	Oil[e] (mg/kg)
Chloromethane	3.2	8.0	7.3	N/A[f]
Bromomethane	2.8	4.9	9.8	N/A[f]
Vinyl chloride	3.5	6.0	9.4	N/A[f]
Chloroethane	5.9	6.0	10.0	N/A[f]
Methylene chloride	3.1	4.0	CONT[g]	0.05
Acetone	5.6	CONT[g]	CONT[g]	0.06
Carbon disulfide	2.5	2.0	4.9	0.18
1,1-Dichloroethene	2.9	3.2	5.7	0.18
1,1-Dichloroethane	2.2	2.0	3.5	0.14
trans-1,2-Dichloroethene	2.2	1.4	4.0	0.10
cis-1,2-Dichloroethene	2.0	2.3	4.1	0.07
Chloroform	2.4	1.8	5.0	0.07
1,2-Dichloroethane	1.7	1.5	3.2	0.06
2-Butanone	7.4	INT[h]	INT[h]	INT[h]
1,1,1-Trichloroethane	1.8	1.7	4.2	0.10
Carbon tetrachloride	1.4	1.5	3.5	0.13
Vinyl acetate	11.8	INT[h]	INT[h]	INT[h]
Bromodichloromethane	1.6	1.4	2.8	0.06
1,1,2,2-Tetrachloroethane	2.5	2.1	3.8	0.02
1,2-Dichloropropane	2.2	2.1	3.7	0.15
trans-1,3-Dichloropropene	1.5	1.7	3.0	0.05
Trichloroethene	1.6	1.7	4.0	0.04
Dibromochloromethane	1.7	1.5	3.2	0.07
1,1,2-Trichloroethane	2.1	1.7	3.3	0.05
Benzene	0.5	1.5	3.2	0.05
cis-1,3-Dichloropropene	1.4	1.7	3.0	0.04
Bromoform	1.8	1.5	4.0	0.05
2-Hexanone	4.6	3.6	8.0	INT[h]
4-Methyl-2-pentanone	3.5	4.6	8.0	INT[h]
Tetrachloroethene	1.4	1.6	4.0	0.10
Toluene	1.0	3.3	2.5	0.05
Chlorobenzene	1.4	1.4	2.8	0.06
Ethylbenzene	1.5	2.8	3.5	0.04
Styrene	1.4	1.4	3.3	0.18
p-Xylene	1.5	2.9	3.5	0.20
o-Xylene	1.7	3.4	4.7	0.07

Footnotes are found on the following page.

[a] Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Method detection limits are the average MDLs for studies on three non-consecutive days.

[b] Method detection limits are the average MDLs for studies of three non-consecutive days. Daily studies were seven replicated analyses of 5 mL aliquots of 4 ppb soil. Daily MDLs were three times the standard deviation.

[c] Daily studies were seven replicated analyses of 10 g fish tissue spiked at 5 ppb. Daily MDLs were three times the standard deviation. Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[d] Method detection limits are estimated analyzing 1 g of cod liver oil samples spiked at 250 ppm. Five replicates were analyzed using Method 8260.

[e] No analyses.

[f] Contamination of sample by analyte prevented determination.

[g] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 25

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES
(METHOD 5032) (EXTERNAL STANDARD METHOD)[a]

Compound	Water[b] (µg/L)	Soil[c] (µg/kg)	Tissue[d] (µg/kg)	Oil[e] (mg/kg)
Chloromethane	3.1	8.6[f]	7.8	N/A[g]
Bromomethane	2.5	4.9[f]	9.7	N/A[g]
Vinyl chloride	4.0	7.1[f]	9.5	N/A[g]
Chloroethane	6.1	7.5[f]	9.2	N/A[g]
Methylene chloride	3.1	3.3	CONT[h]	0.08
Acetone	33.0[f]	CONT[h]	CONT[h]	0.12
Carbon disulfide	2.5	3.2	5.4	0.19
1,1-Dichloroethene	3.4	3.8	4.0	0.19
1,1-Dichloroethane	2.3	1.7	4.0	0.13
trans-1,2-Dichloroethene	3.0	3.2	4.4	0.09
cis-1,2-Dichloroethene	2.4	2.7	4.7	0.08
Chloroform	2.7	2.6	5.6	0.06
1,2-Dichloroethane	1.6	1.7	3.3	0.06
2-Butanone	57.0[f]	INT[i]	INT[i]	INT[i]
1,1,1-Trichloroethane	1.6	2.4	1.1	0.08
Carbon tetrachloride	1.5	1.7	3.2	0.15
Vinyl acetate	23.0[f]	INT[i]	INT[i]	INT[i]
Bromodichloromethane	2.0	2.3	3.2	0.05
1,1,2,2-Tetrachloroethane	3.6	3.2	4.4	0.09
1,2-Dichloropropane	2.9	3.7	3.8	0.12
trans-1,3-Dichloropropene	2.3	2.4	3.8	0.08
Trichloroethene	2.5	3.0	3.1	0.06
Dibromochloromethane	2.1	2.9	3.5	0.04
1,1,2-Trichloroethane	2.7	2.8	4.4	0.07
Benzene	1.7	2.9	3.6	0.03
cis-1,3-Dichloropropene	2.1	2.5	3.5	0.06
Bromoform	2.3	2.5	4.9	0.10
2-Hexanone	4.6	4.6	7.7	INT[i]
4-Methyl-2-pentanone	3.8	3.9	7.5	INT[i]
Tetrachloroethene	1.8	2.6	4.3	0.12
Toluene	1.8	4.4	3.0	0.09
Chlorobenzene	2.4	2.6	3.3	0.07
Ethylbenzene	2.4	4.1	3.6	0.09
Styrene	2.0	2.5	3.5	0.16
p-Xylene	2.3	3.9	3.7	0.18
o-Xylene	2.4	4.1	3.3	0.08

[a] Method detection limits are the average MDLs for studies on three non-consecutive days. Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb. Daily MDLs were three times the standard deviation.

[b] Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb.

[c] These studies were seven replicate analyses of 5-g aliquots of soil spiked at 4 ppb.

[d] These studies were seven replicate analyses of 10-g aliquots of fish tissue spiked at 5 ppb.

[e] Method detection limits were estimated by analyzing cod liver oil samples spiked at 250 ppb. Five replicates were analyzed using Method 8260.

[f] Method detection limits were estimated by analyzing replicate 50 ppb standards five times over a single day.

[g] No analyses.

[h] Contamination of sample by analyte prevented determination.

[l] Interference by co-eluting compound prevented accurate quantitation.

TABLE 26

VOLATILE ORGANIC ANALYTE RECOVERY FROM OIL
USING VACUUM DISTILLATION (METHOD 5032)[a]

Compound	Recovery Mean (%)	RSD (%)
Chloromethane	N/A[b]	
Bromomethane	N/A[b]	
Vinyl chloride	N/A[b]	
Chloroethane	N/A[b]	
Methylene chloride	62	32
Acetone	108	55
Carbon disulfide	98	46
1,1-Dichloroethene	97	24
1,1-Dichloroethane	96	22
trans-1,2-Trichloroethene	86	23
cis-1,2-Dichloroethene	99	11
Chloroform	93	14
1,2-Dichloroethane	138	31
2-Butanone	INT[c]	
1,1,1-Trichloroethane	89	14
Carbon tetrachloride	129	23
Vinyl acetate	INT[c]	
Bromodichloromethane	106	14
1,1,2,2-Tetrachloroethane	205	46
1,2-Dichloropropane	107	24
trans-1,3-Dichloropropene	98	13
Trichloroethene	102	8
Dibromochloromethane	168	21
1,1,2-Trichloroethane	95	7
Benzene	146	10
cis-1,3-Dichloropropene	98	11
Bromoform	94	18
2-Hexanone	INT[c]	
4-Methyl-2-pentanone	INT[c]	
Tetrachloroethene	117	22
Toluene	108	8
Chlorobenzene	101	12
Ethylbenzene	96	10
Styrene	120	46
p-Xylene	87	23
o-Xylene	90	10

TABLE 26 (cont.)

Compound	Recovery Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane	137	30
Toluene-d_8	84	6
Bromofluorobenzene	48	2

[a] Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicates of 10-g fish aliquots spiked at 25 ppb were analyzed. Quantitation was performed with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Standards and samples were replicated and precision value reflects the propagated errors. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

[b] Not analyzed.

[c] Interference by co-evaluating compounds prevented accurate measurement of analyte.

TABLE 27

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES IN OIL (METHOD 5032)[a]

Compound	Method Detection Limit (ppb) External Standard Method	Internal Standard Method
Chloromethane	N/A[b]	N/A[b]
Bromomethane	N/A[b]	N/A[b]
Vinyl chloride	N/A[b]	N/A[b]
Chloroethane	N/A[b]	N/A[b]
Methylene chloride	80	50
Acetone	120	60
Carbon disulfide	190	180
1,1-Dichloroethene	190	180
1,1-Dichloroethane	130	140
trans-1,2-Dichloroethene	90	100
cis-1,2-Dichloroethene	80	70
Chloroform	60	70
1,2-Dichloroethane	60	60
2-Butanone	INT[c]	INT[c]
1,1,1-Trichloroethane	80	100
Carbon tetrachloride	150	130
Vinyl acetate	INT[c]	INT[c]
Bromodichloromethane	50	60
1,1,2,2-Tetrachloroethane	90	20
1,2-Dichloropropane	120	150
trans-1,3-Dichloropropene	80	50
Trichloroethene	60	40
Dibromochloromethane	40	70
1,1,2-Trichloroethane	70	50
Benzene	30	50
cis-1,3-Dichloropropene	60	40
Bromoform	100	50
2-Hexanone	INT[c]	INT[c]
4-Methyl-2-pentanone	INT[c]	INT[c]
Tetrachloroethene	120	100
Toluene	90	50
Chlorobenzene	70	60
Ethylbenzene	90	40
Styrene	160	180
p-Xylene	180	200
o-Xylene	80	70

[a] Method detection limits are estimated as the result of five replicated analyses of 1 g cod liver oil spiked at 25 ppb. MDLs were calculated as three times the standard deviation. Quantitation was performed using a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[b] No analyses.

[c] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 28

INTERNAL STANDARDS FOR ANALYTES AND SURROGATES PREPARED USING EQUILIBRIUM HEADSPACE ANALYSIS (METHOD 5021)

Chloroform-d_1	1,1,2-TCA-d_3	Bromobenzene-d_5
Dichlorodifluoromethane	1,1,1-Trichloroethane	Chlorobenzene
Chloromethane	1,1-Dichloropropene	Bromoform
Vinyl chloride	Carbon tetrachloride	Styrene
Bromomethane	Benzene	iso-Propylbenzene
Chloroethane	Dibromomethane	Bromobenzene
Trichlorofluoromethane	1,2-Dichloropropane	n-Propylbenzene
1,1-Dichloroethene	Trichloroethene	2-Chlorotoluene
Methylene chloride	Bromodichloromethane	4-Chlorotoluene
trans-1,2-Dichloroethene	cis-1,3-Dichloropropene	1,3,5-Trimethylbenzene
1,1-Dichloroethane	trans-1,3-Dichloropropene	tert-Butylbenzene
cis-1,2-Dichloroethene	1,1,2-Trichloroethane	1,2,4-Trimethylbenzene
Bromochloromethane	Toluene	sec-Butylbenzene
Chloroform	1,3-Dichloropropane	1,3-Dichlorobenzene
2,2-Dichloropropane	Dibromochloromethane	1,4-Dichlorobenzene
1,2-Dichloroethane	1,2-Dibromoethane	p-iso-Propyltoluene
	Tetrachloroethene	1,2-Dichlorobenzene
	1,1,2-Trichloroethane	n-Butylbenzene
	Ethylbenzene	1,2-Dibromo-3-chloropropane
	m-Xylene	1,2,4-Trichlorobenzene
	p-Xylene	Naphthalene
	o-Xylene	Hexachlorobutadiene
	1,1,2,2-Tetrachloroethane	1,2,3-Trichlorobenzene
	1,2,3-Trichloropropane	

TABLE 29

PRECISION AND MDL DETERMINED FOR ANALYSIS OF FORTIFIED SAND[a] (METHOD 5021)

Compound	% RSD	MDL (µg/kg)
Benzene	3.0	0.34
Bromochloromethane	3.4	0.27
Bromodichloromethane	2.4	0.21
Bromoform	3.9	0.30
Bromomethane	11.6	1.3
Carbon tetrachloride	3.6	0.32
Chlorobenzene	3.2	0.24
Chloroethane	5.6	0.51
Chloroform	3.1	0.30
Chloromethane	4.1	3.5[b]
1,2-Dibromo-3-chloropropane	5.7	0.40
1,2-Dibromoethane	3.2	0.29
Dibromomethane	2.8	0.20
1,2-Dichlorobenzene	3.3	0.27
1,3-Dichlorobenzene	3.4	0.24
1,4-Dichlorobenzene	3.7	0.30
Dichlorodifluoromethane	3.0	0.28
1,1-Dichloroethane	4.5	0.41
1,2-Dichloroethane	3.0	0.24
1,1-Dichloroethene	3.3	0.28
cis-1,2-Dichloroethene	3.2	0.27
trans-1,2-Dichloroethene	2.6	0.22
1,2-Dichloropropane	2.6	0.21
1,1-Dichloropropene	3.2	0.30
cis-1,3-Dichloropropene	3.4	0.27
Ethylbenzene	4.8	0.47
Hexachlorobutadiene	4.1	0.38
Methylene chloride	8.2	0.62[c]
Naphthalene	16.8	3.4[c]
Styrene	7.9	0.62
1,1,1,2-Tetrachloroethane	3.6	0.27
1,1,2,2-Tetrachloroethane	2.6	0.20
Tetrachloroethene	9.8	1.2[c]
Toluene	3.5	0.38
1,2,4-Trichlorobenzene	4.2	0.44
1,1,1-Trichloroethane	2.7	0.27
1,1,2-Trichloroethane	2.6	0.20
Trichloroethene	2.3	0.19

TABLE 29 (cont.)

Compound	% RSD	MDL (µg/kg)
Trichlorofluoromethane	2.7	0.31
1,2,3-Trichloropropane	1.5	0.11
Vinyl chloride	4.8	0.45
m-Xylene/p-Xylene	3.6	0.37
o-Xylene	3.6	0.33

[a] Most compounds spiked at 2 ng/g (2 µg/kg)
[b] Incorrect ionization due to methanol
[c] Compound detected in unfortified sand at >1 ng

TABLE 30

RECOVERIES IN GARDEN SOIL FORTIFIED AT 20 µg/kg (ANALYSIS BY METHOD 5021)

Compound	Recovery per Replicate (ng) Sample 1	Sample 2	Sample 3	Mean (ng)	RSD	Recovery (%)
Benzene	37.6	35.2	38.4	37.1	3.7	185[a]
Bromochloromethane	20.5	19.4	20.0	20.0	2.3	100
Bromodichloromethane	21.1	20.3	22.8	21.4	4.9	107
Bromoform	23.8	23.9	25.1	24.3	2.4	121
Bromomethane	21.4	19.5	19.7	20.2	4.2	101
Carbon tetrachloride	27.5	26.6	28.6	27.6	3.0	138
Chlorobenzene	25.6	25.4	26.4	25.8	1.7	129
Chloroethane	25.0	24.4	25.3	24.9	1.5	125
Chloroform	21.9	20.9	21.7	21.5	2.0	108
Chloromethane	21.0	19.9	21.3	20.7	2.9	104[a]
1,2-Dibromo-3-chloro-propane	20.8	20.8	21.0	20.9	0.5	104
1,2-Dibromoethane	20.1	19.5	20.6	20.1	2.2	100
Dibromomethane	22.2	21.0	22.8	22.0	3.4	110
1,2-Dichlorobenzene	18.0	17.7	17.1	17.6	2.1	88.0
1,3-Dichlorobenzene	21.2	21.0	20.1	20.8	2.3	104
1,4-Dichlorobenzene	20.1	20.9	19.9	20.3	2.1	102
Dichlorodifluoromethane	25.3	24.1	25.4	24.9	2.4	125
1,1-Dichloroethane	23.0	22.0	22.7	22.6	1.9	113
1,2-Dichloroethane	20.6	19.5	19.8	20.0	2.3	100
1,1-Dichloroethene	24.8	23.8	24.4	24.3	1.7	122
cis-1,2-Dichloroethene	21.6	20.0	21.6	21.1	3.6	105
trans-1,2-Dichloroethene	22.4	21.4	22.2	22.0	2.0	110
1,2-Dichloropropane	22.8	22.2	23.4	22.8	2.1	114
1,1-Dichloropropene	26.3	25.7	28.0	26.7	3.7	133
cis-1,3-Dichloropropene	20.3	19.5	21.1	20.3	3.2	102
Ethylbenzene	24.7	24.5	25.5	24.9	1.7	125
Hexachlorobutadiene	23.0	25.3	25.2	24.5	4.3	123
Methylene chloride	26.0	25.7	26.1	25.9	0.7	130[a]
Naphthalene	13.8	12.7	11.8	12.8	6.4	63.8[a]
Styrene	24.2	23.3	23.3	23.6	1.8	118
1,1,1,2-Tetrachloroethane	21.4	20.2	21.3	21.0	2.6	105
1,1,2,2-Tetrachloroethane	18.6	17.8	19.0	18.5	2.7	92.3
Tetrachloroethene	25.2	24.8	26.4	25.5	2.7	127
Toluene	28.6	27.9	30.9	29.1	4.4	146[a]
1,2,4-Trichlorobenzene	15.0	14.4	12.9	14.1	6.3	70.5
1,1,1-Trichloroethane	28.1	27.2	29.9	28.4	4.0	142
1,1,2-Trichloroethane	20.8	19.6	21.7	20.7	4.2	104

TABLE 30 (cont.)

Compound	Recovery per Replicate (ng) Sample 1	Sample 2	Sample 3	Mean (ng)	RSD	Recovery (%)
Trichloroethene	26.3	24.9	26.8	26.0	3.1	130
Trichlorofluoromethane	25.9	24.8	26.5	25.7	2.7	129
1,2,3-Trichloropropane	18.8	18.3	19.3	18.8	2.2	94.0
Vinyl chloride	24.8	23.2	23.9	24.0	2.7	120
m-Xylene/p-Xylene	24.3	23.9	25.3	24.5	2.4	123
o-Xylene	23.1	22.3	23.4	22.9	2.0	115

[a] Compound found in unfortified garden soil matrix at >5 ng.

TABLE 31

METHOD DETECTION LIMITS AND BOILING POINTS
FOR VOLATILE ORGANICS (ANALYSIS BY METHOD 5041)[a]

Compound	Detection Limit (ng)	Boiling Point (°C)
Chloromethane	58	-24
Bromomethane	26	4
Vinyl chloride	14	-13
Chloroethane	21	13
Methylene chloride	9	40
Acetone	35	56
Carbon disulfide	11	46
1,1-Dichloroethene	14	32
1,1-Dichloroethane	12	57
trans-1,2-Dichloroethene	11	48
Chloroform	11	62
1,2-Dichloroethane	13	83
1,1,1-Trichloroethane	8	74
Carbon tetrachloride	8	77
Bromodichloromethane	11	88
1,1,2,2-Tetrachloroethane**	23	146
1,2-Dichloropropane	12	95
trans-1,3-Dichloropropene	17	112
Trichloroethene	11	87
Dibromochloromethane	21	122
1,1,2-Trichloroethane	26	114
Benzene	26	80
cis-1,3-Dichloropropene	27	112
Bromoform**	26	150
Tetrachloroethene	11	121
Toluene	15	111
Chlorobenzene	15	132
Ethylbenzene**	21	136
Styrene**	46	145
Trichlorofluoromethane	17	24
Iodomethane	9	43
Acrylonitrile	13	78
Dibromomethane	14	97
1,2,3-Trichloropropane**	37	157
total Xylenes**	22	138-144

Footnotes are found on the following page.

* The method detection limit (MDL) is defined in Chapter One. The detection limits cited above were determined according to 40 CFR, Part 136, Appendix B, using standards spiked onto clean VOST tubes. Since clean VOST tubes were used, the values cited above represent the best that the methodology can achieve. The presence of an emissions matrix will affect the ability of the methodology to perform at its optimum level.

** Boiling Point greater than 130°C. Not appropriate for quantitative sampling by Method 0030.

TABLE 32

VOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION (METHOD 5041)

Bromochloromethane	1,4-Difluorobenzene
Acetone	Benzene
Acrylonitrile	Bromodichloromethane
Bromomethane	Bromoform
Carbon disulfide	Carbon tetrachloride
Chloroethane	Chlorodibromomethane
Chloroform	Dibromomethane
Chloromethane	1,2-Dichloropropane
1,1-Dichloroethane	cis-1,3-Dichloropropene
1,2-Dichloroethane	trans-1,3-Dichloropropene
1,2-Dichloroethane-d_4 (surrogate)	1,1,1-Trichloroethane
1,1-Dichloroethene	1,1,2-Trichloroethane
Trichloroethene	
trans-1,2-Dichloroethene	
Iodomethane	
Methylene chloride	
Trichlorofluoromethane	
Vinyl chloride	

Chlorobenzene-d_5

4-Bromofluorobenzene (surrogate)
Chlorobenzene
Ethylbenzene
Styrene
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
Toluene-d_8 (surrogate)
1,2,3-Trichloropropane
Xylenes

TABLE 33

METHOD 0040 - COMPOUNDS DEMONSTRATED TO BE APPLICABLE TO THE METHOD

Compound	Boiling Point (°C)	Condensation Point at 20°C (%)	Estimated Detection Limit[a] (ppm)
Dichlorodifluoromethane	-30	Gas	0.20
Vinyl chloride	-19	Gas	0.11
1,3-Butadiene	-4	Gas	0.90
1,2-Dichloro-1,1,2,2-tetrafluoroethane	4	Gas	0.14
Methyl bromide	4	Gas	0.14
Trichlorofluoromethane	24	88	0.18
1,1-Dichloroethene	31	22	0.07
Methylene chloride	40	44	0.05
1,1,2-Trichloro-trifluoroethane	48	37	0.13
Chloroform	61	21	0.04
1,1,1-Trichloroethane	75	13	0.03
Carbon tetrachloride	77	11	0.03
Benzene	80	10	0.16
Trichloroethene	87	8	0.04
1,2-Dichloropropane	96	5	0.05
Toluene	111	3	0.08
Tetrachloroethene	121	2	0.03

[a] Since this value represents a direct injection (no concentration) from the Tedlar® bag, these values are directly applicable as stack detection limits.

FIGURE 1
GAS CHROMATOGRAM OF VOLATILE ORGANICS

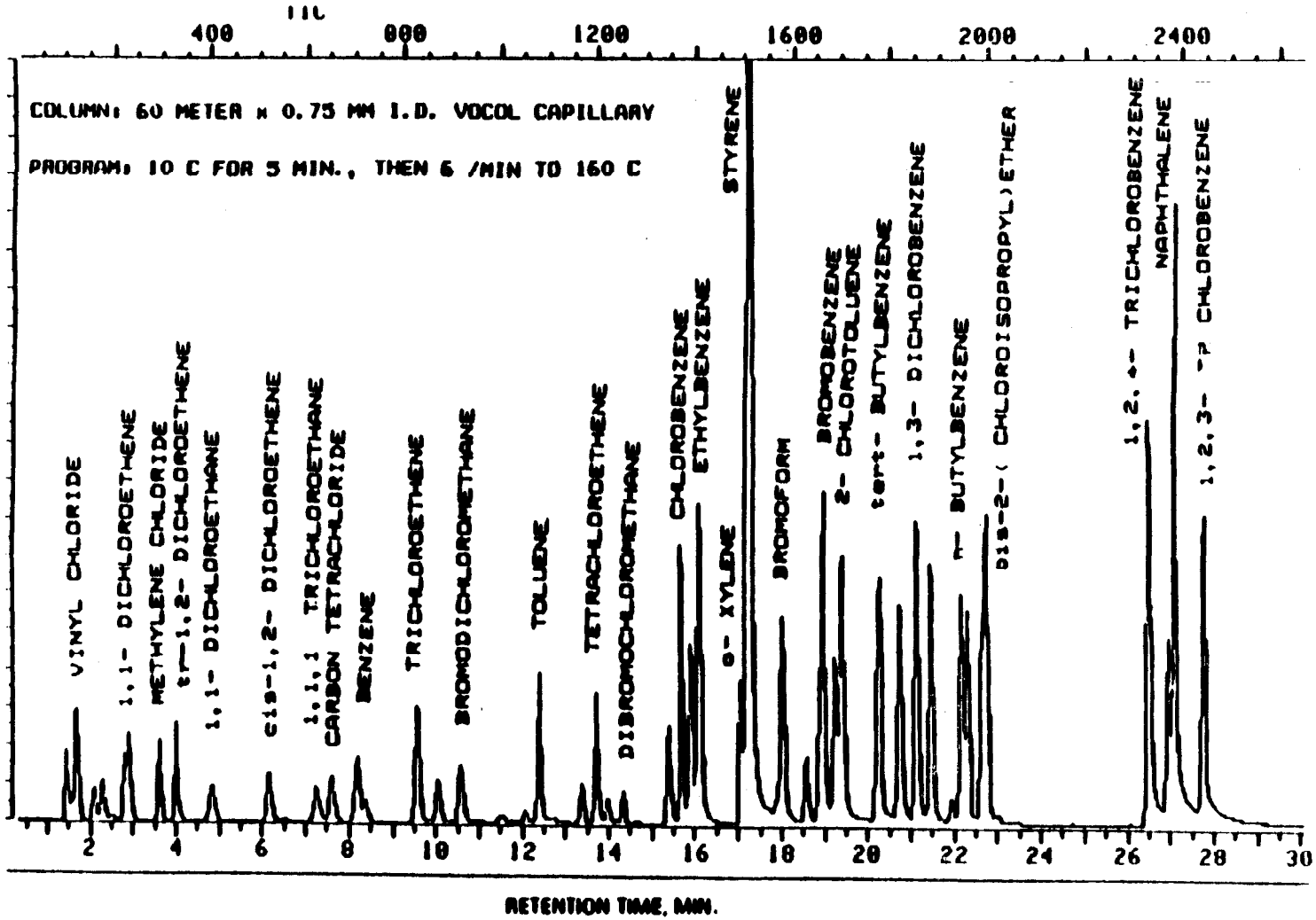


FIGURE 2
GAS CHROMATOGRAM OF VOLATILE ORGANICS

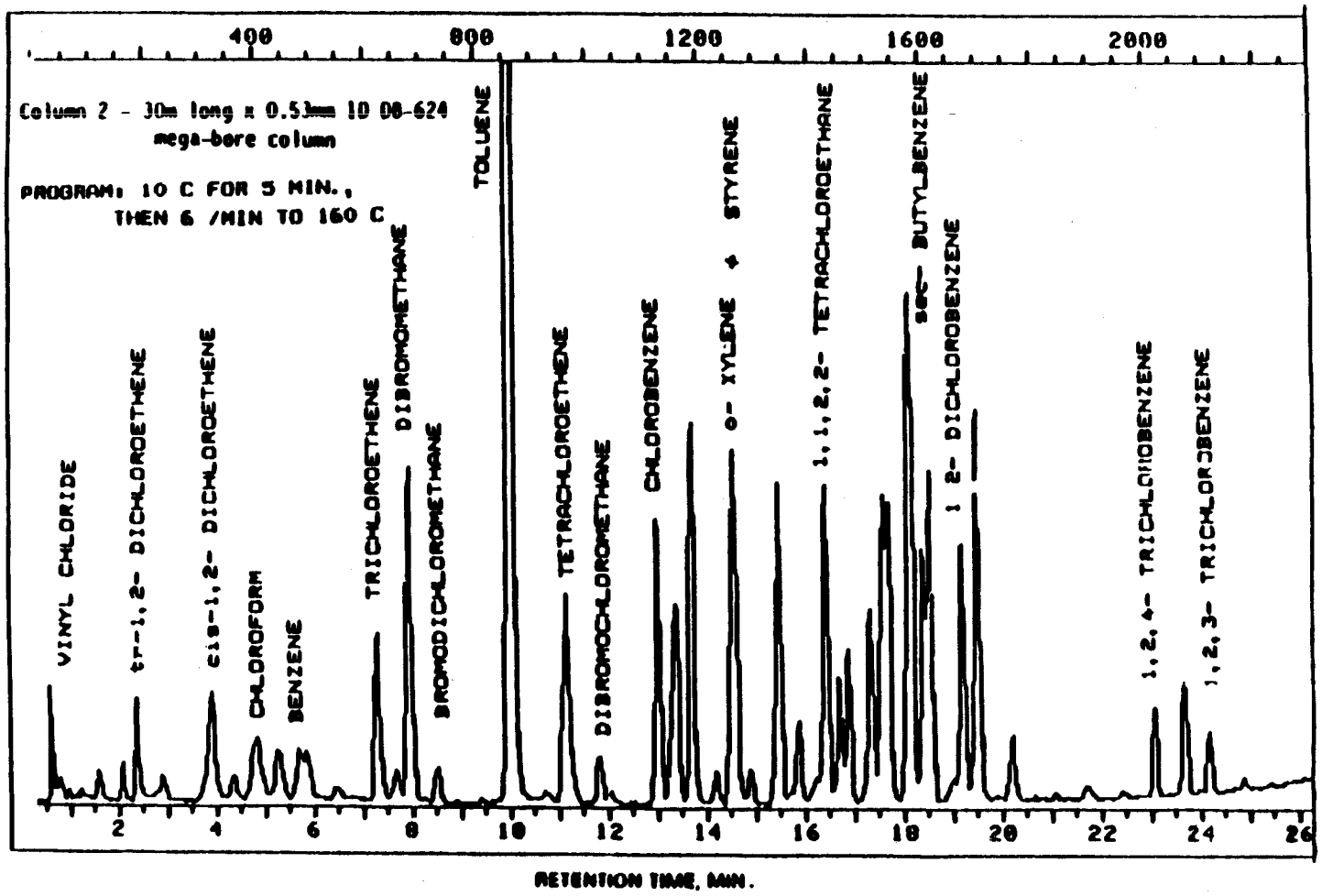


FIGURE 3
GAS CHROMATOGRAM OF VOLATILE ORGANICS

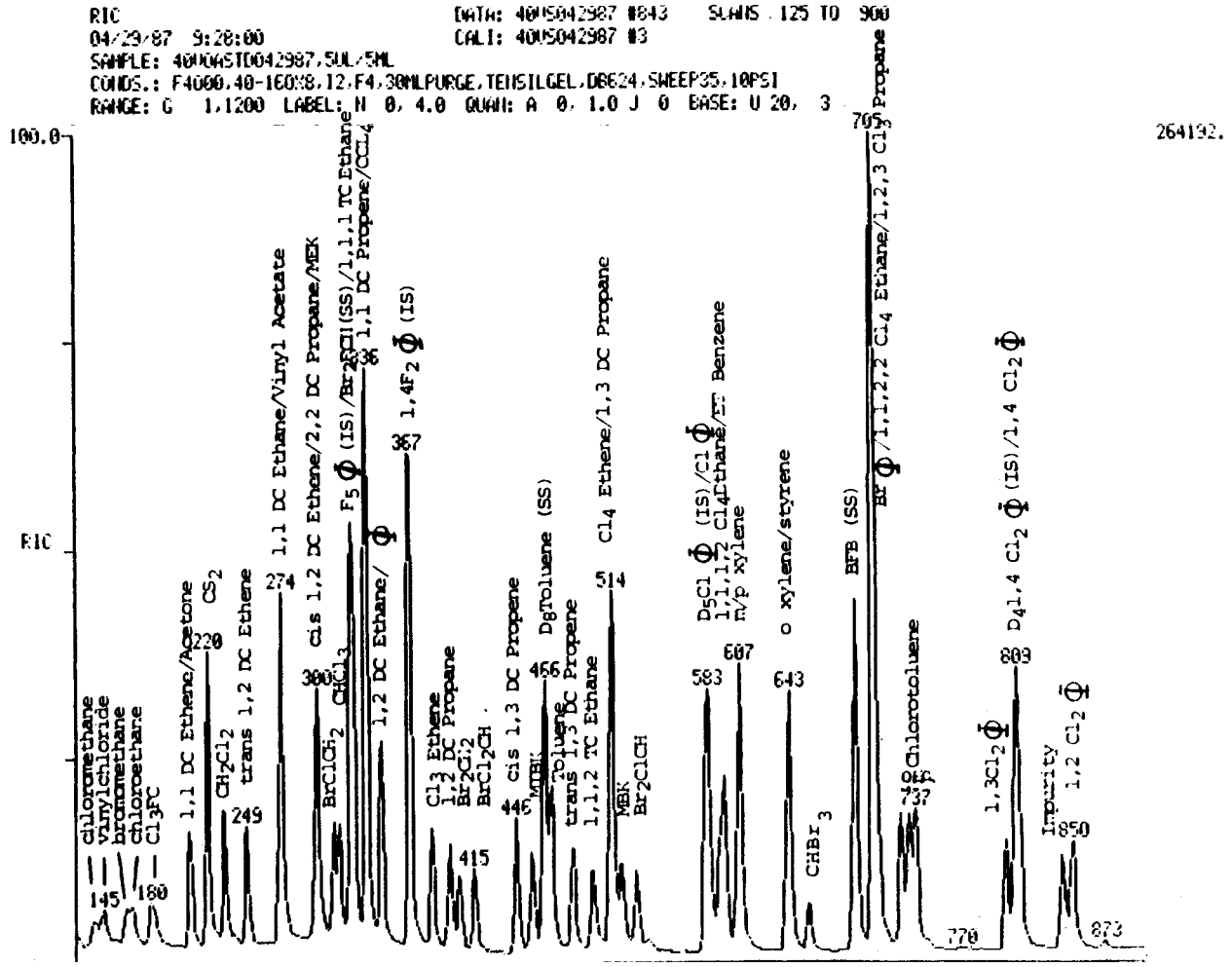
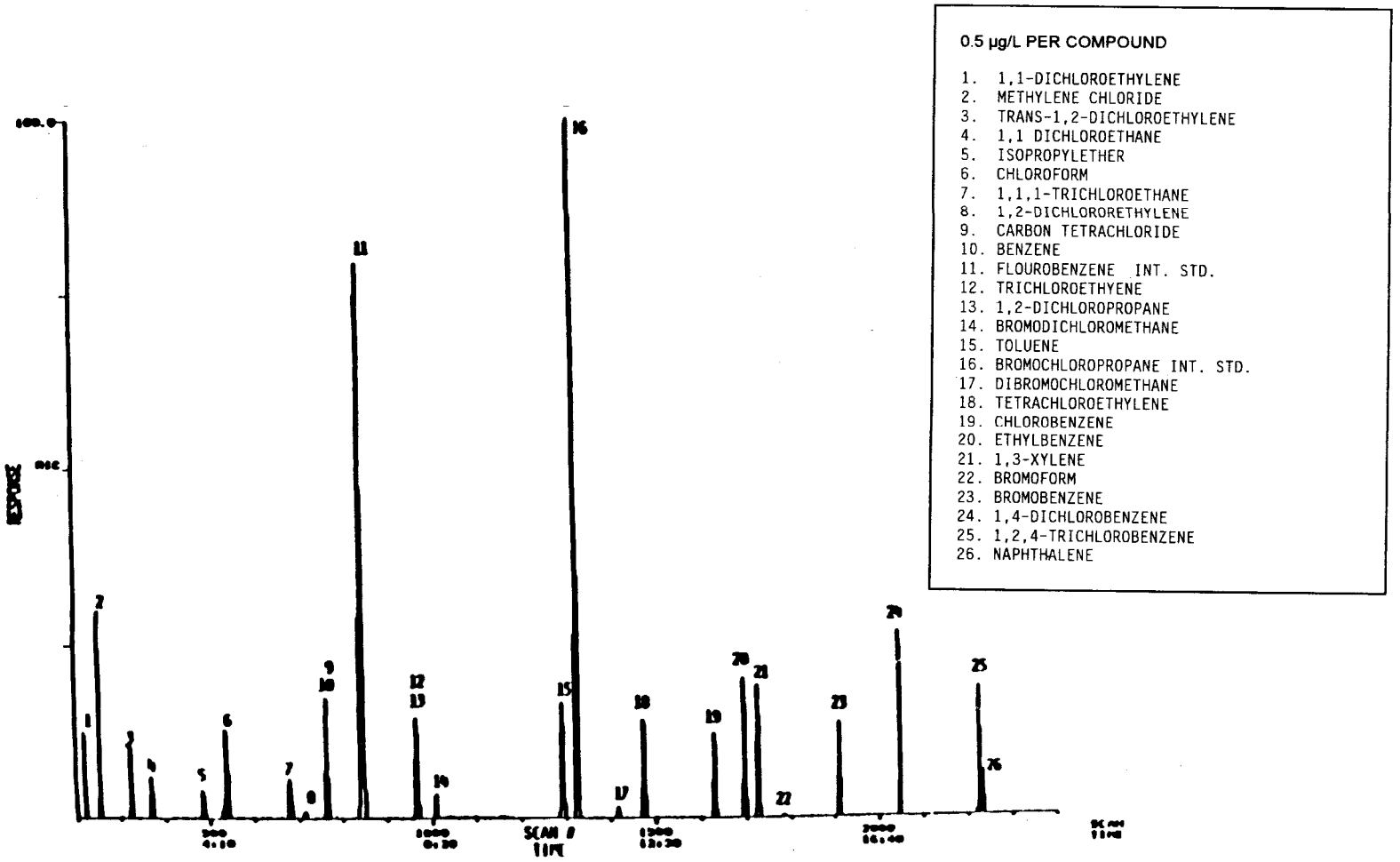
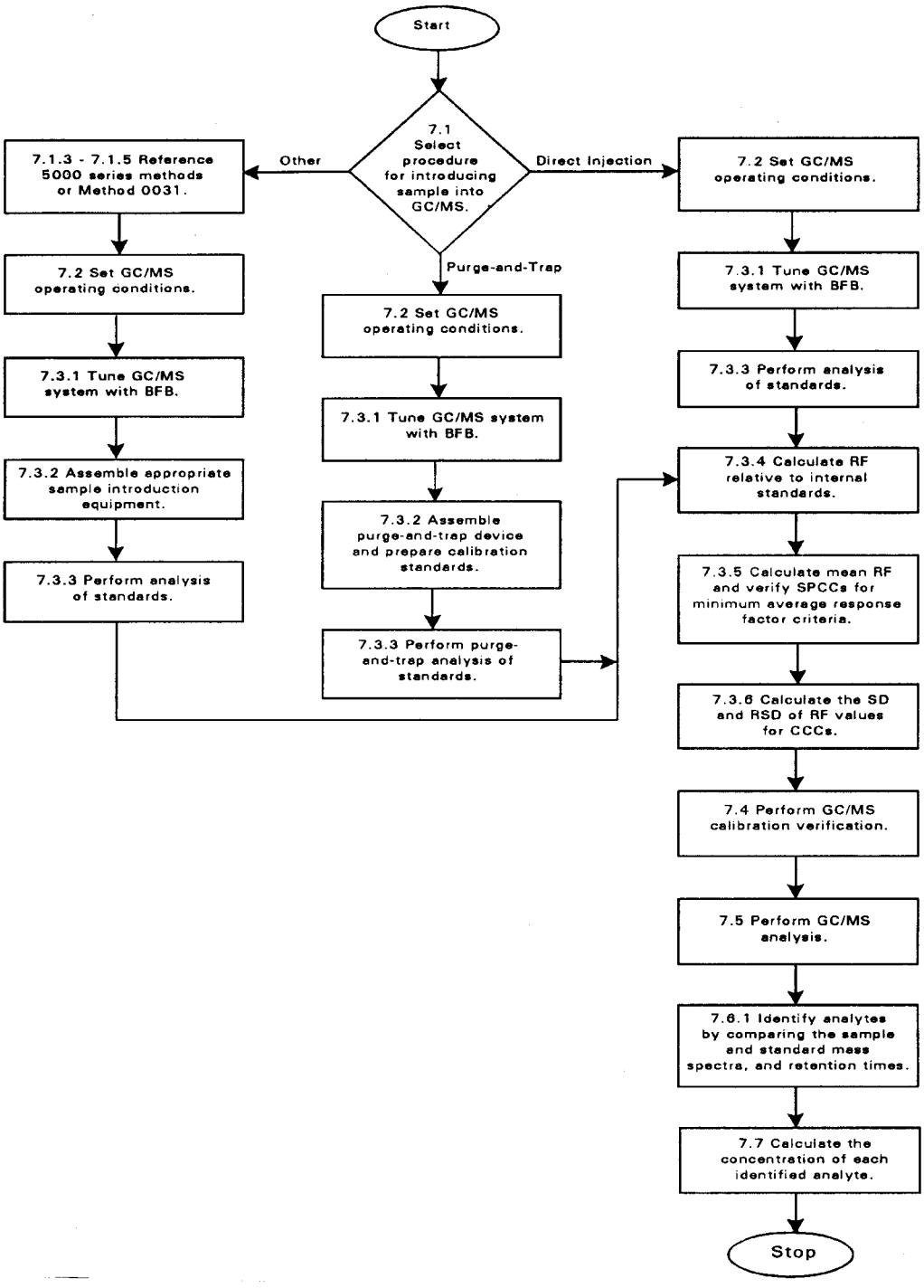


FIGURE 4
GAS CHROMATOGRAM OF TEST MIXTURE




Start
7.1 Select procedure for introducing sample into GC/MS.
Other
Purge-and-Trap
Direct Injection
7.1.3 - 7.1.5 Reference 5000 series methods or Method 0031.
7.2 Set GC/MS operating conditions.
7.3.1 Tune GC/MS system with BFB.
7.3.2 Assemble appropriate sample introduction equipment.
7.3.3 Perform analysis of standards.
7.2 Set GC/MS operating conditions.
7.3.1 Tune GC/MS system with BFB.
7.3.2 Assemble purge-and-trap device and prepare calibration standards.
7.3.3 Perform purge-and-trap analysis of standards.
7.2 Set GC/MS operating conditions.
7.3.1 Tune GC/MS system with BFB.
7.3.3 Perform analysis of standards.
7.3.4 Calculate RF relative to internal standards.
7.3.5 Calculate mean RF and verify SPCCs for minimum average response factor criteria.
7.3.6 Calculate the SD and RSD of RF values for CCCs.
7.4 Perform GC/MS calibration verification.
7.5 Perform GC/MS analysis.
7.6.1 Identify analytes by comparing the sample and standard mass spectra, and retention times.
7.7 Calculate the concentration of each identified analyte.
Stop

Appendix B
EPA Test Method 8270 C

METHOD 8270C

SEMIVOLATILE ORGANIC COMPOUNDS
BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. Direct injection of a sample may be used in limited applications. The following compounds can be determined by this method:

		Appropriate Preparation Techniques[b]				
Compounds	CAS No[a]	3510	3520	3540/ 3541	3550	3580
Acenaphthene	83-32-9	X	X	X	X	X
Acenaphthene-d_{10} (IS)		X	X	X	X	X
Acenaphthylene	208-96-8	X	X	X	X	X
Acetophenone	98-86-2	X	ND	ND	ND	X
2-Acetylaminofluorene	53-96-3	X	ND	ND	ND	X
1-Acetyl-2-thiourea	591-08-2	LR	ND	ND	ND	LR
Aldrin	309-00-2	X	X	X	X	X
2-Aminoanthraquinone	117-79-3	X	ND	ND	ND	X
Aminoazobenzene	60-09-3	X	ND	ND	ND	X
4-Aminobiphenyl	92-67-1	X	ND	ND	ND	X
3-Amino-9-ethylcarbazole	132-32-1	X	X	ND	ND	ND
Anilazine	101-05-3	X	ND	ND	ND	X
Aniline	62-53-3	X	X	ND	X	X
o-Anisidine	90-04-0	X	ND	ND	ND	X
Anthracene	120-12-7	X	X	X	X	X
Aramite	140-57-8	HS(43)	ND	ND	ND	X
Aroclor 1016	12674-11-2	X	X	X	X	X
Aroclor 1221	11104-28-2	X	X	X	X	X
Aroclor 1232	11141-16-5	X	X	X	X	X
Aroclor 1242	53469-21-9	X	X	X	X	X
Aroclor 1248	12672-29-6	X	X	X	X	X
Aroclor 1254	11097-69-1	X	X	X	X	X
Aroclor 1260	11096-82-5	X	X	X	X	X
Azinphos-methyl	86-50-0	HS(62)	ND	ND	ND	X
Barban	101-27-9	LR	ND	ND	ND	LR
Benzidine	92-87-5	CP	CP	CP	CP	CP
Benzoic acid	65-85-0	X	X	ND	X	X
Benz(a)anthracene	56-55-3	X	X	X	X	X
Benzo(b)fluoranthene	205-99-2	X	X	X	X	X
Benzo(k)fluoranthene	207-08-9	X	X	X	X	X
Benzo(g,h,i)perylene	191-24-2	X	X	X	X	X
Benzo(a)pyrene	50-32-8	X	X	X	X	X

Compounds	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
p-Benzoquinone	106-51-4	OE	ND	ND	ND	X
Benzyl alcohol	100-51-6	X	X	ND	X	X
α-BHC	319-84-6	X	X	X	X	X
β-BHC	319-85-7	X	X	X	X	X
δ-BHC	319-86-8	X	X	X	X	X
γ-BHC (Lindane)	58-89-9	X	X	X	X	X
Bis(2-chloroethoxy)methane	111-91-1	X	X	X	X	X
Bis(2-chloroethyl) ether	111-44-4	X	X	X	X	X
Bis(2-chloroisopropyl) ether	108-60-1	X	X	X	X	X
Bis(2-ethylhexyl) phthalate	117-81-7	X	X	X	X	X
4-Bromophenyl phenyl ether	101-55-3	X	X	X	X	X
Bromoxynil	1689-84-5	X	ND	ND	ND	X
Butyl benzyl phthalate	85-68-7	X	X	X	X	X
Captafol	2425-06-1	HS(55)	ND	ND	ND	X
Captan	133-06-2	HS(40)	ND	ND	ND	X
Carbaryl	63-25-2	X	ND	ND	ND	X
Carbofuran	1563-66-2	X	ND	ND	ND	X
Carbophenothion	786-19-6	X	ND	ND	ND	X
Chlordane (NOS)	57-74-9	X	X	X	X	X
Chlorfenvinphos	470-90-6	X	ND	ND	ND	X
4-Chloroaniline	106-47-8	X	ND	ND	ND	X
Chlorobenzilate	510-15-6	X	ND	ND	ND	X
5-Chloro-2-methylaniline	95-79-4	X	ND	ND	ND	X
4-Chloro-3-methylphenol	59-50-7	X	X	X	X	X
3-(Chloromethyl)pyridine hydrochloride	6959-48-4	X	ND	ND	ND	X
1-Chloronaphthalene	90-13-1	X	X	X	X	X
2-Chloronaphthalene	91-58-7	X	X	X	X	X
2-Chlorophenol	95-57-8	X	X	X	X	X
4-Chloro-1,2-phenylenediamine	95-83-0	X	X	ND	ND	ND
4-Chloro-1,3-phenylenediamine	5131-60-2	X	X	ND	ND	ND
4-Chlorophenyl phenyl ether	7005-72-3	X	X	X	X	X
Chrysene	218-01-9	X	X	X	X	X
Chrysene-d_{12} (IS)		X	X	X	X	X
Coumaphos	56-72-4	X	ND	ND	ND	X
p-Cresidine	120-71-8	X	ND	ND	ND	X
Crotoxyphos	7700-17-6	X	ND	ND	ND	X
2-Cyclohexyl-4,6-dinitro-phenol	131-89-5	X	ND	ND	ND	LR
4,4'-DDD	72-54-8	X	X	X	X	X
4,4'-DDE	72-55-9	X	X	X	X	X
4,4'-DDT	50-29-3	X	X	X	X	X
Demeton-O	298-03-3	HS(68)	ND	ND	ND	X
Demeton-S	126-75-0	X	ND	ND	ND	X
Diallate (cis or trans)	2303-16-4	X	ND	ND	ND	X

Compounds	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
2,4-Diaminotoluene	95-80-7	DC,0E(42)	ND	ND	ND	X
Dibenz(a,j)acridine	224-42-0	X	ND	ND	ND	X
Dibenz(a,h)anthracene	53-70-3	X	X	X	X	X
Dibenzofuran	132-64-9	X	X	ND	X	X
Dibenzo(a,e)pyrene	192-65-4	ND	ND	ND	ND	X
1,2-Dibromo-3-chloropropane	96-12-8	X	X	ND	ND	ND
Di-n-butyl phthalate	84-74-2	X	X	X	X	X
Dichlone	117-80-6	OE	ND	ND	ND	X
1,2-Dichlorobenzene	95-50-1	X	X	X	X	X
1,3-Dichlorobenzene	541-73-1	X	X	X	X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X	X
1,4-Dichlorobenzene-d_4 (IS)		X	X	X	X	X
3,3'-Dichlorobenzidine	91-94-1	X	X	X	X	X
2,4-Dichlorophenol	120-83-2	X	X	X	X	X
2,6-Dichlorophenol	87-65-0	X	ND	ND	ND	X
Dichlorovos	62-73-7	X	ND	ND	ND	X
Dicrotophos	141-66-2	X	ND	ND	ND	X
Dieldrin	60-57-1	X	X	X	X	X
Diethyl phthalate	84-66-2	X	X	X	X	X
Diethylstilbestrol	56-53-1	AW,0S(67)	ND	ND	ND	X
Diethyl sulfate	64-67-5	LR	ND	ND	ND	LR
Dihydrosaffrole	56312-13-1	ND	ND	ND	ND	ND
Dimethoate	60-51-5	HE,HS(31)	ND	ND	ND	X
3,3'-Dimethoxybenzidine	119-90-4	X	ND	ND	ND	LR
Dimethylaminoazobenzene	60-11-7	X	ND	ND	ND	X
7,12-Dimethylbenz(a)-anthracene	57-97-6	CP(45)	ND	ND	ND	CP
3,3'-Dimethylbenzidine	119-93-7	X	ND	ND	ND	X
α,α-Dimethylphenethylamine	122-09-8	ND	ND	ND	ND	X
2,4-Dimethylphenol	105-67-9	X	X	X	X	X
Dimethyl phthalate	131-11-3	X	X	X	X	X
1,2-Dinitrobenzene	528-29-0	X	ND	ND	ND	X
1,3-Dinitrobenzene	99-65-0	X	ND	ND	ND	X
1,4-Dinitrobenzene	100-25-4	HE(14)	ND	ND	ND	X
4,6-Dinitro-2-methylphenol	534-52-1	X	X	X	X	X
2,4-Dinitrophenol	51-28-5	X	X	X	X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X	X	X	X	X
Dinocap	39300-45-3	CP,HS(28)	ND	ND	ND	CP
Dinoseb	88-85-7	X	ND	ND	ND	X
Dioxathion	78-34-2	ND	ND	ND	ND	ND
Diphenylamine	122-39-4	X	X	X	X	X
5,5-Diphenylhydantoin	57-41-0	X	ND	ND	ND	X
1,2-Diphenylhydrazine	122-66-7	X	X	X	X	X

Compounds	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
Di-n-octyl phthalate	117-84-0	X	X	X	X	X
Disulfoton	298-04-4	X	ND	ND	ND	X
Endosulfan I	959-98-8	X	X	X	X	X
Endosulfan II	33213-65-9	X	X	X	X	X
Endosulfan sulfate	1031-07-8	X	X	X	X	X
Endrin	72-20-8	X	X	X	X	X
Endrin aldehyde	7421-93-4	X	X	X	X	X
Endrin ketone	53494-70-5	X	X	ND	X	X
EPN	2104-64-5	X	ND	ND	ND	X
Ethion	563-12-2	X	ND	ND	ND	X
Ethyl carbamate	51-79-6	DC(28)	ND	ND	ND	X
Ethyl methanesulfonate	62-50-0	X	ND	ND	ND	X
Famphur	52-85-7	X	ND	ND	ND	X
Fensulfothion	115-90-2	X	ND	ND	ND	X
Fenthion	55-38-9	X	ND	ND	ND	X
Fluchloralin	33245-39-5	X	ND	ND	ND	X
Fluoranthene	206-44-0	X	X	X	X	X
Fluorene	86-73-7	X	X	X	X	X
2-Fluorobiphenyl (surr)	321-60-8	X	X	X	X	X
2-Fluorophenol (surr)	367-12-4	X	X	X	X	X
Heptachlor	76-44-8	X	X	X	X	X
Heptachlor epoxide	1024-57-3	X	X	X	X	X
Hexachlorobenzene	118-74-1	X	X	X	X	X
Hexachlorobutadiene	87-68-3	X	X	X	X	X
Hexachlorocyclopentadiene	77-47-4	X	X	X	X	X
Hexachloroethane	67-72-1	X	X	X	X	X
Hexachlorophene	70-30-4	AW,CP(62)	ND	ND	ND	CP
Hexachloropropene	1888-71-7	X	ND	ND	ND	X
Hexamethylphosphoramide	680-31-9	X	ND	ND	ND	X
Hydroquinone	123-31-9	ND	ND	ND	ND	X
Indeno(1,2,3-cd)pyrene	193-39-5	X	X	X	X	X
Isodrin	465-73-6	X	ND	ND	ND	X
Isophorone	78-59-1	X	X	X	X	X
Isosafrole	120-58-1	DC(46)	ND	ND	ND	X
Kepone	143-50-0	X	ND	ND	ND	X
Leptophos	21609-90-5	X	ND	ND	ND	X
Malathion	121-75-5	HS(5)	ND	ND	ND	X
Maleic anhydride	108-31-6	HE	ND	ND	ND	X
Mestranol	72-33-3	X	ND	ND	ND	X
Methapyrilene	91-80-5	X	ND	ND	ND	X
Methoxychlor	72-43-5	X	ND	ND	ND	X
3-Methylcholanthrene	56-49-5	X	ND	ND	ND	X
4,4'-Methylenebis (2-chloroaniline)	101-14-4	OE,OS(0)	ND	ND	ND	LR

Compounds	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
4,4'-Methylenebis (N,N-dimethylaniline)	101-61-1	X	X	ND	ND	ND
Methyl methanesulfonate	66-27-3	X	ND	ND	ND	X
2-Methylnaphthalene	91-57-6	X	X	ND	X	X
Methyl parathion	298-00-0	X	ND	ND	ND	X
2-Methylphenol	95-48-7	X	ND	ND	ND	X
3-Methylphenol	108-39-4	X	ND	ND	ND	X
4-Methylphenol	106-44-5	X	ND	ND	ND	X
Mevinphos	7786-34-7	X	ND	ND	ND	X
Mexacarbate	315-18-4	HE,HS(68)	ND	ND	ND	X
Mirex	2385-85-5	X	ND	ND	ND	X
Monocrotophos	6923-22-4	HE	ND	ND	ND	X
Naled	300-76-5	X	ND	ND	ND	X
Naphthalene	91-20-3	X	X	X	X	X
Naphthalene-d_8 (IS)		X	X	X	X	X
1,4-Naphthoquinone	130-15-4	X	ND	ND	ND	X
1-Naphthylamine	134-32-7	OS(44)	ND	ND	ND	X
2-Naphthylamine	91-59-8	X	ND	ND	ND	X
Nicotine	54-11-5	DE(67)	ND	ND	ND	X
5-Nitroacenaphthene	602-87-9	X	ND	ND	ND	X
2-Nitroaniline	88-74-4	X	X	ND	X	X
3-Nitroaniline	99-09-2	X	X	ND	X	X
4-Nitroaniline	100-01-6	X	X	ND	X	X
5-Nitro-o-anisidine	99-59-2	X	ND	ND	ND	X
Nitrobenzene	98-95-3	X	X	X	X	X
Nitrobenzene-d_5 (surr)		X	X	X	X	X
4-Nitrobiphenyl	92-93-3	X	ND	ND	ND	X
Nitrofen	1836-75-5	X	ND	ND	ND	X
2-Nitrophenol	88-75-5	X	X	X	X	X
4-Nitrophenol	100-02-7	X	X	X	X	X
5-Nitro-o-toluidine	99-55-8	X	X	ND	ND	X
Nitroquinoline-1-oxide	56-57-5	X	ND	ND	ND	X
N-Nitrosodi-n-butylamine	924-16-3	X	ND	ND	ND	X
N-Nitrosodiethylamine	55-18-5	X	ND	ND	ND	X
N-Nitrosodimethylamine	62-75-9	X	X	X	X	X
N-Nitrosomethylethylamine	10595-95-6	X	ND	ND	ND	X
N-Nitrosodiphenylamine	86-30-6	X	X	X	X	X
N-Nitrosodi-n-propylamine	621-64-7	X	X	X	X	X
N-Nitrosomorpholine	59-89-2	ND	ND	ND	ND	X
N-Nitrosopiperidine	100-75-4	X	ND	ND	ND	X
N-Nitrosopyrrolidine	930-55-2	X	ND	ND	ND	X
Octamethyl pyrophosphoramide	152-16-9	LR	ND	ND	ND	LR
4,4'-Oxydianiline	101-80-4	X	ND	ND	ND	X

Compounds	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
Parathion	56-38-2	X	X	ND	ND	X
Pentachlorobenzene	608-93-5	X	ND	ND	ND	X
Pentachloronitrobenzene	82-68-8	X	ND	ND	ND	X
Pentachlorophenol	87-86-5	X	X	X	X	X
Perylene-d_{12} (IS)		X	X	X	X	X
Phenacetin	62-44-2	X	ND	ND	ND	X
Phenanthrene	85-01-8	X	X	X	X	X
Phenanthrene-d_{10} (IS)		X	X	X	X	X
Phenobarbital	50-06-6	X	ND	ND	ND	X
Phenol	108-95-2	DC(28)	X	X	X	X
Phenol-d_6 (surr)		DC(28)	X	X	X	X
1,4-Phenylenediamine	106-50-3	X	ND	ND	ND	X
Phorate	298-02-2	X	ND	ND	ND	X
Phosalone	2310-17-0	HS(65)	ND	ND	ND	X
Phosmet	732-11-6	HS(15)	ND	ND	ND	X
Phosphamidon	13171-21-6	HE(63)	ND	ND	ND	X
Phthalic anhydride	85-44-9	CP,HE(1)	ND	ND	ND	CP
2-Picoline (2-Methylpyridine)	109-06-8	X	X	ND	ND	ND
Piperonyl sulfoxide	120-62-7	X	ND	ND	ND	X
Pronamide	23950-58-5	X	ND	ND	ND	X
Propylthiouracil	51-52-5	LR	ND	ND	ND	LR
Pyrene	129-00-0	X	X	X	X	X
Pyridine	110-86-1	ND	ND	ND	ND	ND
Resorcinol	108-46-3	DC,OE(10)	ND	ND	ND	X
Safrole	94-59-7	X	ND	ND	ND	X
Strychnine	57-24-9	AW,0S(55)	ND	ND	ND	X
Sulfallate	95-06-7	X	ND	ND	ND	X
Terbufos	13071-79-9	X	ND	ND	ND	X
Terphenyl-d_{14}(surr)	1718-51-0	X	X	ND	X	X
1,2,4,5-Tetrachlorobenzene	95-94-3	X	ND	ND	ND	X
2,3,4,6-Tetrachlorophenol	58-90-2	X	ND	ND	ND	X
Tetrachlorvinphos	961-11-5	X	ND	ND	ND	X
Tetraethyl dithiopyrophosphate	3689-24-5	X	X	ND	ND	ND
Tetraethyl pyrophosphate	107-49-3	X	ND	ND	ND	X
Thionazine	297-97-2	X	ND	ND	ND	X
Thiophenol (Benzenethiol)	108-98-5	X	ND	ND	ND	X
Toluene diisocyanate	584-84-9	HE(6)	ND	ND	ND	X
o-Toluidine	95-53-4	X	ND	ND	ND	X
Toxaphene	8001-35-2	X	X	X	X	X
2,4,6-Tribromophenol (surr)	118-79-6	X	X	X	X	X
1,2,4-Trichlorobenzene	120-82-1	X	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	ND	X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X	X
Trifluralin	1582-09-8	X	ND	ND	ND	X

Compounds	CAS No[a]	Appropriate Preparation Techniques[b] 3510	3520	3540/ 3541	3550	3580
2,4,5-Trimethylaniline	137-17-7	X	ND	ND	ND	X
Trimethyl phosphate	512-56-1	HE(60)	ND	ND	ND	X
1,3,5-Trinitrobenzene	99-35-4	X	ND	ND	ND	X
Tris(2,3-dibromopropyl) phosphate	126-72-7	X	ND	ND	ND	LR
Tri-p-tolyl phosphate	78-32-0	X	ND	ND	ND	X
O,O,O-Triethyl phosphorothioate	126-68-1	X	ND	ND	ND	X

[a] Chemical Abstract Service Registry Number
[b] See Sec. 1.2 for other acceptable preparation methods.

KEY TO ANALYTE LIST

IS = This compound may be used as an internal standard.
surr = This compound may be used as a surrogate.
AW = Adsorption to walls of glassware during extraction and storage.
CP = Nonreproducible chromatographic performance.
DC = Unfavorable distribution coefficient (number in parenthesis is percent recovery).
HE = Hydrolysis during extraction accelerated by acidic or basic conditions (number in parenthesis is percent recovery).
HS = Hydrolysis during storage (number in parenthesis is percent stability).
LR = Low response.
ND = Not determined.
OE = Oxidation during extraction accelerated by basic conditions (number in parenthesis is percent recovery).
OS = Oxidation during storage (number in parenthesis is percent stability).
X = Greater than 70 percent recovery by this technique.

1.2 In addition to the sample preparation methods listed in the above analyte list, Method 3542 describes sample preparation for semivolatile organic compounds in air sampled by Method 0010 (Table 11 contains surrogate performance data), Method 3545 describes an automated solvent extraction device for semivolatiles in solids (Table 12 contains performance data), and Method 3561 describes a supercritical fluid extraction of solids for PAHs (see Tables 13, 14, and 15 for performance data).

1.3 Method 8270 can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1 for a list of compounds and their characteristic ions that have been evaluated.

In most cases, Method 8270 is not appropriate for the quantitation of multicomponent analytes, e.g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analytes. When these analytes have been identified by another technique, Method 8270 is appropriate for confirmation of the presence of these analytes when concentration in the extract permits. Refer to Sec. 7.0 of Methods 8081 and 8082 for guidance on calibration and quantitation of multicomponent analytes such as the Aroclors, Toxaphene, and Chlordane.

1.4 The following compounds may require special treatment when being determined by this method:

1.4.1 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.

1.4.2 Under the alkaline conditions of the extraction step from aqueous matrices, α-BHC, γ-BHC, Endosulfan I and II, and Endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected.

1.4.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.4.4 N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.

1.4.5 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

1.4.6 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.4.7 Pyridine may perform poorly at the GC injection port temperatures listed in the method. Lowering the injection port temperature may reduce the amount of degradation. The analyst needs to use caution if modifying the injection port temperature as the performance of other analytes may be adversely affected.

1.4.8 Toluene diisocyanate rapidly hydrolyses in water (half-life of less then 30 min.). Therefore, recoveries of this compound from aqueous matrices should not be expected. In addition, in solid matrices, toluene diisocyanate often reacts with alcohols and amines to produce urethane and ureas and consequently cannot usually coexist in a solution containing these materials.

1.4.9 In addition, analytes in the list provided above are flagged when there are limitations caused by sample preparation and/or chromatographic problems.

1.5 The estimated quantitation limit (EQL) of Method 8270 for determining an individual compound is approximately 660 µg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for ground water samples (see Table 2). EQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation (refer to Method 3500) and, if necessary, sample cleanup procedures (refer to Method 3600).

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.4 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph/mass spectrometer system

4.1.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

4.1.2 Column - 30 m x 0.25 mm ID (or 0.32 mm ID) 1 µm film thickness silicone-coated fused-silica capillary column (J&W Scientific DB-5 or equivalent).

4.1.3 Mass spectrometer

4.1.3.1 Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 3 when 1 µL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

4.1.3.2 An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. The mass spectrometer must be capable of producing a mass spectrum for DFTPP which meets the criteria in Table 3 when 5 or 50 ng are introduced.

4.1.4 GC/MS interface - Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

4.1.5 Data system - A computer system should be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer should have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software should also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.1.6 Guard column (optional) - (J&W Deactivated Fused Silica, 0.25 mm ID x 6 m, or equivalent) between the injection port and the analytical column joined with column joiners (Hewlett-Packard Catalog No. 5062-3556, or equivalent).

4.2 Syringe - 10-µL.

4.3 Volumetric flasks, Class A - Appropriate sizes with ground-glass stoppers.

4.4 Balance - Analytical, capable of weighing 0.0001 g.

4.5 Bottles - glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Stock standard solutions (1000 mg/L) - Standard solutions can be prepared from pure standard materials or purchased as certified solutions.

5.3.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality acetone or other suitable solvent and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.3.2 Transfer the stock standard solutions into bottles with PTFE-lined screw-caps. Store, protected from light, at -10°C or less or as recommended by the standard manufacturer. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.3.3 Stock standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.

5.3.4 It is recommended that nitrosamine compounds be placed together in a separate calibration mix and not combined with other calibration mixes. When using a premixed certified standard, consult the manufacturer's instructions for additional guidance.

5.3.5 Mixes with hydrochloride salts may contain hydrochloric acid, which can cause analytical difficulties. When using a premixed certified standard, consult the manufacturer's instructions for additional guidance.

5.4 Internal standard solutions - The internal standards recommended are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (see Table 5). Other compounds may be used as internal standards as long as the requirements given in Sec. 7.3.2 are met.

5.4.1 Dissolve 0.200 g of each compound with a small volume of carbon disulfide. Transfer to a 50 mL volumetric flask and dilute to volume with methylene chloride so that the final solvent is approximately 20% carbon disulfide. Most of the compounds are also soluble in small volumes of methanol, acetone, or toluene, except for perylene-d_{12}. The resulting solution will contain each standard at a concentration of 4,000 ng/µL. Each 1 mL sample extract undergoing analysis should be spiked with 10 µL of the internal standard solution, resulting in a concentration of 40 ng/µL of each internal standard. Store at -10°C or less when not in use. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.4.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute internal standard solution may be required. Area counts of the internal standard peaks should be between 50-200% of the area of the target analytes in the mid-point calibration analysis.

5.5 GC/MS tuning standard - A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP) should be prepared. The standard should also contain 50 ng/µL each of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Store at -10°C or less when not in use. If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute tuning solution may be necessary. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.6 Calibration standards - A minimum of five calibration standards should be prepared at five different concentrations. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in actual samples but should not exceed the working range of the GC/MS system. Each standard should contain each analyte for detection by this method.

5.6.1 It is the intent of EPA that all target analytes for a particular analysis be included in the calibration standard(s). These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.6.2 Each 1-mL aliquot of calibration standard should be spiked with 10 µL of the internal standard solution prior to analysis. All standards should be stored at -10°C or less, and should be freshly prepared once a year, or sooner if check standards indicate a problem. The calibration verification standard should be prepared weekly and stored at 4°C. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.7 Surrogate standards - The recommended surrogates are phenol-d_6, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluorobiphenyl, and p-terphenyl-d_{14}. See Method 3500 for instructions on preparing the surrogate solutions.

5.7.1 Surrogate Standard Check: Determine what the appropriate concentration should be for the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery of surrogate standards. It is recommended that this check be done whenever a new surrogate spiking solution is prepared.

NOTE: Method 3561 (SFE Extraction of PAHs) recommends the use of bromobenzene and p-quaterphenyl to better cover the range of PAHs listed in the method.

5.7.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute surrogate solution may be necessary.

5.8 Matrix spike and laboratory control standards - See Method 3500 for instructions on preparing the matrix spike standard. The same standard may be used as the laboratory control standard (LCS).

5.8.1 Matrix Spike Check: Determine what concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery. It is recommended that this check be done whenever a new matrix spiking solution is prepared.

5.8.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, a more dilute matrix and LCS spiking solution may be necessary.

5.8.3 Some projects may require the spiking of the specific compounds of interest, since the spiking compounds listed in Method 3500 would not be representative of the compounds of interest required for the project. When this occurs, the matrix and LCS spiking

standards should be prepared in methanol, with each compound present at a concentration appropriate for the project.

5.9 Acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, and other appropriate solvents - All solvents should be pesticide quality or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2 Store the sample extracts at -10°C, protected from light, in sealed vials (e.g., screw-cap vials or crimp-capped vials) equipped with unpierced PTFE-lined septa.

7.0 PROCEDURE

7.1 Sample preparation

7.1.1 Samples are normally prepared by one of the following methods prior to GC/MS analysis.

Matrix	Methods
Air	3542
Water	3510, 3520, 3535
Soil/sediment	3540, 3541, 3545, 3550, 3560, 3561
Waste	3540, 3541, 3545, 3550, 3560, 3561, 3580

7.1.2 In very limited applications, direct injection of the sample into the GC/MS system with a 10-µL syringe may be appropriate. The detection limit is very high (approximately 10,000 µg/L). Therefore, it is only permitted where concentrations in excess of 10,000 µg/L are expected.

7.2 Extract cleanup - Extracts may be cleaned up by any of the following methods prior to GC/MS analysis.

Analytes of interest	Methods
Aniline & aniline derivatives	3620
Phenols	3630, 3640, 8041[a]
Phthalate esters	3610, 3620, 3640
Nitrosamines	3610, 3620, 3640
Organochlorine pesticides & PCBs	3610, 3620, 3630, 3660, 3665
Nitroaromatics and cyclic ketones	3620, 3640
Polynuclear aromatic hydrocarbons	3611, 3630, 3640
Haloethers	3620, 3640
Chlorinated hydrocarbons	3620, 3640
Organophosphorus pesticides	3620

Analytes of interest	Methods
Petroleum waste	3611, 3650
All base, neutral, and acid priority pollutants	3640

[a] Method 8041 includes a derivatization technique followed by GC/ECD analysis, if interferences are encountered on GC/FID.

7.3 Initial calibration

Establish the GC/MS operating conditions, using the following recommendations as guidance.

Mass range:	35-500 amu
Scan time:	1 sec/scan
Initial temperature:	40°C, hold for 4 minutes
Temperature program:	40-270°C at 10°C/min
Final temperature:	270°C, hold until benzo[g,h,i]perylene elutes
Injector temperature:	250-300°C
Transfer line temperature:	250-300°C
Source temperature:	According to manufacturer's specifications
Injector:	Grob-type, splitless
Injection volume:	1-2 µL
Carrier gas:	Hydrogen at 50 cm/sec or helium at 30 cm/sec
Ion trap only:	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

Split injection is allowed if the sensitivity of the mass spectrometer is sufficient.

7.3.1 The GC/MS system must be hardware-tuned using a 50 ng injection of DFTPP. Analyses must not begin until the tuning criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of DFTPP from the instrument manufacturer, the following approach has been shown to be useful: Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak.

7.3.1.2 Use the DFTPP mass intensity criteria in Table 3 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g. CLP, Method 525, or manufacturer's instructions), provided that method performance is not adversely affected.

NOTE: All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

7.3.1.3 The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD

should not exceed 20%. (See Sec. 8.0 of Method 8081 for the percent breakdown calculation). Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible.

7.3.1.4 If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6-12 in. of the capillary column. The use of a guard column (Sec. 4.1.6) between the injection port and the analytical column may help prolong analytical column performance.

7.3.2 The internal standards selected in Sec. 5.4 should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion (i.e. for 1,4-dichlorobenzene-d_4, use 152 m/z for quantitation).

7.3.3 Analyze 1-2 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each target analyte (as indicated in Table 1). A set of at least five calibration standards is necessary (see Sec. 5.6 and Method 8000). The injection volume must be the same for all standards and sample extracts. Figure 1 shows a chromatogram of a calibration standard containing base/neutral and acid analytes.

Calculate response factors (RFs) for each target analyte relative to one of the internal standards as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

7.3.4 System performance check compounds (SPCCs)

7.3.4.1 A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compounds (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol.

7.3.4.2 The minimum acceptable average RF for these compounds is 0.050. These SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

7.3.4.3 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

7.3.5 Calibration check compounds (CCCs)

7.3.5.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Section 7.0 of Method 8000.

7.3.5.2 Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte. The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual CCC (see Table 4) must be less than or equal to 30%.

$$\text{mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}}$$

$$RSD = \frac{SD}{\overline{RF}} \times 100$$

7.3.5.3 If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure beginning with Sec. 7.3.

7.3.5.4 If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000.

7.3.6 Evaluation of retention times - The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement.

7.3.7 Linearity of target analytes - If the RSD of any target analytes is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation (Sec. 7.6.2).

7.3.7.1 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 in Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

NOTE: Method 8000 designates a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8270 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.7.2 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject 50 ng of the DFTPP standard into the GC/MS system. The resultant mass spectrum for DFTPP must meet the criteria given in Table 3 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique and conditions used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The DFTPP and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000B for method blank performance criteria.

7.4.4 System performance check compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC in the calibration verification standard must meet a minimum response factor of 0.050. This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

7.4.5 Calibration check compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Table 4 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference for each CCC is less than or equal to 20%, then the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift) for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCCs are not included in the list of analytes for a project,

and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.5 GC/MS analysis of samples

7.5.1 It is highly recommended that sample extracts be screened on a GC/FID or GC/PID using the same type of capillary column used in the GC/MS system. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

7.5.2 Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 µL of the internal standard solution to the 1-mL concentrated sample extract obtained from sample preparation.

7.5.3 Inject a 1-2 µL aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration (Sec. 7.3). The volume to be injected should contain 100 ng of base/neutral and 200 ng of acid surrogates (assuming 100% recovery), unless a more sensitive GC/MS system is being used and the surrogate solution is less concentrated then that listed in Sec. 5.7. The injection volume must be the same volume used for the calibration standards.

7.5.4 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (40 ng/µL, unless a more sensitive GC/MS system is being used).

NOTE: It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

7.5.5 The use of selected ion monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry. However, SIM

may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

7.6 Qualitative analysis

7.6.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

7.6.1.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.6.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

7.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. Diastereomeric pairs (e.g., Aramite and Isosafrol) that may be separable by the GC should be identified, quantitated and reported as the sum of both compounds by the GC.

7.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the

analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

7.7.2 If the RSD of a compound's response factor is 15% or less, then the concentration in the extract may be determined using the average response factor ($\overline{RF}$) from initial calibration data (Sec. 7.3.5). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

7.7.5 Quantitation of multicomponent compounds (e.g., Toxaphene, Aroclors, etc.) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD, by Methods 8081 or 8082. However, Method 8270 may be used to confirm the identification of these compounds, when the concentrations are at least 10 ng/µL in the concentrated sample extract.

7.7.6 Structural isomers that produce very similar mass spectra should be quantitated as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are quantitated as isomeric pairs. Diastereomeric pairs (e.g., Aramite and Isosafrol) that may be separable by the GC should be summed and reported as the sum of both compounds.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Method 3500. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Sec. 7.0 of Method 8000 and include calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8270:

8.2.1 The GC/MS system must be tuned to meet the DFTPP criteria listed in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the calibration verification acceptance criteria in Sec. 7.4, each 12 hours.

8.2.4 The RRT of the sample component must fall within the RRT window of the standard component provided in Sec. 7.6.1.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed, a septum is changed), see the guidance in Sec 8.2 of Method 8000 regarding whether recalibration of the system must take place.

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

9.1 Method 8250 (the packed column version of Method 8270) was tested by 15 laboratories using organic-free reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations ranging from 5 to 1,300 µg/L. Single operator accuracy and precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 7. These values are presented as guidance only and are not intended as absolute acceptance criteria. Laboratories should generate their own acceptance criteria for capillary column method performance. (See Method 8000.)

9.2 Chromatograms from calibration standards analyzed with Day 0 and Day 7 samples were compared to detect possible deterioration of GC performance. These recoveries (using Method 3510 extraction) are presented in Table 8.

9.3 Method performance data (using Method 3541 Automated Soxhlet extraction) are presented in Table 9. Single laboratory accuracy and precision data were obtained for semivolatile organics in a clay soil by spiking at a concentration of 6 mg/kg for each compound. The spiking solution was mixed into the soil during addition and then allowed to equilibrate for approximately 1 hour prior to extraction. The spiked samples were then extracted by Method 3541 (Automated Soxhlet). Three determinations were performed and each extract was analyzed by gas chromatography/ mass spectrometry following Method 8270. The low recovery of the more volatile compounds is probably due to volatilization losses during equilibration. These data are listed in Table 10 and were taken from Reference 7.

9.4 Surrogate precision and accuracy data are presented in Table 11 from a field dynamic spiking study based on air sampling by Method 0010. The trapping media were prepared for analysis by Method 3542 and subsequently analyzed by Method 8270.

9.5 Single laboratory precision and bias data (using Method 3545 Accelerated Solvent Extraction) for semivolatile organic compounds are presented in Table 12. The samples were conditioned spiked samples prepared and certified by a commercial supplier that contained 57 semivolatile organics at three concentrations (250, 2500, and 12,500 µg/kg) on three types of soil (clay, loam and sand). Spiked samples were extracted both by the Dionex Accelerated Solvent Extraction system and by Perstorp Environmental Soxtec™ (automated Soxhlet). The data presented in Table 12 represents seven replicate extractions and analyses for each individual sample and were taken from reference 9. The average recoveries from the three matrices for all analytes and all replicates relative to the automated Soxhlet data are as follows: clay 96.8%, loam 98.7% and sand 102.1%. The average recoveries from the three concentrations also relative to the automated Soxhlet data are as follows: low-101.2%, mid-97.2% and high-99.2%.

9.6 Single laboratory precision and bias data (using Method 3561 SFE Extraction of PAHs with a variable restrictor and solid trapping material) were obtained for the method analytes by the extraction of two certified reference materials (one, EC-1, a lake sediment from Environment Canada and the other, HS-3, a marine sediment from the National Science and Engineering Research Council of Canada, both naturally-contaminated with PAHs). The SFE instrument used for these extractions was a Hewlett-Packard Model 7680. Analysis was by GC/MS. Average recoveries from six replicate extractions range from 85 to 148% (overall average of 100%) based on the certified value (or a Soxhlet value if a certified value was unavailable for a specific analyte) for the lake sediment. Average recoveries from three replicate extractions range from 73 to 133% (overall average of 92%) based on the certified value for the marine sediment. The data are found in Tables 13 and 14 and were taken from Reference 10.

9.7 Single laboratory precision and accuracy data (using Method 3561 SFE Extraction of PAHs with a fixed restrictor and liquid trapping) were obtained for twelve of the method analytes by the extraction of a certified reference material (a soil naturally contaminated with PAHs). The SFE instrument used for these extractions was a Dionex Model 703-M. Analysis was by GC/MS. Average recoveries from four replicate extractions range from 60 to 122% (overall average of 89%) based on the certified value. Following are the instrument conditions that were utilized to extract a 3.4 g sample: Pressure - 300 atm; Time - 60 min.; Extraction fluid - CO_2; Modifier - 10% 1:1 (v/v) methanol/methylene chloride; Oven temperature - 80°C; Restrictor temperature - 120°C; and, Trapping fluid - chloroform (methylene chloride has also been used). The data are found in Table 15 and were taken from Reference 11.

10.0 REFERENCES

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TABLE 1

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
2-Picoline	3.75[a]	93	66,92
Aniline	5.68	93	66,65
Phenol	5.77	94	65,66
Bis(2-chloroethyl) ether	5.82	93	63,95
2-Chlorophenol	5.97	128	64,130
1,3-Dichlorobenzene	6.27	146	148,111
1,4-Dichlorobenzene-d_4 (IS)	6.35	152	150,115
1,4-Dichlorobenzene	6.40	146	148,111
Benzyl alcohol	6.78	108	79,77
1,2-Dichlorobenzene	6.85	146	148,111
N-Nitrosomethylethylamine	6.97	88	42,43,56
Bis(2-chloroisopropyl) ether	7.22	45	77,121
Ethyl carbamate	7.27	62	44,45,74
Thiophenol (Benzenethiol)	7.42	110	66,109,84
Methyl methanesulfonate	7.48	80	79,65,95
N-Nitrosodi-n-propylamine	7.55	70	42,101,130
Hexachloroethane	7.65	117	201,199
Maleic anhydride	7.65	54	98,53,44
Nitrobenzene	7.87	77	123,65
Isophorone	8.53	82	95,138
N-Nitrosodiethylamine	8.70	102	42,57,44,56
2-Nitrophenol	8.75	139	109,65
2,4-Dimethylphenol	9.03	122	107,121
p-Benzoquinone	9.13	108	54,82,80
Bis(2-chloroethoxy)methane	9.23	93	95,123
Benzoic acid	9.38	122	105,77
2,4-Dichlorophenol	9.48	162	164,98
Trimethyl phosphate	9.53	110	79,95,109,140
Ethyl methanesulfonate	9.62	79	109,97,45,65
1,2,4-Trichlorobenzene	9.67	180	182,145
Naphthalene-d_8 (IS)	9.75	136	68
Naphthalene	9.82	128	129,127
Hexachlorobutadiene	10.43	225	223,227
Tetraethyl pyrophosphate	11.07	99	155,127,81,109
Diethyl sulfate	11.37	139	45,59,99,111,125
4-Chloro-3-methylphenol	11.68	107	144,142
2-Methylnaphthalene	11.87	142	141
2-Methylphenol	12.40	107	108,77,79,90
Hexachloropropene	12.45	213	211,215,117,106,141
Hexachlorocyclopentadiene	12.60	237	235,272

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
N-Nitrosopyrrolidine	12.65	100	41,42,68,69
Acetophenone	12.67	105	71,51,120
4-Methylphenol	12.82	107	108,77,79,90
2,4,6-Trichlorophenol	12.85	196	198,200
o-Toluidine	12.87	106	107,77,51,79
3-Methylphenol	12.93	107	108,77,79,90
2-Chloronaphthalene	13.30	162	127,164
N-Nitrosopiperidine	13.55	114	42,55,56,41
1,4-Phenylenediamine	13.62	108	80,53,54,52
1-Chloronaphthalene	13.65[a]	162	127,164
2-Nitroaniline	13.75	65	92,138
5-Chloro-2-methylaniline	14.28	106	141,140,77,89
Dimethyl phthalate	14.48	163	194,164
Acenaphthylene	14.57	152	151,153
2,6-Dinitrotoluene	14.62	165	63,89
Phthalic anhydride	14.62	104	76,50,148
o-Anisidine	15.00	108	80,123,52
3-Nitroaniline	15.02	138	108,92
Acenaphthene-d_{10} (IS)	15.05	164	162,160
Acenaphthene	15.13	154	153,152
2,4-Dinitrophenol	15.35	184	63,154
2,6-Dinitrophenol	15.47	162	164,126,98,63
4-Chloroaniline	15.50	127	129,65,92
Isosafrole	15.60	162	131,104,77,51
Dibenzofuran	15.63	168	139
2,4-Diaminotoluene	15.78	121	122,94,77,104
2,4-Dinitrotoluene	15.80	165	63,89
4-Nitrophenol	15.80	139	109,65
2-Naphthylamine	16.00[a]	143	115,116
1,4-Naphthoquinone	16.23	158	104,102,76,50,130
p-Cresidine	16.45	122	94,137,77,93
Dichlorovos	16.48	109	185,79,145
Diethyl phthalate	16.70	149	177,150
Fluorene	16.70	166	165,167
2,4,5-Trimethylaniline	16.70	120	135,134,91,77
N-Nitrosodi-n-butylamine	16.73	84	57,41,116,158
4-Chlorophenyl phenyl ether	16.78	204	206,141
Hydroquinone	16.93	110	81,53,55
4,6-Dinitro-2-methylphenol	17.05	198	51,105
Resorcinol	17.13	110	81,82,53,69
N-Nitrosodiphenylamine	17.17	169	168,167
Safrole	17.23	162	104,77,103,135

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Hexamethyl phosphoramide	17.33	135	44,179,92,42
3-(Chloromethyl)pyridine hydrochloride	17.50	92	127,129,65,39
Diphenylamine	17.54[a]	169	168,167
1,2,4,5-Tetrachlorobenzene	17.97	216	214,179,108,143,218
1-Naphthylamine	18.20	143	115,89,63
1-Acetyl-2-thiourea	18.22	118	43,42,76
4-Bromophenyl phenyl ether	18.27	248	250,141
Toluene diisocyanate	18.42	174	145,173,146,132,91
2,4,5-Trichlorophenol	18.47	196	198,97,132,99
Hexachlorobenzene	18.65	284	142,249
Nicotine	18.70	84	133,161,162
Pentachlorophenol	19.25	266	264,268
5-Nitro-o-toluidine	19.27	152	77,79,106,94
Thionazine	19.35	107	96,97,143,79,68
4-Nitroaniline	19.37	138	65,108,92,80,39
Phenanthrene-d_{10} (IS)	19.55	188	94,80
Phenanthrene	19.62	178	179,176
Anthracene	19.77	178	176,179
1,4-Dinitrobenzene	19.83	168	75,50,76,92,122
Mevinphos	19.90	127	192,109,67,164
Naled	20.03	109	145,147,301,79,189
1,3-Dinitrobenzene	20.18	168	76,50,75,92,122
Diallate (cis or trans)	20.57	86	234,43,70
1,2-Dinitrobenzene	20.58	168	50,63,74
Diallate (trans or cis)	20.78	86	234,43,70
Pentachlorobenzene	21.35	250	252,108,248,215,254
5-Nitro-o-anisidine	21.50	168	79,52,138,153,77
Pentachloronitrobenzene	21.72	237	142,214,249,295,265
4-Nitroquinoline-1-oxide	21.73	174	101,128,75,116
Di-n-butyl phthalate	21.78	149	150,104
2,3,4,6-Tetrachlorophenol	21.88	232	131,230,166,234,168
Dihydrosaffrole	22.42	135	64,77
Demeton-O	22.72	88	89,60,61,115,171
Fluoranthene	23.33	202	101,203
1,3,5-Trinitrobenzene	23.68	75	74,213,120,91,63
Dicrotophos	23.82	127	67,72,109,193,237
Benzidine	23.87	184	92,185
Trifluralin	23.88	306	43,264,41,290
Bromoxynil	23.90	277	279,88,275,168
Pyrene	24.02	202	200,203
Monocrotophos	24.08	127	192,67,97,109
Phorate	24.10	75	121,97,93,260

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Sulfallate	24.23	188	88,72,60,44
Demeton-S	24.30	88	60,81,89,114,115
Phenacetin	24.33	108	180,179,109,137,80
Dimethoate	24.70	87	93,125,143,229
Phenobarbital	24.70	204	117,232,146,161
Carbofuran	24.90	164	149,131,122
Octamethyl pyrophosphoramide	24.95	135	44,199,286,153,243
4-Aminobiphenyl	25.08	169	168,170,115
Dioxathion	25.25	97	125,270,153
Terbufos	25.35	231	57,97,153,103
α,α-Dimethylphenylamine	25.43	58	91,65,134,42
Pronamide	25.48	173	175,145,109,147
Aminoazobenzene	25.72	197	92,120,65,77
Dichlone	25.77	191	163,226,228,135,193
Dinoseb	25.83	211	163,147,117,240
Disulfoton	25.83	88	97,89,142,186
Fluchloralin	25.88	306	63,326,328,264,65
Mexacarbate	26.02	165	150,134,164,222
4,4'-Oxydianiline	26.08	200	108,171,80,65
Butyl benzyl phthalate	26.43	149	91,206
4-Nitrobiphenyl	26.55	199	152,141,169,151
Phosphamidon	26.85	127	264,72,109,138
2-Cyclohexyl-4,6-Dinitrophenol	26.87	231	185,41,193,266
Methyl parathion	27.03	109	125,263,79,93
Carbaryl	27.17	144	115,116,201
Dimethylaminoazobenzene	27.50	225	120,77,105,148,42
Propylthiouracil	27.68	170	142,114,83
Benz(a)anthracene	27.83	228	229,226
Chrysene-d_{12} (IS)	27.88	240	120,236
3,3'-Dichlorobenzidine	27.88	252	254,126
Chrysene	27.97	228	226,229
Malathion	28.08	173	125,127,93,158
Kepone	28.18	272	274,237,178,143,270
Fenthion	28.37	278	125,109,169,153
Parathion	28.40	109	97,291,139,155
Anilazine	28.47	239	241,143,178,89
Bis(2-ethylhexyl) phthalate	28.47	149	167,279
3,3'-Dimethylbenzidine	28.55	212	106,196,180
Carbophenothion	28.58	157	97,121,342,159,199
5-Nitroacenaphthene	28.73	199	152,169,141,115
Methapyrilene	28.77	97	50,191,71
Isodrin	28.95	193	66,195,263,265,147

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Captan	29.47	79	149,77,119,117
Chlorfenvinphos	29.53	267	269,323,325,295
Crotoxyphos	29.73	127	105,193,166
Phosmet	30.03	160	77,93,317,76
EPN	30.11	157	169,185,141,323
Tetrachlorvinphos	30.27	329	109,331,79,333
Di-n-octyl phthalate	30.48	149	167,43
2-Aminoanthraquinone	30.63	223	167,195
Barban	30.83	222	51,87,224,257,153
Aramite	30.92	185	191,319,334,197,321
Benzo(b)fluoranthene	31.45	252	253,125
Nitrofen	31.48	283	285,202,139,253
Benzo(k)fluoranthene	31.55	252	253,125
Chlorobenzilate	31.77	251	139,253,111,141
Fensulfothion	31.87	293	97,308,125,292
Ethion	32.08	231	97,153,125,121
Diethylstilbestrol	32.15	268	145,107,239,121,159
Famphur	32.67	218	125,93,109,217
Tri-p-tolyl phosphate[b]	32.75	368	367,107,165,198
Benzo(a)pyrene	32.80	252	253,125
Perylene-d_{12} (IS)	33.05	264	260,265
7,12-Dimethylbenz(a)anthracene	33.25	256	241,239,120
5,5-Diphenylhydantoin	33.40	180	104,252,223,209
Captafol	33.47	79	77,80,107
Dinocap	33.47	69	41,39
Methoxychlor	33.55	227	228,152,114,274,212
2-Acetylaminofluorene	33.58	181	180,223,152
4,4'-Methylenebis(2-chloroaniline)	34.38	231	266,268,140,195
3,3'-Dimethoxybenzidine	34.47	244	201,229
3-Methylcholanthrene	35.07	268	252,253,126,134,113
Phosalone	35.23	182	184,367,121,379
Azinphos-methyl	35.25	160	132,93,104,105
Leptophos	35.28	171	377,375,77,155,379
Mirex	35.43	272	237,274,270,239,235
Tris(2,3-dibromopropyl) phosphate	35.68	201	137,119,217,219,199
Dibenz(a,j)acridine	36.40	279	280,277,250
Mestranol	36.48	277	310,174,147,242
Coumaphos	37.08	362	226,210,364,97,109
Indeno(1,2,3-cd)pyrene	39.52	276	138,227
Dibenz(a,h)anthracene	39.82	278	139,279
Benzo(g,h,i)perylene	41.43	276	138,277
1,2:4,5-Dibenzopyrene	41.60	302	151,150,300

TABLE 1
(continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Strychnine	45.15	334	334,335,333
Piperonyl sulfoxide	46.43	162	135,105,77
Hexachlorophene	47.98	196	198,209,211,406,408
Aldrin	--	66	263,220
Aroclor 1016	--	222	260,292
Aroclor 1221	--	190	224,260
Aroclor 1232	--	190	224,260
Aroclor 1242	--	222	256,292
Aroclor 1248	--	292	362,326
Aroclor 1254	--	292	362,326
Aroclor 1260	--	360	362,394
α-BHC	--	183	181,109
β-BHC	--	181	183,109
δ-BHC	--	183	181,109
γ-BHC (Lindane)	--	183	181,109
4,4'-DDD	--	235	237,165
4,4'-DDE	--	246	248,176
4,4'-DDT	--	235	237,165
Dieldrin	--	79	263,279
1,2-Diphenylhydrazine	--	77	105,182
Endosulfan I	--	195	339,341
Endosulfan II	--	337	339,341
Endosulfan sulfate	--	272	387,422
Endrin	--	263	82,81
Endrin aldehyde	--	67	345,250
Endrin ketone	--	317	67,319
2-Fluorobiphenyl (surr)	--	172	171
2-Fluorophenol (surr)	--	112	64
Heptachlor	--	100	272,274
Heptachlor epoxide	--	353	355,351
Nitrobenzene-d_5 (surr)	--	82	128,54
N-Nitrosodimethylamine	--	42	74,44
Phenol-d_6 (surr)	--	99	42,71
Terphenyl-d_{14} (surr)	--	244	122,212
2,4,6-Tribromophenol (surr)	--	330	332,141
Toxaphene	--	159	231,233

IS = internal standard
surr = surrogate
[a]Estimated retention times
[b]Substitute for the non-specific mixture, tricresyl phosphate

TABLE 2

ESTIMATED QUANTITATION LIMITS (EQLs) FOR SEMIVOLATILE ORGANICS

Compound	Estimated Quantitation Limits[a] Ground water µg/L	Low Soil/Sediment[b] µg/kg
Acenaphthene	10	660
Acenaphthylene	10	660
Acetophenone	10	ND
2-Acetylaminofluorene	20	ND
1-Acetyl-2-thiourea	1000	ND
2-Aminoanthraquinone	20	ND
Aminoazobenzene	10	ND
4-Aminobiphenyl	20	ND
Anilazine	100	ND
o-Anisidine	10	ND
Anthracene	10	660
Aramite	20	ND
Azinphos-methyl	100	ND
Barban	200	ND
Benz(a)anthracene	10	660
Benzo(b)fluoranthene	10	660
Benzo(k)fluoranthene	10	660
Benzoic acid	50	3300
Benzo(g,h,i)perylene	10	660
Benzo(a)pyrene	10	660
p-Benzoquinone	10	ND
Benzyl alcohol	20	1300
Bis(2-chloroethoxy)methane	10	660
Bis(2-chloroethyl) ether	10	660
Bis(2-chloroisopropyl) ether	10	660
4-Bromophenyl phenyl ether	10	660
Bromoxynil	10	ND
Butyl benzyl phthalate	10	660
Captafol	20	ND
Captan	50	ND
Carbaryl	10	ND
Carbofuran	10	ND
Carbophenothion	10	ND
Chlorfenvinphos	20	ND
4-Chloroaniline	20	1300
Chlorobenzilate	10	ND
5-Chloro-2-methylaniline	10	ND
4-Chloro-3-methylphenol	20	1300
3-(Chloromethyl)pyridine hydrochloride	100	ND
2-Chloronaphthalene	10	660

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	Low Soil/Sediment[b] µg/kg
2-Chlorophenol	10	660
4-Chlorophenyl phenyl ether	10	660
Chrysene	10	660
Coumaphos	40	ND
p-Cresidine	10	ND
Crotoxyphos	20	ND
2-Cyclohexyl-4,6-dinitrophenol	100	ND
Demeton-O	10	ND
Demeton-S	10	ND
Diallate (cis or trans)	10	ND
Diallate (trans or cis)	10	ND
2,4-Diaminotoluene	20	ND
Dibenz(a,j)acridine	10	ND
Dibenz(a,h)anthracene	10	660
Dibenzofuran	10	660
Dibenzo(a,e)pyrene	10	ND
Di-n-butyl phthalate	10	ND
Dichlone	NA	ND
1,2-Dichlorobenzene	10	660
1,3-Dichlorobenzene	10	660
1,4-Dichlorobenzene	10	660
3,3'-Dichlorobenzidine	20	1300
2,4-Dichlorophenol	10	660
2,6-Dichlorophenol	10	ND
Dichlorovos	10	ND
Dicrotophos	10	ND
Diethyl phthalate	10	660
Diethylstilbestrol	20	ND
Diethyl sulfate	100	ND
Dimethoate	20	ND
3,3'-Dimethoxybenzidine	100	ND
Dimethylaminoazobenzene	10	ND
7,12-Dimethylbenz(a)anthracene	10	ND
3,3'-Dimethylbenzidine	10	ND
a,a-Dimethylphenethylamine	ND	ND
2,4-Dimethylphenol	10	660
Dimethyl phthalate	10	660
1,2-Dinitrobenzene	40	ND
1,3-Dinitrobenzene	20	ND
1,4-Dinitrobenzene	40	ND
4,6-Dinitro-2-methylphenol	50	3300
2,4-Dinitrophenol	50	3300

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	Low Soil/Sediment[b] µg/kg
2,4-Dinitrotoluene	10	660
2,6-Dinitrotoluene	10	660
Dinocap	100	ND
Dinoseb	20	ND
5,5-Diphenylhydantoin	20	ND
Di-n-octyl phthalate	10	660
Disulfoton	10	ND
EPN	10	ND
Ethion	10	ND
Ethyl carbamate	50	ND
Bis(2-ethylhexyl) phthalate	10	660
Ethyl methanesulfonate	20	ND
Famphur	20	ND
Fensulfothion	40	ND
Fenthion	10	ND
Fluchloralin	20	ND
Fluoranthene	10	660
Fluorene	10	660
Hexachlorobenzene	10	660
Hexachlorobutadiene	10	660
Hexachlorocyclopentadiene	10	660
Hexachloroethane	10	660
Hexachlorophene	50	ND
Hexachloropropene	10	ND
Hexamethylphosphoramide	20	ND
Hydroquinone	ND	ND
Indeno(1,2,3-cd)pyrene	10	660
Isodrin	20	ND
Isophorone	10	660
Isosafrole	10	ND
Kepone	20	ND
Leptophos	10	ND
Malathion	50	ND
Maleic anhydride	NA	ND
Mestranol	20	ND
Methapyrilene	100	ND
Methoxychlor	10	ND
3-Methylcholanthrene	10	ND
4,4'-Methylenebis(2-chloroaniline)	NA	ND
Methyl methanesulfonate	10	ND
2-Methylnaphthalene	10	660
Methyl parathion	10	ND
2-Methylphenol	10	660
3-Methylphenol	10	ND

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	Low Soil/Sediment[b] µg/kg
4-Methylphenol	10	660
Mevinphos	10	ND
Mexacarbate	20	ND
Mirex	10	ND
Monocrotophos	40	ND
Naled	20	ND
Naphthalene	10	660
1,4-Naphthoquinone	10	ND
1-Naphthylamine	10	ND
2-Naphthylamine	10	ND
Nicotine	20	ND
5-Nitroacenaphthene	10	ND
2-Nitroaniline	50	3300
3-Nitroaniline	50	3300
4-Nitroaniline	20	ND
5-Nitro-o-anisidine	10	ND
Nitrobenzene	10	660
4-Nitrobiphenyl	10	ND
Nitrofen	20	ND
2-Nitrophenol	10	660
4-Nitrophenol	50	3300
5-Nitro-o-toluidine	10	ND
4-Nitroquinoline-1-oxide	40	ND
N-Nitrosodi-n-butylamine	10	ND
N-Nitrosodiethylamine	20	ND
N-Nitrosodiphenylamine	10	660
N-Nitroso-di-n-propylamine	10	660
N-Nitrosopiperidine	20	ND
N-Nitrosopyrrolidine	40	ND
Octamethyl pyrophosphoramide	200	ND
4,4'-Oxydianiline	20	ND
Parathion	10	ND
Pentachlorobenzene	10	ND
Pentachloronitrobenzene	20	ND
Pentachlorophenol	50	3300
Phenacetin	20	ND
Phenanthrene	10	660
Phenobarbital	10	ND
Phenol	10	660
1,4-Phenylenediamine	10	ND
Phorate	10	ND
Phosalone	100	ND
Phosmet	40	ND
Phosphamidon	100	ND

TABLE 2 (cont.)

Compound	Estimated Quantitation Limits[a] Ground water µg/L	Low Soil/Sediment[b] µg/kg
Phthalic anhydride	100	ND
2-Picoline	ND	ND
Piperonyl sulfoxide	100	ND
Pronamide	10	ND
Propylthiouracil	100	ND
Pyrene	10	660
Pyridine	ND	ND
Resorcinol	100	ND
Safrole	10	ND
Strychnine	40	ND
Sulfallate	10	ND
Terbufos	20	ND
1,2,4,5-Tetrachlorobenzene	10	ND
2,3,4,6-Tetrachlorophenol	10	ND
Tetrachlorvinphos	20	ND
Tetraethyl pyrophosphate	40	ND
Thionazine	20	ND
Thiophenol (Benzenethiol)	20	ND
o-Toluidine	10	ND
1,2,4-Trichlorobenzene	10	660
2,4,5-Trichlorophenol	10	660
2,4,6-Trichlorophenol	10	660
Trifluralin	10	ND
2,4,5-Trimethylaniline	10	ND
Trimethyl phosphate	10	ND
1,3,5-Trinitrobenzene	10	ND
Tris(2,3-dibromopropyl) phosphate	200	ND
Tri-p-tolyl phosphate(h)	10	ND
O,O,O-Triethyl phosphorothioate	NT	ND

[a] Sample EQLs are highly matrix-dependent. The EQLs listed here are provided for guidance and may not always be achievable.

[b] EQLs listed for soil/sediment are based on wet weight. Normally, data are reported on a dry weight basis, therefore, EQLs will be higher based on the % dry weight of each sample. These EQLs are based on a 30-g sample and gel permeation chromatography cleanup.

ND = Not Determined
NA = Not Applicable
NT = Not Tested

Other Matrices	Factor[c]
High-concentration soil and sludges by ultrasonic extractor	7.5
Non-water miscible waste	75

[c]EQL = (EQL for Low Soil/Sediment given above in Table 2) x (Factor)

TABLE 3

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA[a,b]

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

[a] Data taken from Reference 3.

[b] Alternate tuning criteria may be used, (e.g., CLP, Method 525, or manufacturers' instructions), provided that method performance is not adversely affected.

TABLE 4

CALIBRATION CHECK COMPOUNDS (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

TABLE 5

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethyl-phenethylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	Hexachlorocyclo-pentadiene
N-Nitrosodimethylamine	Nitrobenzene-d_8 (surr)	1-Naphthylamine
N-Nitroso-di-n-propyl-amine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodi-n-butylamine	2-Nitroaniline
Phenol-d_6 (surr)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetra-chlorobenzene
		2,3,4,6-Tetra-chlorophenol
		2,4,6-Tribromo-phenol (surr)
		2,4,6-Trichloro-phenol
		2,4,5-Trichloro-phenol

(surr) = surrogate

TABLE 5
(continued)

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Aminobiphenyl	Benzidine	Benzo(b)fluor-anthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluor-anthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)-perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methyl-phenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'-Dichlorobenzidine	Dibenz(a,h)-anthracene
Fluoranthene	p-Dimethylaminoazobenzene	
Hexachlorobenzene	Pyrene	
N-Nitrosodiphenylamine	Terphenyl-d_{14} (surr)	
Pentachlorophenol	7,12-Dimethylbenz-(a)anthracene	
Pentachloronitrobenzene	Di-n-octyl phthalate	
Phenacetin	Indeno(1,2,3-cd) pyrene	
Phenanthrene	3-Methylchol-anthrene	
Pronamide		

(surr) = surrogate

TABLE 6

MULTILABORATORY PERFORMANCE DATA[a]

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{x}$ (µg/L)	Range p, p_s (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27-133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(g,h,i)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
β-BHC	100	31.5	41.5-130.6	24-149
δ-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192

TABLE 6
(continued)

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{x}$ (µg/L)	Range p, p_s (%)
Heptachlor epoxide	100	54.7	70.9-109.4	26.155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
Aroclor 1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation of four recovery measurements, in µg/L

$\bar{x}$ = Average recovery for four recovery measurements, in µg/L

p, p_s = Measured percent recovery

D = Detected; result must be greater than zero

[a] Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 7

METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION[a]

Compound	Accuracy, as recovery, x' (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S' (µg/L)
Acenaphthene	0.96C+0.19	0.15$\bar{x}$-0.12	0.21$\bar{x}$-0.67
Acenaphthylene	0.89C+0.74	0.24$\bar{x}$-1.06	0.26$\bar{x}$-0.54
Aldrin	0.78C+1.66	0.27$\bar{x}$-1.28	0.43$\bar{x}$+1.13
Anthracene	0.80C+0.68	0.21$\bar{x}$-0.32	0.27$\bar{x}$-0.64
Benz(a)anthracene	0.88C-0.60	0.15$\bar{x}$+0.93	0.26$\bar{x}$-0.21
Benzo(b)fluoranthene	0.93C-1.80	0.22$\bar{x}$+0.43	0.29$\bar{x}$+0.96
Benzo(k)fluoranthene	0.87C-1.56	0.19$\bar{x}$+1.03	0.35$\bar{x}$+0.40
Benzo(a)pyrene	0.90C-0.13	0.22$\bar{x}$+0.48	0.32$\bar{x}$+1.35
Benzo(g,h,i)perylene	0.98C-0.86	0.29$\bar{x}$+2.40	0.51$\bar{x}$-0.44
Benzyl butyl phthalate	0.66C-1.68	0.18$\bar{x}$+0.94	0.53$\bar{x}$+0.92
β-BHC	0.87C-0.94	0.20$\bar{x}$-0.58	0.30$\bar{x}$+1.94
δ-BHC	0.29C-1.09	0.34$\bar{x}$+0.86	0.93$\bar{x}$-0.17
Bis(2-chloroethyl) ether	0.86C-1.54	0.35$\bar{x}$-0.99	0.35$\bar{x}$+0.10
Bis(2-chloroethoxy)methane	1.12C-5.04	0.16$\bar{x}$+1.34	0.26$\bar{x}$+2.01
Bis(2-chloroisopropyl) ether	1.03C-2.31	0.24$\bar{x}$+0.28	0.25$\bar{x}$+1.04
Bis(2-ethylhexyl) phthalate	0.84C-1.18	0.26$\bar{x}$+0.73	0.36$\bar{x}$+0.67
4-Bromophenyl phenyl ether	0.91C-1.34	0.13$\bar{x}$+0.66	0.16$\bar{x}$+0.66
2-Chloronaphthalene	0.89C+0.01	0.07$\bar{x}$+0.52	0.13$\bar{x}$+0.34
4-Chlorophenyl phenyl ether	0.91C+0.53	0.20$\bar{x}$-0.94	0.30$\bar{x}$-0.46
Chrysene	0.93C-1.00	0.28$\bar{x}$+0.13	0.33$\bar{x}$-0.09
4,4'-DDD	0.56C-0.40	0.29$\bar{x}$-0.32	0.66$\bar{x}$-0.96
4,4'-DDE	0.70C-0.54	0.26$\bar{x}$-1.17	0.39$\bar{x}$-1.04
4,4'-DDT	0.79C-3.28	0.42$\bar{x}$+0.19	0.65$\bar{x}$-0.58
Dibenzo(a,h)anthracene	0.88C+4.72	0.30$\bar{x}$+8.51	0.59$\bar{x}$+0.25
Di-n-butyl phthalate	0.59C+0.71	0.13$\bar{x}$+1.16	0.39$\bar{x}$+0.60
1,2-Dichlorobenzene	0.80C+0.28	0.20$\bar{x}$+0.47	0.24$\bar{x}$+0.39
1,3-Dichlorobenzene	0.86C-0.70	0.25$\bar{x}$+0.68	0.41$\bar{x}$+0.11
1,4-Dichlorobenzene	0.73C-1.47	0.24$\bar{x}$+0.23	0.29$\bar{x}$+0.36
3,3'-Dichlorobenzidine	1.23C-12.65	0.28$\bar{x}$+7.33	0.47$\bar{x}$+3.45
Dieldrin	0.82C-0.16	0.20$\bar{x}$-0.16	0.26$\bar{x}$-0.07
Diethyl phthalate	0.43C+1.00	0.28$\bar{x}$+1.44	0.52$\bar{x}$+0.22
Dimethyl phthalate	0.20C+1.03	0.54$\bar{x}$+0.19	1.05$\bar{x}$-0.92
2,4-Dinitrotoluene	0.92C-4.81	0.12$\bar{x}$+1.06	0.21$\bar{x}$+1.50
2,6-Dinitrotoluene	1.06C-3.60	0.14$\bar{x}$+1.26	0.19$\bar{x}$+0.35
Di-n-octyl phthalate	0.76C-0.79	0.21$\bar{x}$+1.19	0.37$\bar{x}$+1.19
Endosulfan sulfate	0.39C+0.41	0.12$\bar{x}$+2.47	0.63$\bar{x}$-1.03
Endrin aldehyde	0.76C-3.86	0.18$\bar{x}$+3.91	0.73$\bar{x}$-0.62
Fluoranthene	0.81C+1.10	0.22$\bar{x}$-0.73	0.28$\bar{x}$-0.60
Fluorene	0.90C-0.00	0.12$\bar{x}$+0.26	0.13$\bar{x}$+0.61
Heptachlor	0.87C-2.97	0.24$\bar{x}$-0.56	0.50$\bar{x}$-0.23
Heptachlor epoxide	0.92C-1.87	0.33$\bar{x}$-0.46	0.28$\bar{x}$+0.64

TABLE 7
(continued)

Compound	Accuracy, as recovery, x' (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S' (µg/L)
Hexachlorobenzene	0.74C+0.66	0.18$\bar{x}$-0.10	0.43$\bar{x}$-0.52
Hexachlorobutadiene	0.71C-1.01	0.19$\bar{x}$+0.92	0.26$\bar{x}$+0.49
Hexachloroethane	0.73C-0.83	0.17$\bar{x}$+0.67	0.17$\bar{x}$+0.80
Indeno(1,2,3-cd)pyrene	0.78C-3.10	0.29$\bar{x}$+1.46	0.50$\bar{x}$-0.44
Isophorone	1.12C+1.41	0.27$\bar{x}$+0.77	0.33$\bar{x}$+0.26
Naphthalene	0.76C+1.58	0.21$\bar{x}$-0.41	0.30$\bar{x}$-0.68
Nitrobenzene	1.09C-3.05	0.19$\bar{x}$+0.92	0.27$\bar{x}$+0.21
N-Nitrosodi-n-propylamine	1.12C-6.22	0.27$\bar{x}$+0.68	0.44$\bar{x}$+0.47
Aroclor 1260	0.81C-10.86	0.35$\bar{x}$+3.61	0.43$\bar{x}$+1.82
Phenanthrene	0.87C+0.06	0.12$\bar{x}$+0.57	0.15$\bar{x}$+0.25
Pyrene	0.84C-0.16	0.16$\bar{x}$+0.06	0.15$\bar{x}$+0.31
1,2,4-Trichlorobenzene	0.94C-0.79	0.15$\bar{x}$+0.85	0.21$\bar{x}$+0.39
4-Chloro-3-methylphenol	0.84C+0.35	0.23$\bar{x}$+0.75	0.29$\bar{x}$+1.31
2-Chlorophenol	0.78C+0.29	0.18$\bar{x}$+1.46	0.28$\bar{x}$+0.97
2,4-Dichlorophenol	0.87C-0.13	0.15$\bar{x}$+1.25	0.21$\bar{x}$+1.28
2,4-Dimethylphenol	0.71C+4.41	0.16$\bar{x}$+1.21	0.22$\bar{x}$+1.31
2,4-Dinitrophenol	0.81C-18.04	0.38$\bar{x}$+2.36	0.42$\bar{x}$+26.29
2-Methyl-4,6-dinitrophenol	1.04C-28.04	0.10$\bar{x}$+42.29	0.26$\bar{x}$+23.10
2-Nitrophenol	0.07C-1.15	0.16$\bar{x}$+1.94	0.27$\bar{x}$+2.60
4-Nitrophenol	0.61C-1.22	0.38$\bar{x}$+2.57	0.44$\bar{x}$+3.24
Pentachlorophenol	0.93C+1.99	0.24$\bar{x}$+3.03	0.30$\bar{x}$+4.33
Phenol	0.43C+1.26	0.26$\bar{x}$+0.73	0.35$\bar{x}$+0.58
2,4,6-Trichlorophenol	0.91C-0.18	0.16$\bar{x}$+2.22	0.22$\bar{x}$+1.81

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration of $\bar{x}$, in µg/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{x}$, in µg/L.

C = True value for the concentration, in µg/L.

$\bar{x}$ = Average recovery found for measurements of samples containing a concentration of C, in µg/L.

[a] Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 8

EXTRACTION EFFICIENCY AND AQUEOUS STABILITY RESULTS

Compound	Percent Recovery on Day 0		Percent Recovery on Day 7	
	Mean	RSD	Mean	RSD
3-Amino-9-ethylcarbazole	80	8	73	3
4-Chloro-1,2-phenylenediamine	91	1	108	4
4-Chloro-1,3-phenylenediamine	84	3	70	3
1,2-Dibromo-3-chloropropane	97	2	98	5
Dinoseb	99	3	97	6
Parathion	100	2	103	4
4,4'-Methylenebis(N,N-dimethylaniline)	108	4	90	4
5-Nitro-o-toluidine	99	10	93	4
2-Picoline	80	4	83	4
Tetraethyl dithiopyrophosphate	92	7	70	1

Data taken from Reference 6.

TABLE 9

MEAN PERCENT RECOVERIES AND PERCENT RSD VALUES FOR SEMIVOLATILE ORGANICS FROM SPIKED CLAY SOIL AND TOPSOIL BY AUTOMATED SOXHLET EXTRACTION (METHOD 3541) WITH HEXANE-ACETONE (1:1)[a]

Compound	Clay Soil		Topsoil	
	Mean Recovery	RSD	Mean Recovery	RSD
1,3-Dichlorobenzene	0	--	0	--
1,2-Dichlorobenzene	0	--	0	--
Nitrobenzene	0	--	0	--
Benzal chloride	0	--	0	--
Benzotrichloride	0	--	0	--
4-Chloro-2-nitrotoluene	0	--	0	--
Hexachlorocyclopentadiene	4.1	15	7.8	23
2,4-Dichloronitrobenzene	35.2	7.6	21.2	15
3,4-Dichloronitrobenzene	34.9	15	20.4	11
Pentachlorobenzene	13.7	7.3	14.8	13
2,3,4,5-Tetrachloronitrobenzene	55.9	6.7	50.4	6.0
Benefin	62.6	4.8	62.7	2.9
alpha-BHC	58.2	7.3	54.8	4.8
Hexachlorobenzene	26.9	13	25.1	5.7
delta-BHC	95.8	4.6	99.2	1.3
Heptachlor	46.9	9.2	49.1	6.3
Aldrin	97.7	12	102	7.4
Isopropalin	102	4.3	105	2.3
Heptachlor epoxide	90.4	4.4	93.6	2.4
trans-Chlordane	90.1	4.5	95.0	2.3
Endosulfan I	96.3	4.4	101	2.2
Dieldrin	129	4.7	104	1.9
2,5-Dichlorophenyl-4-nitrophenyl ether	110	4.1	112	2.1
Endrin	102	4.5	106	3.7
Endosulfan II	104	4.1	105	0.4
p,p'-DDT	134	2.1	111	2.0
2,3,6-Trichlorophenyl-4'-nitrophenyl ether	110	4.8	110	2.8
2,3,4-Trichlorophenyl-4'-nitrophenyl ether	112	4.4	112	3.3
Mirex	104	5.3	108	2.2

[a] The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g; the spiking concentration was 500 ng/g, except for the surrogate compounds at 1000 ng/g, 2,5-Dichlorophenyl-4-nitrophenyl ether, 2,3,6-Trichlorophenyl-4-nitrophenyl ether, and 2,3,4-Trichlorophenyl-4-nitrophenyl ether at 1500 ng/g, Nitrobenzene at 2000 ng/g, and 1,3-Dichlorobenzene and 1,2-Dichlorobenzene at 5000 ng/g.

TABLE 10

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR THE EXTRACTION OF SEMIVOLATILE ORGANICS FROM SPIKED CLAY BY AUTOMATED SOXHLET (METHOD 3541)[a]

Compound	Mean Recovery	RSD
Phenol	47.8	5.6
Bis(2-chloroethyl)ether	25.4	13
2-Chlorophenol	42.7	4.3
Benzyl alcohol	55.9	7.2
2-Methylphenol	17.6	6.6
Bis(2-chloroisopropyl)ether	15.0	15
4-Methylphenol	23.4	6.7
N-Nitroso-di-n-propylamine	41.4	6.2
Nitrobenzene	28.2	7.7
Isophorone	56.1	4.2
2-Nitrophenol	36.0	6.5
2,4-Dimethylphenol	50.1	5.7
Benzoic acid	40.6	7.7
Bis(2-chloroethoxy)methane	44.1	3.0
2,4-Dichlorophenol	55.6	4.6
1,2,4-Trichlorobenzene	18.1	31
Naphthalene	26.2	15
4-Chloroaniline	55.7	12
4-Chloro-3-methylphenol	65.1	5.1
2-Methylnaphthalene	47.0	8.6
Hexachlorocyclopentadiene	19.3	19
2,4,6-Trichlorophenol	70.2	6.3
2,4,5-Trichlorophenol	26.8	2.9
2-Chloronaphthalene	61.2	6.0
2-Nitroaniline	73.8	6.0
Dimethyl phthalate	74.6	5.2
Acenaphthylene	71.6	5.7
3-Nitroaniline	77.6	5.3
Acenaphthene	79.2	4.0
2,4-Dinitrophenol	91.9	8.9
4-Nitrophenol	62.9	16
Dibenzofuran	82.1	5.9
2,4-Dinitrotoluene	84.2	5.4
2,6-Dinitrotoluene	68.3	5.8
Diethyl phthalate	74.9	5.4
4-Chlorophenyl-phenyl ether	67.2	3.2
Fluorene	82.1	3.4
4-Nitroaniline	79.0	7.9

TABLE 10
(continued)

Compound	Mean Recovery	RSD
4,6-Dinitro-2-methylphenol	63.4	6.8
N-Nitrosodiphenylamine	77.0	3.4
4-Bromophenyl-phenyl ether	62.4	3.0
Hexachlorobenzene	72.6	3.7
Pentachlorophenol	62.7	6.1
Phenanthrene	83.9	5.4
Anthracene	96.3	3.9
Di-n-butyl phthalate	78.3	40
Fluoranthene	87.7	6.9
Pyrene	102	0.8
Butyl benzyl phthalate	66.3	5.2
3,3'-Dichlorobenzidine	25.2	11
Benzo(a)anthracene	73.4	3.8
Bis(2-ethylhexyl) phthalate	77.2	4.8
Chrysene	76.2	4.4
Di-n-octyl phthalate	83.1	4.8
Benzo(b)fluoranthene	82.7	5.0
Benzo(k)fluoranthene	71.7	4.1
Benzo(a)pyrene	71.7	4.1
Indeno(1,2,3-cd)pyrene	72.2	4.3
Dibenzo(a,h)anthracene	66.7	6.3
Benzo(g,h,i)perylene	63.9	8.0
1,2-Dichlorobenzene	0	--
1,3-Dichlorobenzene	0	--
1,4-Dichlorobenzene	0	--
Hexachloroethane	0	--
Hexachlorobutadiene	0	--

[a] Number of determinations was three. The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g clay soil; the spike concentration was 6 mg/kg per compound. The sample was allowed to equilibrate 1 hour after spiking.

Data taken from Reference 7.

TABLE 11

PRECISION AND BIAS VALUES FOR METHOD 3542[1]

Compound	Mean Recovery	Standard Deviation	Relative Standard Deviation Percent
2-Fluorophenol	74.6	28.6	38.3
Phenol-d_5	77.8	27.7	35.6
Nitrobenzene-d_5	65.6	32.5	49.6
2-Fluorobiphenyl	75.9	30.3	39.9
2,4,6-Tribromophenol	67.0	34.0	50.7
Terphenyl-d_{14}	78.6	32.4	41.3

[1] The surrogate values shown in Table 11 represent mean recoveries for surrogates in all Method 0010 matrices in a field dynamic spiking study.

TABLE 12

ACCELERATED SOLVENT EXTRACTION (METHOD 3545) RECOVERY VALUES
AS PERCENT OF SOXTEC™

COMPOUND	CLAY			LOAM			SAND			AVE
	LOW	MID	HIGH	LOW	MID	HIGH	LOW	MID	HIGH	
Phenol	93.3	78.7	135.9	73.9	82.8	124.6	108.8	130.6	89.7	**102.0**
Bis(2-chloroethyl) ether	102.1	85.1	109.1	96.0	88.0	103.6	122.3	119.9	90.8	**101.9**
2-Chlorophenol	100.8	82.6	115.0	93.8	88.9	111.1	115.0	115.3	91.9	**101.6**
1,3-Dichlorobenzene	127.7	129.7	110.0	*364.2	129.9	119.0	*241.3	*163.7	107.1	**120.6**
1,4-Dichlorobenzene	127.9	127.0	110.5	*365.9	127.8	116.4	*309.6	*164.1	105.8	**119.2**
1,2-Dichlorobenzene	116.8	115.8	101.3	*159.2	113.4	105.5	*189.3	134.0	100.4	**112.5**
2-Methylphenol	98.9	82.1	119.7	87.6	89.4	111.0	133.2	128.0	92.1	**104.7**
Bis(2-chloroisopropyl)ether	109.4	71.5	108.0	81.8	81.0	88.6	118.1	148.3	94.8	**100.2**
o-Toluidine	100.0	89.7	117.2	100.0	*152.5	120.3	100.0	*199.5	102.7	**110.3**
N-Nitroso-di-n-propylamine	103.0	79.1	107.7	83.9	88.1	96.2	109.9	123.3	91.4	**98.1**
Hexachloroethane	97.1	125.1	111.0	*245.4	117.1	128.1	*566.7	147.9	103.7	**118.6**
Nitrobenzene	104.8	82.4	106.6	86.8	84.6	101.7	119.7	122.1	93.3	**100.2**
Isophorone	100.0	86.4	98.2	87.1	87.5	109.7	135.5	118.4	92.7	**101.7**
2,4-Dimethylphenol	100.0	104.5	140.0	100.0	114.4	123.1	100.0	*180.6	96.3	**109.8**
2-Nitrophenol	80.7	80.5	107.9	91.4	86.7	103.2	122.1	107.1	87.0	**96.3**
Bis(chloroethoxy)methane	94.4	80.6	94.7	86.5	84.4	99.6	130.6	110.7	93.2	**97.2**
2,4-Dichlorophenol	88.9	87.8	111.4	85.9	87.6	103.5	123.3	107.0	92.1	**98.6**
1,2,4-Trichlorobenzene	98.0	97.8	98.8	123.0	93.7	94.5	137.0	99.4	95.3	**104.2**
Naphthalene	101.7	97.2	123.6	113.2	102.9	129.5	*174.5	114.0	89.8	**106.1**
4-Chloroaniline	100.0	*150.2	*162.4	100.0	125.5	*263.6	100.0	*250.8	114.9	**108.1**
Hexachlorobutadiene	101.1	98.7	102.2	124.1	90.3	98.0	134.9	96.1	96.8	**104.7**
4-Chloro-3-methylphenol	90.4	80.2	114.7	79.0	85.2	109.8	131.6	116.2	90.1	**99.7**
2-Methylnaphthalene	93.2	89.9	94.6	104.1	92.2	105.9	146.2	99.1	93.3	**102.1**
Hexachlorocyclopentadiene	100.0	100.0	0.0	100.0	100.0	6.8	100.0	100.0	*238.3	**75.8**
2,4,6-Trichlorophenol	94.6	90.0	112.0	84.2	91.2	103.6	101.6	95.9	89.8	**95.9**
2,4,5-Trichlorophenol	84.4	91.9	109.6	96.1	80.7	103.6	108.9	83.9	87.9	**94.1**
2-Chloronaphthalene	100.0	91.3	93.6	97.6	93.4	98.3	106.8	93.0	92.0	**96.2**
2-Nitroaniline	90.0	83.4	97.4	71.3	88.4	89.9	112.1	113.3	87.7	**92.6**
2,6-Dinitrotoluene	83.1	90.6	91.6	86.4	90.6	90.3	104.3	84.7	90.9	**90.3**
Acenaphthylene	104.9	95.9	100.5	99.0	97.9	108.8	118.5	97.8	92.0	**101.7**
3-Nitroaniline	*224.0	115.6	97.6	100.0	111.8	107.8	0.0	111.7	99.0	**92.9**
Acenaphthene	102.1	92.6	97.6	97.2	96.9	104.4	114.2	92.0	89.0	**98.4**
4-Nitrophenol	0.0	93.2	121.5	18.1	87.1	116.6	69.1	90.5	84.5	**75.6**
2,4-Dinitrotoluene	73.9	91.9	100.2	84.7	93.8	98.9	100.9	84.3	87.3	**90.7**

TABLE 12 (cont.)

ACCELERATED SOLVENT EXTRACTION (METHOD 3545) RECOVERY VALUES
AS PERCENT OF SOXTEC™

COMPOUND	CLAY			LOAM			SAND			AVE
	LOW	MID	HIGH	LOW	MID	HIGH	LOW	MID	HIGH	
Dibenzofuran	89.5	91.7	109.3	98.5	92.2	111.4	113.8	92.7	90.4	**98.8**
4-Chlorophenyl phenyl ether	83.0	94.5	98.7	95.7	94.3	94.2	111.4	87.7	90.3	**94.4**
Fluorene	85.2	94.9	89.2	102.0	95.5	93.8	121.3	85.7	90.9	**95.4**
4-Nitroaniline	77.8	114.8	94.5	129.6	103.6	95.4	*154.1	89.3	87.5	**99.1**
N-Nitrosodiphenylamine	82.6	96.7	93.8	92.9	93.4	116.4	97.5	110.9	86.7	**96.8**
4-Bromophenyl phenyl ether	85.6	92.9	92.8	91.1	107.6	89.4	118.0	97.5	87.1	**95.8**
Hexachlorobenzene	95.4	91.7	92.3	95.4	93.6	83.7	106.8	94.3	90.0	**93.7**
Pentachlorophenol	68.2	85.9	107.7	53.2	89.8	88.1	96.6	59.8	81.3	**81.2**
Phenanthrene	92.1	93.7	93.3	100.0	97.8	113.3	124.4	101.0	89.9	**100.6**
Anthracene	101.6	95.0	93.5	92.5	101.8	118.4	123.0	94.5	90.6	**101.2**
Carbazole	94.4	99.3	96.6	105.5	96.7	111.4	115.7	83.2	88.9	**99.1**
Fluoranthene	109.9	101.4	94.3	111.6	96.6	109.6	123.2	85.4	92.7	**102.7**
Pyrene	106.5	105.8	107.6	116.7	90.7	127.5	103.4	95.5	93.2	**105.2**
3,3'-Dichlorobenzidine	100.0	*492.3	131.4	100.0	*217.6	*167.6	100.0	*748.8	100.0	**116.5**
Benzo(a)anthracene	98.1	107.0	98.4	119.3	98.6	104.0	105.0	93.4	89.3	**101.5**
Chrysene	100.0	108.5	100.2	116.8	93.0	117.0	106.7	93.6	90.2	**102.9**
Benzo(b)fluoranthene	106.6	109.9	75.6	121.7	100.7	93.9	106.9	81.9	93.6	**99.0**
Benzo(k)fluoranthene	102.4	105.2	88.4	125.5	99.4	95.1	144.7	89.2	78.1	**103.1**
Benzo(a)pyrene	107.9	105.5	80.8	122.3	97.7	104.6	101.7	86.2	92.0	**99.9**
Indeno(1,2,3-cd)pyrene	95.1	105.7	93.8	126.0	105.2	90.4	133.6	82.6	91.9	**102.7**
Dibenz(a,h)anthracene	85.0	102.6	82.0	118.8	100.7	91.9	142.3	71.0	93.1	**98.6**
Benzo(g,h,i)perylene	98.0	0.0	81.2	0.0	33.6	78.6	128.7	83.0	94.2	**66.4**
Average	**95.1**	**94.3**	**101.0**	**95.5**	**96.5**	**104.1**	**113.0**	**100.9**	**92.5**	

* Values greater than 150% were not used to determine the averages, but the 0% values were used.

TABLE 13

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SEDIMENT EC-1, USING METHOD 3561 (SFE - SOLID TRAP)

Compound	Certified Value (mg/kg)	SFE Value[a] (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	(27.9)[b]	41.3 ± 3.6	(148)	8.7
Acenaphthylene	(0.8)	0.9 ± 0.1	(112)	11.1
Acenaphthene	(0.2)	0.2 ± 0.01	(100)	0.05
Fluorene	(15.3)	15.6 ± 1.8	(102)	11.5
Phenanthrene	15.8 ± 1.2	16.1 ± 1.8	102	11.2
Anthracene	(1.3)	1.1 ± 0.2	(88)	18.2
Fluoranthene	23.2 ± 2.0	24.1 ± 2.1	104	8.7
Pyrene	16.7 ± 2.0	17.2 ± 1.9	103	11.0
Benz(a)anthracene	8.7 ± 0.8	8.8 ± 1.0	101	11.4
Chrysene	(9.2)	7.9 ± 0.9	(86)	11.4
Benzo(b)fluoranthene	7.9 ± 0.9	8.5 ± 1.1	108	12.9
Benzo(k)fluoranthene	4.4 ± 0.5	4.1 ± 0.5	91	12.2
Benzo(a)pyrene	5.3 ± 0.7	5.1 ± 0.6	96	11.8
Indeno(1,2,3-cd)pyrene	5.7 ± 0.6	5.2 ± 0.6	91	11.5
Benzo(g,h,i)perylene	4.9 ± 0.7	4.3 ± 0.5	88	11.6
Dibenz(a,h)anthracene	(1.3)	1.1 ± 0.2	(85)	18.2

[a] Relative standard deviations for the SFE values are based on six replicate extractions.

[b] Values in parentheses were obtained from, or compared to, Soxhlet extraction results which were not certified.

Data are taken from Reference 10.

TABLE 14

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SEDIMENT HS-3, USING METHOD 3561 (SFE - SOLID TRAP)

Compound	Certified Value (mg/kg)	SFE Value[a] (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	9.0 ± 0.7	7.4 ± 0.6	82	8.1
Acenaphthylene	0.3 ± 0.1	0.4 ± 0.1	133	25.0
Acenaphthene	4.5 ± 1.5	3.3 ± 0.3	73	9.0
Fluorene	13.6 ± 3.1	10.4 ± 1.3	77	12.5
Phenanthrene	85.0 ± 20.0	86.2 ± 9.5	101	11.0
Anthracene	13.4 ± 0.5	12.1 ± 1.5	90	12.4
Fluoranthene	60.0 ± 9.0	54.0 ± 6.1	90	11.3
Pyrene	39.0 ± 9.0	32.7 ± 3.7	84	11.3
Benz(a)anthracene	14.6 ± 2.0	12.1 ± 1.3	83	10.7
Chrysene	14.1 ± 2.0	12.0 ± 1.3	85	10.8
Benzo(b)fluoranthene	7.7 ± 1.2	8.4 ± 0.9	109	10.7
Benzo(k)fluoranthene	2.8 ± 2.0	3.2 ± 0.5	114	15.6
Benzo(a)pyrene	7.4 ± 3.6	6.6 ± 0.8	89	12.1
Indeno(1,2,3-cd)pyrene	5.0 ± 2.0	4.5 ± 0.6	90	13.3
Benzo(g,h,i)perylene	5.4 ± 1.3	4.4 ± 0.6	82	13.6
Dibenz(a,h)anthracene	1.3 ± 0.5	1.1 ± 0.3	85	27.3

[a] Relative standard deviations for the SFE values are based on three replicate extractions.

Data are taken from Reference 10.

TABLE 15

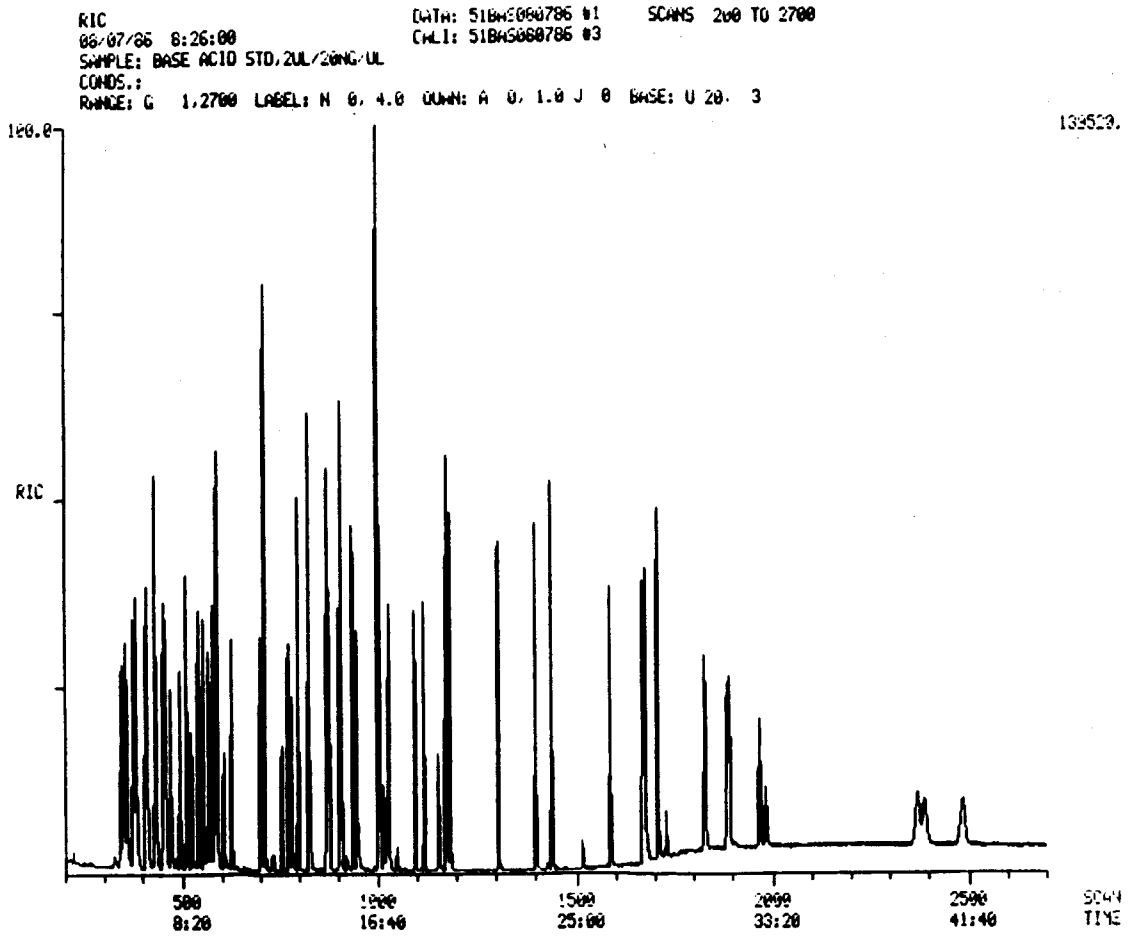
SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SOIL SRS103-100, USING METHOD 3561 (SFE - LIQUID TRAP)

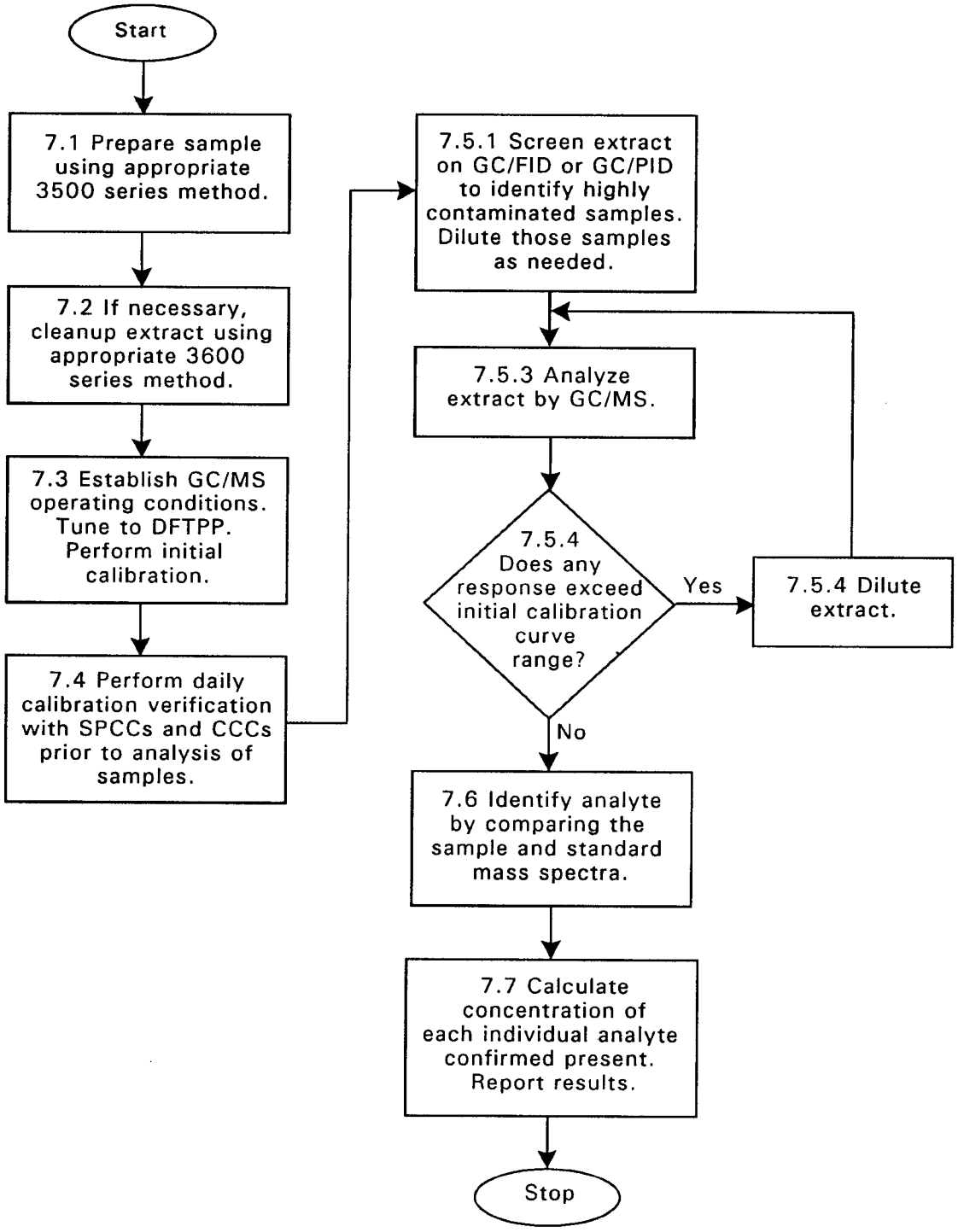
Compound	Certified Value (mg/kg)			SFE Value[a] (mg/kg)	Percent of Certified Value	SFE RSD
Naphthalene	32.4	±	8.2	29.55	91	10.5
2-Methylnaphthalene	62.1	±	11.5	76.13	122	2.0
Acenaphthene	632	±	105	577.28	91	2.9
Dibenzofuran	307	±	49	302.25	98	4.1
Fluorene	492	±	78	427.15	87	3.0
Phenanthrene	1618	±	340	1278.03	79	3.4
Anthracene	422	±	49	400.80	95	2.6
Fluoranthene	1280	±	220	1019.13	80	4.5
Pyrene	1033	±	285	911.82	88	3.1
Benz(a)anthracene	252	±	38	225.50	89	4.8
Chrysene	297	±	26	283.00	95	3.8
Benzo(b)fluoranthene + Benzo(k)fluoranthene	153	±	22	130.88	86	10.7
Benzo(a)pyrene	97.2	±	17.1	58.28	60	6.5

[a] Relative standard deviations for the SFE values are based on four replicate extractions.

Data are taken from Reference 11.

FIGURE 1
GAS CHROMATOGRAM OF BASE/NEUTRAL AND ACID CALIBRATION STANDARD




Start
7.1 Prepare sample using appropriate 3500 series method.
7.2 If necessary, cleanup extract using appropriate 3600 series method.
7.3 Establish GC/MS operating conditions. Tune to DFTPP. Perform initial calibration.
7.4 Perform daily calibration verification with SPCCs and CCCs prior to analysis of samples.
7.5.1 Screen extract on GC/FID or GC/PID to identify highly contaminated samples. Dilute those samples as needed.
7.5.3 Analyze extract by GC/MS.
7.5.4 Does any response exceed initial calibration curve range?
Yes
7.5.4 Dilute extract.
No
7.6 Identify analyte by comparing the sample and standard mass spectra.
7.7 Calculate concentration of each individual analyte confirmed present. Report results.
Stop

Appendix C
EPA Test Method 6010 C

METHOD 6010C

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) may be used to determine trace elements in solution. The method is applicable to all of the elements listed below. With the exception of groundwater samples, all aqueous and solid matrices require acid digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion. Samples which are not digested require either an internal standard or should be matrix-matched with the standards. If either option is used, instrument software should be programmed to correct for intensity differences of the internal standard between samples and standards. Refer to Chapter Three for the appropriate digestion procedures.

Element	Symbol	CAS Number	Element	Symbol	CAS Number
Aluminum	Al	7429-90-5	Mercury	Hg	7439-97-6
Antimony	Sb	7440-36-0	Molybdenum	Mo	7439-98-7
Arsenic	As	7440-38-2	Nickel	Ni	7440-02-0
Barium	Ba	7440-39-3	Phosphorus	P	7723-14-0
Beryllium	Be	7440-41-7	Potassium	K	7440-09-7
Boron	B	7440-42-8	Selenium	Se	7782-49-2
Cadmium	Cd	7440-43-9	Silica	SiO_2	7631-86-9
Calcium	Ca	7440-70-2	Silver	Ag	7440-22-4
Chromium	Cr	7440-47-3	Sodium	Na	7440-23-5
Cobalt	Co	7440-48-4	Strotium	Sr	7440-24-6
Copper	Cu	7440-50-8	Thallium	Tl	7440-28-0
Iron	Fe	7439-89-6	Tin	Sn	7440-31-5
Lead	Pb	7439-92-1	Titanium	Ti	7440-32-6
Lithium	Li	7439-93-2	Vanadium	V	7440-62-2
Magnesium	Mg	7439-95-4	Zinc	Zn	7440-66-6
Manganese	Mn	7439-96-5			

1.2 Table 1 lists the elements for which this method has been validated. The sensitivity and the optimum and linear ranges for each element will vary with the wavelength, spectrometer, matrix, and operating conditions. Table 1 lists the recommended analytical wavelengths and estimated instrumental detection limits for the elements in clean aqueous matrices with insignificant background interferences. Other elements and matrices may be analyzed by this method if performance at the concentrations of interest (see Sec. 9.0) is demonstrated.

1.3 In addition, method detection limits (MDLs) should be empirically established annually, at a minimum, for each matrix type analyzed (refer to Chapters One and Three for guidance) and are required for each preparatory/determinative method combination used. MDLs are instrument-specific, so an MDL study must be conducted for each instrument in a laboratory.

1.4 Analysts should clearly understand the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to using the method for analysis.

1.5 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two, Sec. 2.1, for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.6 Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation methods (see Chapter Three). When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis (refer to Sec. 1.1).

2.2 This method describes multielemental determinations by ICP-AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices.

2.3 Background correction is required for trace element determination. Background emission 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 should be as free as possible from spectral interference and should 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. The possibility of additional interferences identified in Sec. 4.0 should also be recognized and appropriate corrections made; tests for their presence are described in Secs. 9.5 and 9.6. Alternatively, users may choose multivariate calibration methods. In this case, point selections for background correction are superfluous since whole spectral regions are processed.

3.0 DEFINITIONS

Refer to Chapter One and Chapter Three for applicable definitions.

4.0 INTERFERENCES

4.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

4.1.1 Compensation for background emission and stray light can usually be conducted by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

4.1.2 To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single-element solutions are sufficient. However, for analytes such as iron that may be found in the sample at high concentration, a more appropriate test would be to use a concentration near the upper limit of the analytical range (refer to Chapter Three).

4.1.3 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated for by equations that correct for interelement contributions. Instruments that use equations for interelement correction require that the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive or positively biased determinations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off-line or on-line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelengths are given in Table 2. For multivariate calibration methods using whole spectral

regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

4.1.4 When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e., false positive analyte concentrations) arising from 100 mg/L of the interference element. For example, if As is to be determined at 193.696 nm in a sample containing approximately 10 mg/L of Al, according to Table 2, 100 mg/L of Al will yield a false positive signal for an As level equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al will result in a false positive signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects must be evaluated for each individual instrument, since the intensities will vary.

4.1.5 Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users should continuously note that some samples may contain uncommon elements that could contribute spectral interferences.

4.1.6 The interference effects must be evaluated for each individual instrument, whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences (Table 2) as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.

4.1.7 Users of sequential instruments must verify the absence of spectral interference by scanning over a range of 0.5 nm centered on the wavelength of interest for several samples. The range for lead, for example, would be from 220.6 to 220.1 nm. This procedure must be repeated whenever a new matrix is to be analyzed and when a new calibration curve using different instrumental conditions is to be prepared. Samples that show an elevated background emission across the range may be background corrected by applying a correction factor equal to the emission adjacent to the line or at two points on either side of the line and interpolating between them. An alternate wavelength that does not exhibit a background shift or spectral overlap may also be used.

4.1.8 If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and dividing by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range, in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected.

The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

4.1.9 When interelement corrections are applied, their accuracy should be verified daily, by analyzing spectral interference check solutions. The correction factors or multivariate correction matrices tested on a daily basis must be within the 20% criteria for five consecutive days. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change occurs, such as one in the torch, nebulizer, injector, or plasma conditions. Standard solutions should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

4.1.10 When interelement corrections are <u>not</u> used, verification of absence of interferences is required.

4.1.10.1 One method to verify the absence of interferences is to use a computer software routine for comparing the determinative data to established limits for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration (i.e., greater than the analyte instrument detection limit), or a false negative analyte concentration (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).

4.1.10.2 Another way to verify the absence of interferences is to analyze an interference check solution which contains similar concentrations of the major components of the samples (>10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is $\geq$20% of the analyte concentration, the analyte must be determined using (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.

4.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, by using a peristaltic pump, by using an internal standard, or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, by using a tip washer, by using a high solids nebulizer, or by diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance. This may be accomplished with the use of mass flow controllers. The test described in Sec. 9.6 will help determine if a physical interference is present.

4.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard

addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.3.1 At the analyst's discretion, the method of standard additions (MSA) can be used. This technique can be useful when certain interferences are encountered. The analyst is encouraged to review the information in Sec. 4.0 to deal with the majority of interferences likely to be encountered when using this method. Refer to Method 7000 for a more detailed discussion of the MSA.

4.3.2 An alternative to using the method of standard additions is to use the internal standard technique. Add one or more elements that are both not found in the samples and verified to not cause an interelement spectral interference to the samples, standards, and blanks. Yttrium or scandium are often used. The concentration should be sufficient for optimum precision, but not so high as to alter the salt concentration of the matrix. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation. This technique is very useful in overcoming matrix interferences, especially in high solids matrices.

4.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals equal to or less than the method detection limit should be noted. Until the required rinse time is established, it is suggested that the rinse period be at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon the project specific DQOs.

4.5 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

4.6 The dashes in Table 2 indicate that no measurable interferences were observed even at higher interferant concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

4.7 The calibration blank (Sec. 7.5.1) may restrict the sensitivity of the detection limit or degrade the precision and accuracy of the analysis. Chapter Three should be consulted for clean chemistry methods and procedures necessary in reducing the magnitude and variability of the calibration blank.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

5.2 Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a hood and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection when working with these reagents. Hydrofluoric acid is a very toxic acid and penetrates the skin and tissues deeply if not treated immediately. Injury occurs in two stages; first, by hydration that induces tissue necrosis and then by penetration of fluoride ions deep into the tissue and by reaction with calcium. Boric acid and other complexing reagents and appropriate treatment agents should be administered immediately. Consult appropriate safety literature and have the appropriate treatment materials readily available prior to working with this acid. See Method 3052 for specific suggestions for handling hydrofluoric acid from a safety and an instrument standpoint.

5.3 Many metal salts, including, but not limited to, those of osmium, are extremely toxic if inhaled or swallowed. Extreme care must be taken to ensure that samples and standards are handled properly and that all exhaust gases are properly vented. Wash hands thoroughly after handling.

6.0 EQUIPMENT AND SUPPLIES

6.1 Inductively coupled argon plasma emission spectrometer

6.1.1 Computer-controlled emission spectrometer with background correction.

6.1.2 Radio-frequency generator compliant with FCC regulations.

6.1.3 Optional mass flow controller for argon nebulizer gas supply.

6.1.4 Optional peristaltic pump.

6.1.5 Optional autosampler.

6.1.6 Argon gas supply - high purity.

6.2 Volumetric flasks of suitable precision and accuracy.

6.3 Volumetric pipets of suitable precision and accuracy.

7.0 REAGENTS AND STANDARDS

7.1 Reagent or trace metals grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high

purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration of the contamination is less than the MDL, then the reagent is acceptable.

7.1.1 Hydrochloric acid (conc), HCl.

7.1.2 Hydrochloric acid HCl (1:1) - Add 500 mL concentrated HCl to 400 mL water and dilute to 1 L in an appropriately- sized beaker.

7.1.3 Nitric acid (conc), HNO_3.

7.1.4 Nitric acid, HNO_3 (1:1) - Add 500 mL concentrated HNO_3 to 400 mL water and dilute to 1 L in an appropriately-sized beaker.

7.2 Reagent water - All references to water in the method refer to reagent water, as defined in Chapter One, unless otherwise specified. Reagent water must be free of interferences.

7.3 Standard stock solutions may be purchased or prepared from ultra-high purity grade chemicals or metals (99.99% pure or greater). With several exceptions specifically noted, all salts must be dried for 1 hour at 105°C.

CAUTION: Many metal salts are extremely toxic if inhaled or swallowed. Wash hands thoroughly after handling.

Typical stock solution preparation procedures follow. Concentrations are calculated based upon the weight of pure metal added, or with the use of the element fraction and the weight of the metal salt added.

NOTE: This section does not apply when analyzing samples prepared by Method 3040.

NOTE: The weight of the analyte is expressed to four significant figures for consistency with the weights below because rounding to two decimal places can contribute up to 4 % error for some of the compounds.

For metals:

$$\text{Concentration (ppm)} = \frac{\text{weight (mg)}}{\text{volume (L)}}$$

For metal salts:

$$\text{Concentration (ppm)} = \frac{\text{weight (mg) x mole fraction}}{\text{volume (L)}}$$

7.3.1 Aluminum solution, stock, 1 mL = 1000 µg Al

Dissolve 1.000 g of aluminum metal, accurately weighed to at least four significant figures, in an acid mixture of 4.0 mL of HCl (1:1) and 1.0 mL of concentrated $HN0_3$ in a beaker. Warm beaker slowly to dissolve the metal. When dissolution is complete, transfer solution quantitatively to a 1000-mL volumetric flask, add an additional 10.0 mL of HCl (1:1) and dilute to volume with reagent water.

7.3.2 Antimony solution, stock, 1 mL = 1000 µg Sb

Dissolve 2.6673 g $K(SbO)C_4H_4O_6$ (element fraction Sb = 0.3749), accurately weighed to at least four significant figures, in reagent water, add 10 mL HCl (1:1), and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.3 Arsenic solution, stock, 1 mL = 1000 µg As

Dissolve 1.3203 g of As_2O_3 (element fraction As = 0.7574), accurately weighed to at least four significant figures, in 100 mL of reagent water containing 0.4 g NaOH. Acidify the solution with 2 mL concentrated HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.4 Barium solution, stock, 1 mL = 1000 µg Ba

Dissolve 1.5163 g $BaCl_2$ (element fraction Ba = 0.6595), dried at 250°C for 2 hours, accurately weighed to at least four significant figures, in 10 mL of reagent water with 1 mL HCl (1:1). Add 10.0 mL HCl (1:1) and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.5 Beryllium solution, stock, 1 mL = 1000 µg Be

Do not dry. Dissolve 19.6463 g $BeSO_4 \cdot 4H_2O$ (element fraction Be = 0.0509), accurately weighed to at least four significant figures, in reagent water, add 10.0 mL concentrated HNO_3, and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.6 Boron solution, stock, 1 mL = 1000 µg B

Do not dry. Dissolve 5.716 g anhydrous H_3BO_3 (B fraction = 0.1749), accurately weighed to at least four significant figures, in reagent water and dilute in a 1-L volumetric flask with reagent water. Transfer immediately after mixing in a clean polytetrafluoroethylene (PTFE) bottle to minimize any leaching of boron from the glass container. The use of a non-glass volumetric flask is recommended to avoid boron contamination from glassware.

7.3.7 Cadmium solution, stock, 1 mL = 1000 µg Cd

Dissolve 1.1423 g CdO (element fraction Cd = 0.8754), accurately weighed to at least four significant figures, in a minimum amount of (1:1) HNO_3. Heat to increase the rate of dissolution. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.8 Calcium solution, stock, 1 mL = 1000 µg Ca

Suspend 2.4969 g $CaCO_3$ (element Ca fraction = 0.4005), dried at 180°C for 1 hour before weighing, accurately weighed to at least four significant figures, in reagent water and dissolve cautiously with a minimum amount of (1:1) HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.9 Chromium solution, stock, 1 mL = 1000 µg Cr

Dissolve 1.9231 g CrO_3 (element fraction Cr = 0.5200), accurately weighed to at least four significant figures, in reagent water. When dissolution is complete, acidify with 10 mL concentrated HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.10 Cobalt solution, stock, 1 mL = 1000 µg Co

Dissolve 1.000 g of cobalt metal, accurately weighed to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10.0 mL HCl (1:1) and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.11 Copper solution, stock, 1 mL = 1000 µg Cu

Dissolve 1.2564 g CuO (element fraction Cu = 0.7989), accurately weighed to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.12 Iron solution, stock, 1 mL = 1000 µg Fe

Dissolve 1.4298 g Fe_2O_3 (element fraction Fe = 0.6994), accurately weighed to at least four significant figures, in a warm mixture of 20 mL HCl (1:1) and 2 mL of concentrated HNO_3. Cool, add an additional 5.0 mL of concentrated HNO_3, and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.13 Lead solution, stock, 1 mL = 1000 µg Pb

Dissolve 1.5985 g $Pb(NO_3)_2$ (element fraction Pb = 0.6256), accurately weighed to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10 mL (1:1) HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.14 Lithium solution, stock, 1 mL = 1000 µg Li

Dissolve 5.3248 g lithium carbonate (element fraction Li = 0.1878), accurately weighed to at least four significant figures, in a minimum amount of HCl (1:1) and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.15 Magnesium solution, stock, 1 mL = 1000 µg Mg

Dissolve 1.6584 g MgO (element fraction Mg = 0.6030), accurately weighed to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10.0 mL (1:1) concentrated HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.16 Manganese solution, stock, 1 mL = 1000 µg Mn

Dissolve 1.00 g of manganese metal, accurately weighed to at least four significant figures, in acid mixture (10 mL concentrated HCl and 1 mL concentrated HNO_3) and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.17 Mercury solution, stock, 1 mL = 1000 µg Hg

Do not dry, **highly toxic element**. Dissolve 1.354 g $HgCl_2$ (Hg fraction = 0.7388) in reagent water. Add 50.0 mL concentrated HNO_3 and dilute to volume in 1000-mL volumetric flask with reagent water.

7.3.18 Molybdenum solution, stock, 1 mL = 1000 µg Mo

Dissolve 1.7325 g $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ (element fraction Mo = 0.5772), accurately weighed to at least four significant figures, in reagent water and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.19 Nickel solution, stock, 1 mL = 1000 µg Ni

Dissolve 1.000 g of nickel metal, accurately weighed to at least four significant figures, in 10.0 mL hot concentrated HNO_3, cool, and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.20 Phosphate solution, stock, 1 mL = 1000 µg P

Dissolve 4.3937 g anhydrous KH_2PO_4 (element fraction P = 0.2276), accurately weighed to at least four significant figures, in water. Dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.21 Potassium solution, stock, 1 mL = 1000 µg K

Dissolve 1.9069 g KCl (element fraction K = 0.5244) dried at 110°C, accurately weighed to at least four significant figures, in reagent water, and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.22 Selenium solution, stock, 1 mL = 1000 µg Se

Do not dry. Dissolve 1.6332 g H_2SeO_3 (element fraction Se = 0.6123), accurately weighed to at least four significant figures, in reagent water and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.23 Silica solution, stock, 1 mL = 1000 µg SiO_2

Do not dry. Dissolve 2.964 g NH_4SiF_6, accurately weighed to at least four significant figures, in 200 mL (1:20) HCl with heating at 85°C to dissolve the solid. Let solution cool and dilute to volume in a 1000-mL volumetric flask with reagent water. Store in an PTFE container and protect from light.

7.3.24 Silver solution, stock, 1 mL = 1000 µg Ag

Dissolve 1.5748 g $AgNO_3$ (element fraction Ag = 0.6350), accurately weighed to at least four significant figures, in water and 10 mL concentrated HNO_3. Dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.25 Sodium solution, stock, 1 mL = 1000 µg Na

Dissolve 2.5419 g NaCl (element fraction Na = 0.3934), accurately weighed to at least four significant figures, in reagent water. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.26 Strontium solution, stock, 1 mL = 1000 µg Sr

Dissolve 2.4154 g of strontium nitrate ($Sr(NO_3)_2$) (element fraction Sr = 0.4140), accurately weighed to at least four significant figures, in a 1000-mL flask containing 10 mL of concentrated HCl and 700 mL of reagent water. Dilute to volume with reagent water.

7.3.27 Thallium solution, stock, 1 mL = 1000 µg Tl

Dissolve 1.3034 g $TlNO_3$ (element fraction Tl = 0.7672), accurately weighed to at least four significant figures, in reagent water. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.28 Tin solution, stock, 1 mL = 1000 µg Sn

Dissolve 1.000 g Sn shot, accurately weighed to at least 4 significant figures, in 200 mL HCl (1:1) with heating to dissolve the metal. Let solution cool and dilute with HCl (1:1) in a 1000-mL volumetric flask.

7.3.29 Vanadium solution, stock, 1 mL = 1000 µg V

Dissolve 2.2957 g NH_4VO_3 (element fraction V = 0.4356), accurately weighed to at least four significant figures, in a minimum amount of concentrated HNO_3. Heat to dissolve the metal. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.30 Zinc solution, stock, 1 mL = 1000 µg Zn

Dissolve 1.2447 g ZnO (element fraction Zn = 0.8034), accurately weighed to at least four significant figures, in a minimum amount of dilute HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.3.31 Yttrium solution, stock, 1 mL = 1000 µg Y

Dissolve 4.3081 g $Y(NO_3)_3 \cdot 6H_20$ (element fraction Y = 0.2321), accurately weighed to at least four significant figures, in a minimum amount of dilute HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1000-mL volumetric flask with reagent water.

7.4 Mixed calibration standard solutions

Prepare mixed calibration standard solutions (see Table 3) by combining appropriate volumes of the stock solutions above in volumetric flasks. Add the appropriate types and volumes of acids so that the standards are matrix-matched with the sample digestates. Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to FEP fluorocarbon or previously unused polyethylene or polypropylene bottles for storage. For all intermediate and working standards, especially low level standards (i.e., <1 ppm), stability must be demonstrated prior to use. Freshly-mixed standards should be prepared, as needed, with the realization that concentration can change with age. (Refer to Sec. 10.4.1 for guidance on determining the viability of standards.) Some typical calibration standard combinations are listed in Table 3.

NOTE: If the addition of silver to the recommended acid combination initially results in a precipitate, then add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. For this acid combination, the silver concentration should be limited to 2 mg/L. Silver is stable under these conditions in a water matrix for 30 days, if protected from the light. Higher concentrations of silver require additional HCl.

7.5 Blanks

Two types of blanks are required for the analysis of samples prepared by any method other than Method 3040. The calibration blank is used in establishing the analytical curve and the method blank is used to identify possible contamination resulting from either the reagents (acids) or the equipment used during sample processing including filtration.

7.5.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations.

7.5.2 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis (refer to Sec. 9.3).

7.6 The initial calibration verification (ICV) standard is prepared by the analyst (or a purchased second source reference material) by combining compatible elements from a standard source different from that of the calibration standard, and at concentration near the midpoint of the calibration curve (see Sec. 10.4.1 for use). This standard may also be purchased.

7.7 The continuing calibration verification (CCV) standard should be prepared in the same acid matrix using the same standards used for calibration, at a concentration near the mid-point of the calibration curve (see Sec. 10.4.4 for use).

7.8 The interference check solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest, particularly those with known interferences at 0.5 to 1 mg/L. In the absence of measurable analyte, overcorrection could go undetected because a negative value

could be reported as zero. If the particular instrument will display overcorrection as a negative number, this spiking procedure will not be necessary.

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See Chapter Three, Inorganic Analytes, for sample collection and preservation instructions.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for additional guidance on quality assurance protocols. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Dilute and reanalyze samples that exceed the linear calibration range or use an alternate, less sensitive line for which quality control data are already established.

9.3 For each batch of samples processed, at least one method blank must be carried throughout the entire sample preparation and analytical process, as described in Chapter One. A method blank is prepared by using a volume or weight of reagent water at the volume or weight specified in the preparation method, and then carried through the appropriate steps of the analytical process. These steps may include, but are not limited to, prefiltering, digestion, dilution, filtering, and analysis. If the method blank does not contain target analytes at a level that interferes with the project-specific DQOs, then the method blank would be considered acceptable.

In the absence of project-specific DQOs, if the blank is less than 5% of the MDL Check Sample, less than 5% of the regulatory limit, or less than 5% of the lowest sample concentration for each analyte, whichever is greater, then the method blank is considered acceptable. If the method blank cannot be considered acceptable, the method blank should be re-run once, and if still unacceptable, then all samples after the last acceptable method blank must be reprepared and reanalyzed along with the other appropriate batch QC samples. These blanks will be useful in determining if samples are being contaminated. If the method blank exceeds the criteria, but the samples are all either below the reporting level or below the applicable action level or other DQOs, then the sample data may be used despite the contamination of the method blank. Refer to Chapter One for the proper protocol when analyzing blanks.

9.4 Laboratory control sample (LCS)

For each batch of samples processed, at least one LCS must be carried throughout the entire sample preparation and analytical process as described in Chapter One. The laboratory control samples should be spiked with each analyte of interest at the project-specific action level or, when lacking project-specific action levels, between the low and mid-level standards. Acceptance criteria should be set at a laboratory derived limit developed through the use of historical analyses. In the absence of historical data this limit should be set at ± 20% of the spiked value. Acceptance limits derived from historical data must be no wider that ± 20%. If the laboratory control sample is not acceptable, then the laboratory control sample should be re-run once and, if still unacceptable, all samples after the last acceptable laboratory control sample must be reprepared and reanalyzed.

Concurrent analyses of reference materials (SRMs) containing known amounts of analytes in the media of interest are recommended and may be used as an LCS. For solid SRMs, 80 -120% accuracy may not be achievable and the manufacturer's established acceptance criterion should be used for soil SRMs. Refer to Chapter One for more information.

9.5 Matrix spike/matrix spike duplicates (MS/MSDs)

For each batch of samples processed, at least one MS/MSD sample must be carried throughout the entire sample preparation and analytical process as described in Chapter One. MS/MSDs are intralaboratory split samples spiked with identical concentrations of each analyte of interest. The spiking occurs prior to sample preparation and analysis. An MS/MSD is used to document the bias and precision of a method in a given sample matrix. At the laboratory's discretion, a separate spiked sample and a separate duplicate sample may be analyzed in lieu of the MS/MSD.

Refer to the definitions of bias and precision, in Chapter One, for the proper data reduction protocols. MS/MSD samples should be spiked at the same level, and with the same spiking material, as the corresponding laboratory control sample that is at the project-specific action level or, when lacking project-specific action levels, between the low and mid-level standards. Acceptance criteria should be set at a laboratory-derived limit developed through the use of historical analyses per matrix type analyzed. In the absence of historical data this limit should be set at ± 25% of the spiked value for accuracy and 20 relative percent difference (RPD) for precision. Acceptance limits derived from historical data must be no wider that ± 25% for accuracy and 20% for precision. Refer to Sec. 4.4.2 of Chapter One for guidance. If the bias and precision indicators are outside the laboratory control limits, if the percent recovery is less than 75% or greater than 125%, or if the relative percent difference is greater than 20%, then the interference test discussed in Sec. 9.6 should be conducted.

9.5.1 The relative percent difference between spiked matrix duplicate determinations is to be calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{\left(\frac{|D_1 + D_2|}{2}\right)} \times 100$$

where:

RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (duplicate).

9.5.2 The spiked sample or spiked duplicate sample recovery should be within ± 25% of the actual value, or within the documented historical acceptance limits for each matrix.

9.6 The following tests are recommended prior to reporting concentration data for the elements in this method. These tests, outlined in Secs. 9.6.1 and 9.6.2, should be performed with each batch of samples prepared/analyzed and will ensure that neither positive nor negative interferences are affecting the measurement of any of the elements or distorting the accuracy of the reported values. If matrix effects are confirmed, then an alternative test method should be

considered or the current test method modified so that the analysis is not impacted by the same interference.

9.6.1 Post digestion spike addition

The same sample from which the MS/MSD aliquots were prepared should also be spiked with a post digestion spike. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 80% to 120% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the method detection limit. If this spike fails, then the dilution test (Sec. 9.6.2) should be run on this sample. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed.

9.6.2 Dilution test

If the analyte concentration is sufficiently high (minimally, a factor of 10 above the method detection limit after dilution), an analysis of a 1:5 dilution should agree within ± 10% of the original determination. If not, then a chemical or physical interference effect should be suspected.

CAUTION: If spectral overlap is suspected, then the use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Set up the instrument with proper operating parameters established as detailed below. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration). For operating conditions, the analyst should follow the instructions provided by the instrument manufacturer.

10.1.1 Before using this procedure to analyze samples, data must be available documenting the initial demonstration of performance. The required data document the selection criteria for background correction points; analytical dynamic ranges, the applicable equations, and the upper limits of those ranges; the method and instrument detection limits; and the determination and verification of interelement correction equations or other routines for correcting spectral interferences. These data must be generated using the same instrument, operating conditions, and calibration routine to be used for sample analysis. These data must be kept on file and be available for review by the data user or auditor.

10.1.2 Specific wavelengths are listed in Table 1. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. Because of differences among various makes and models of spectrometers, specific instrument operating conditions cannot be provided. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality to the program and data user. The analyst should follow the instructions provided by the instrument manufacturer unless other conditions provide similar or better performance for a task.

Operating conditions for aqueous solutions usually vary from:

- 1100 to 1200 watts forward power,
- 14 to 18 mm viewing height,
- 15 to 19 L/min argon coolant flow,
- 0.6 to 1.5 L/min argon nebulizer flow,
- 1 to 1.8 mL/min sample pumping rate with a 1 minute preflush time and measurement time near 1 second per wavelength peak for sequential instruments and 10 seconds per sample for simultaneous instruments.

For an axial plasma, the conditions will usually vary from:

- 1100 to 1500 watts forward power,
- 15 to 19 liters/min argon coolant flow,
- 0.6 to 1.5 L/min argon nebulizer flow,
- 1 to 1.8 mL/min sample pumping rate with a 1 minute preflush time and measurement time near 1 second per wavelength peak for sequential instruments and 10 seconds per sample for simultaneous instruments.

One recommended way in which to achieve repeatable interference correction factors is to adjust the argon aerosol flow to reproduce the Cu/Mn intensity ratio at 324.754 nm and 257.610 nm respectively.

10.1.3 Plasma optimization

The plasma operating conditions need to be optimized prior to use of the instrument. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure. This routine is not required on a daily basis, but only is required when first setting up a new instrument, or following a change in operating conditions. The following procedure is recommended, or follow manufacturer's recommendations.

10.1.3.1 Ignite the radial plasma and select an appropriate incident RF power. Allow the instrument to become thermally stable before beginning, about 30 to 60 minutes of operation. While aspirating a 1000 µg/L solution of yttrium, follow the instrument manufacturer's instructions and adjust the aerosol carrier gas flow rate through the nebulizer so a definitive blue emission region of the plasma extends approximately from 5 to 20 mm above the top of the load coil. Record the nebulizer gas flow rate or pressure setting for future reference. The yttrium solution can also be used for coarse optical alignment of the torch by observing the overlay of the blue light over the entrance slit to the optical system.

10.1.3.2 After establishing the nebulizer gas flow rate, determine the solution uptake rate of the nebulizer in mL/min by aspirating a known volume of a calibration blank for a period of at least three minutes. Divide the volume aspirated by the time in minutes and record the uptake rate. Set the peristaltic pump to deliver that rate in a steady even flow.

10.1.3.3 Profile the instrument to align it optically as it will be used during analysis. The following procedure can be used for both horizontal and vertical optimization in the radial mode, but is written for vertical.

Aspirate a solution containing 10 µg/L of several selected elements. As, Se, Tl, and Pb are the least sensitive of the elements and most in need of optimization. However, other elements may be used, based no the judgement of the analyst. (V, Cr, Cu, Li and Mn also have been used with success). Collect intensity data at the wavelength peak for each analyte at 1 mm intervals from 14 to 18 mm above the load coil. (This region of the plasma is referred to as the analytical zone.) Repeat the process using the calibration blank. Determine the net signal to blank intensity ratio for each analyte for each viewing height setting. Choose the height for viewing the plasma that provides the best net intensity ratios for the elements analyzed or the highest intensity ratio for the least sensitive element. For optimization in the axial mode, follow the instrument manufacturer's instructions.

10.1.3.4 The instrument operating conditions finally selected as being optimum should provide the lowest reliable instrument detection limits.

10.1.3.5 If the instrument operating conditions, such as incident power or nebulizer gas flow rate, are changed, or if a new torch injector tube with a different orifice internal diameter is installed, then the plasma and viewing height should be re-optimized.

10.1.3.6 After completing the initial optimization of operating conditions, and before analyzing samples, the laboratory must establish and initially verify an interelement spectral interference correction routine to be used during sample analysis. A general description of spectral interferences and the analytical requirements for background correction, in particular, are discussed in Sec. 4.0. The criterion for determining that an interelement spectral interference is present is an apparent positive or negative concentration for the analyte that falls beyond ± one reporting limit from zero. The upper control limit is the analyte instrument detection limit. Once established, the entire routine must be verified every six months. Only a portion of the correction routine must be verified more frequently or on a daily basis. Initial and periodic verifications of the routine should be kept on file.

10.1.3.7 Before daily calibration, and after the instrument warmup period, the nebulizer gas flow rate must be reset to the determined optimized flow. If a mass flow controller is being used, it should be set to the recorded optimized flow rate. In order to maintain valid spectral interelement correction routines, the nebulizer gas flow rate should be the same (< 2% change) from day to day.

10.2 For operation with organic solvents, the use of the auxiliary argon inlet is recommended, as is the use of solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power, to obtain stable operation and precise measurements.

10.3 Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

10.3.1 Method detection limits must be established for all wavelengths utilized for each type of matrix analyzed and for each preparation method used and for each instrument. The matrix used for the MDL calculation must contain analytes of known concentrations within 3-5 times the anticipated detection limit. Refer to Chapter One for additional guidance on the performance of MDL studies.

10.3.2 Determination of detection limits using reagent water represents a best case situation and does not represent possible matrix effects of real-world samples. For application of MDLs on a project-specific basis with established DQOs, matrix-specific MDL studies may provide data users with a more reliable estimate of method detection capabilities.

10.3.3 MDL check sample

The MDL check sample must be analyzed after the completion of the MDL study and on a quarterly basis to demonstrate detection capability. The MDL check sample is spiked into reagent water at 2-3 times the detection limit and is carried throughout the entire analytical procedure. Detection limits are verified when all analytes in the MDL check sample are detected. This is a qualitative check and also establishes the lowest reporting limit.

10.3.4 The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses from a minimum of three, preferably five, different concentration standards across the range. The ranges which may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made should be documented and kept on file. A standard at the upper limit must be prepared, analyzed and quantitated against the normal calibration curve. The calculated value must be within 10% (±10%) of the true value. New upper range limits should be determined whenever there is a significant change in instrument response. At a minimum, the range should be checked every six months. The analyst should be aware that if an analyte that is present above its upper range limit is used to apply an interelement correction, the correction may not be valid and those analytes where the interelement correction has been applied may be inaccurately reported.

NOTE: Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self-absorption effects. These curves may be used if the instrument allows it; however the effective range must be checked and the second order curve fit should have a correlation coefficient of 0.995 or better. Third order fits are not acceptable. These non-linear response curves should be revalidated and recalculated every six months. These curves are much more sensitive to changes in operating conditions than the linear lines and should be checked whenever there have been moderate equipment changes.

10.3.5 The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

10.4 All analyses require that a calibration curve be prepared to cover the appropriate concentration range. Usually, this means the preparation of a calibration blank and standards, the highest of which would not exceed the linear dynamic range of the instrument as previously established. Check the instrument standardization by analyzing appropriate QC samples as follows.

10.4.1 Calibration standards should be prepared fresh each time a batch of samples is analyzed. If the ICV solution is prepared daily and the results of the ICV analyses are within the acceptance criteria, then the calibration standards do not need to be prepared daily and may be prepared and stored for as long as the calibration standard viability can be verified through the use of the ICV. If the ICV is outside of the acceptance criteria, then the calibration standards must be prepared fresh and the instrument recalibrated.

10.4.1.1 The calibration standards should be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following processing.

10.4.1.2 The absolute value of the results of the calibration blank should be less than the value of the MDL check sample for each analyte or less than the level of acceptable blank contamination specified in the approved quality assurance project plan. If this is not the case, the reason for the out-of-control condition must be found and corrected, and the previous ten samples reanalyzed.

10.4.2 A calibration curve must be prepared daily with a minimum of a calibration blank and three standards. The curve must have a correlation coefficient of 0.995. Alternatively, the initial calibration curve may be prepared daily with a minimum of a calibration blank and a single high standard. The resulting curve must then be verified with mid-level and low level calibration verification standards. An acceptance range of 80 - 120% will be used for verification of both standards. In either case, sample values that are measured above the high standard must be diluted in the calibration range and reanalyzed. The laboratory's quantitation limit cannot be reported lower than either the low standard used during initial calibration or the low-level calibration verification standard.

10.4.3 After initial calibration, the calibration curve must be verified by use of an initial calibration verification (ICV) standard. The ICV standard must be prepared from an independent (second source) material at or near the mid-range of the calibration curve. The acceptance criteria for the ICV standard must be ±10% of its true value. If the calibration curve cannot be verified within the specified limits, the cause must be determined and the instrument recalibrated before samples are analyzed. The analysis data for the ICV must be kept on file with the sample analysis data.

10.4.4 The calibration curve must be verified at the end of each analysis batch and after every 10 samples by use of a continuing calibration verification (CCV) standard and a continuing calibration blank (CCB). The CCV should be made from the same material as the initial calibration standards at or near mid-range. The acceptance criteria for the CCV standard must be ±10% of its true value and the CCB must not contain target analytes above 2 - 3 times the MDL for the curve to be considered valid. If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable CCV/CCB must be reanalyzed. The analysis data for the CCV/CCB must be kept on file with the sample analysis data.

10.4.5 If a single calibration standard and blank are used to establish the initial calibration curve, then the calibration curve must also be verified prior to the analysis of any samples by use of a low-level continuing calibration verification (LLCCV) standard. The LLCCV standard should be made from the same material as the initial calibration standards at the quantitation limit as reported by the laboratory. The acceptance criteria for the LLCCV

standard must be ±20% of its true value. If the calibration cannot be verified within the specified limits, the sample analysis cannot begin until the cause is determined and the LLCCV standard successfully analyzed. The instrument may need to be recalibrated or the quantitation limit adjusted. The analysis data for the LLCCV standard must be kept on file with the sample analysis data.

11.0 PROCEDURE

11.1 Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been prefiltered and acidified will not need acid digestion. However, all associated QC samples (i.e., method blank, LCS and MS/MSD) must undergo the same filtration and acidification procedures. Samples which are not digested must either use an internal standard or be matrix-matched with the standards. Solubilization and digestion procedures are presented in Chapter Three, Inorganic Analytes.

11.2 Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Sec. 7.4. Flush the system with the calibration blank (Sec. 7.5.1) between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve must be prepared as detailed in Sec. 10.4.2.

11.3 When the initial calibration is performed using a single high standard and the calibration blank, the laboratory must analyze an LLCCV (Sec. 10.4.5). For all analytes and determinations, the laboratory must analyze an ICV (Sec. 7.6) immediately following daily calibration. A CCV (Secs. 7.7 and 10.4.4) and a CCB (Secs. 7.5.1 and 10.4.4) must be analyzed after every ten samples and at the end of the analysis batch.

11.4 Rinse the system with the calibration blank solution (Sec. 7.5.1) before the analysis of each sample. The rinse time will be one minute. Each laboratory may establish a reduction in this rinse time through a suitable demonstration. Analyze the samples and record the results.

12.0 DATA ANALYSIS AND CALCULATIONS

If dilutions were performed, the appropriate factors must be applied to sample values. All results should be reported with up to three significant figures.

13.0 METHOD PERFORMANCE

13.1 In an EPA round-robin study, seven laboratories applied the ICP technique to acid-digested water matrices that had been spiked with various metal concentrates. Table 4 lists the true values, the mean reported values, and the mean percent relative standard deviations.

13.2 Performance data for aqueous solutions and solid samples from a multilaboratory study are provided in Tables 5 and 6.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St. NW, Washington, D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. C.L. Jones, *et al*., "An Interlaboratory Study of Inductively Coupled Plasma Atomic Emission Spectroscopy Method 6010 and Digestion Method 3050," EPA-600/4-87-032, U.S. Environmental Protection Agency, Las Vegas, NV, 1987.

17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

The pages to follow contain Tables 1 through 6 and a flow diagram of the method.

TABLE 1

RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Element	Wavelength[a] (nm)	Estimated IDL[b] (µg/L)
Aluminum	308.215	30
Antimony	206.833	21
Arsenic	193.696	35
Barium	455.403	0.87
Beryllium	313.042	0.18
Boron	249.678 x2	3.8
Cadmium	226.502	2.3
Calcium	317.933	6.7
Chromium	267.716	4.7
Cobalt	228.616	4.7
Copper	324.754	3.6
Iron	259.940	4.1
Lead	220.353	28
Lithium	670.784	2.8
Magnesium	279.079	20
Manganese	257.610	0.93
Mercury	194.227 x2	17
Molybdenum	202.030	5.3
Nickel	231.604 x2	10
Phosphorus	213.618	51
Potassium	766.491	See note c
Selenium	196.026	50
Silica (SiO_2)	251.611	17
Silver	328.068	4.7
Sodium	588.995	19
Strontium	407.771	0.28
Thallium	190.864	27
Tin	189.980 x2	17
Titanium	334.941	5.0
Vanadium	292.402	5.0
Zinc	213.856 x2	1.2

TABLE 1
(continued)

[a] The wavelengths listed (where x2 indicates second order) are recommended because of their sensitivity. Other wavelengths may be substituted (e.g., in the case of an interference) if they provide the needed sensitivity and are treated with the same corrective techniques for spectral interference.

[b] The estimated instrumental detection limits shown are provided for illustrative purposes only. Each laboratory must determine IDLs and MDLs, as necessary, for their specific application of the method. These IDLs represent radial plasma data and axial plasma IDLs may be lower.

[c] Highly dependent on operating conditions and plasma position.

TABLE 2

POTENTIAL INTERFERENCES AND ANALYTE CONCENTRATION EQUIVALENTS (mg/L) ARISING FROM INTERFERENCE AT THE 100-mg/L LEVEL

Analyte	Wavelenth (nm)	Interferant[a,b]									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

[a] Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al at 1000 mg/L	Cu at 200 mg/L	Mn at 200 mg/L
Ca at 1000 mg/L	Fe at 1000 mg/L	Ti at 200 mg/L
Cr at 200 mg/L	Mg at 1000 mg/L	V at 200 mg/L

[b] The figures shown above as analyte concentration equivalents are not the actual observed concentrations. To obtain those figures, add the listed concentration to the interferant figure.

[c] Interferences will be affected by background choice and other interferences may be present.

TABLE 3

MIXED STANDARD SOLUTIONS

Solution	Elements
I	Be, Cd, Mn, Pb, Se and Zn
II	Ba, Co, Cu, Fe, and V
III	As and Mo
IV	Al, Ca, Cr, K, Na, Ni, Li, and Sr
V	Ag[a], Mg, Sb, and Tl
VI	P

[a] See the note in Sec. 7.4.

TABLE 4

ICP PRECISION AND ACCURACY DATA[a]

Element	Sample No. 1				Sample No. 2				Sample No. 3			
	True Conc. (µg/L)	Mean Conc. (µg/L)	RSD[b] (%)	Accuracy[d] (%)	True Conc. (µg/L)	Mean Conc. (µg/L)	RSD[b] (%)	Accuracy[d] (%)	True Conc. (µg/L)	Mean Conc. (µg/L)	RSD[b] (%)	Accuracy[d] (%)
Be	750	733	6.2	98	20	20	9.8	100	180	176	5.2	98
Mn	350	345	2.7	99	15	15	6.7	100	100	99	3.3	99
V	750	749	1.8	100	70	69	2.9	99	170	169	1.1	99
As	200	208	7.5	104	22	19	23	86	60	63	17	105
Cr	150	149	3.8	99	10	10	18	100	50	50	3.3	100
Cu	250	235	5.1	94	11	11	40	100	70	67	7.9	96
Fe	600	594	3.0	99	20	19	15	95	180	178	6.0	99
Al	700	696	5.6	99	60	62	33	103	160	161	13	101
Cd	50	48	12	96	2.5	2.9	16	116	14	13	16	93
Co	700	512	10	73	20	20	4.1	100	120	108	21	90
Ni	250	245	5.8	98	30	28	11	93	60	55	14	92
Pb	250	236	16	94	24	30	32	125	80	80	14	100
Zn	200	201	5.6	100	16	19	45	119	80	82	9.4	102
Se[c]	40	32	21.9	80	6	8.5	42	142	10	8.5	8.3	85

[a] Not all elements were analyzed by all laboratories.
[b] RSD = relative standard deviation.
[c] Results for Se are from two laboratories.
[d] Accuracy is expressed as the mean concentration divided by the true concentration times 100.

TABLE 5

EXAMPLE ICP-AES PRECISION AND ACCURACY FOR AQUEOUS SOLUTIONS

Element	Mean Conc. (mg/L)	n	RSD (%)	Accuracy (%)
Al	14.8	8	6.3	100
Sb	15.1	8	7.7	102
As	14.7	7	6.4	99
Ba	3.66	7	3.1	99
Be	3.78	8	5.8	102
Cd	3.61	8	7.0	97
Ca	15.0	8	7.4	101
Cr	3.75	8	8.2	101
Co	3.52	8	5.9	95
Cu	3.58	8	5.6	97
Fe	14.8	8	5.9	100
Pb	14.4	7	5.9	97
Mg	14.1	8	6.5	96
Mn	3.70	8	4.3	100
Mo	3.70	8	6.9	100
Ni	3.70	7	5.7	100
K	14.1	8	6.6	95
Se	15.3	8	7.5	104
Ag	3.69	6	9.1	100
Na	14.0	8	4.2	95
Tl	15.1	7	8.5	102
V	3.51	8	6.6	95
Zn	3.57	8	8.3	96

These performance values are independent of sample preparation because the labs analyzed portions of the same solutions and are provided for illustrative purposes only.

n= Number of measurements.

Accuracy is expressed as a percentage of the nominal value for each analyte in acidified, multi-element solutions.

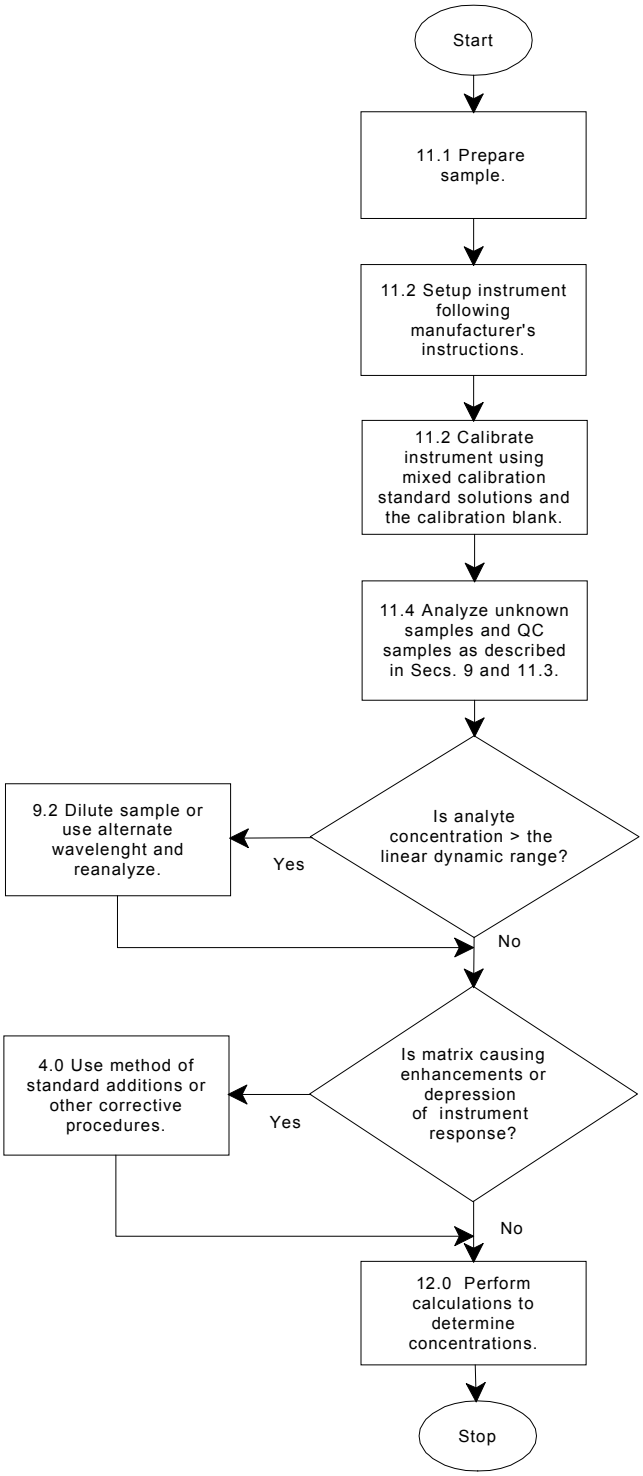
TABLE 6

EXAMPLE ICP-AES PRECISION AND BIAS FOR SOLID WASTE DIGESTS

	Spiked Coal Fly Ash (NIST-SRM 1633a)				Spiked Electroplating Sludge			
Element	Mean Conc. (mg/L)	n	RSD (%)	Bias (% AA)	Mean Conc. (mg/L)	n	RSD (%)	Bias (% AA)
Al	330	8	16	104	127	8	13	110
Sb	3.4	6	73	96	5.3	7	24	120
As	21	8	83	270	5.2	7	8.6	87
Ba	133	8	8.7	101	1.6	8	20	58
Be	4.0	8	57	460	0.9	7	9.9	110
Cd	0.97	6	5.7	101	2.9	7	9.9	90
Ca	87	6	5.6	208	954	7	7.0	97
Cr	2.1	7	36	106	154	7	7.8	93
Co	1.2	6	21	94	1.0	7	11	85
Cu	1.9	6	9.7	118	156	8	7.8	97
Fe	602	8	8.8	102	603	7	5.6	98
Pb	4.6	7	22	94	25	7	5.6	98
Mg	15	8	15	110	35	8	20	84
Mn	1.8	7	14	104	5.9	7	9.6	95
Mo	891	8	19	105	1.4	7	36	110
Ni	1.6	6	8.1	91	9.5	7	9.6	90
K	46	8	4.2	98	51	8	5.8	82
Se	6.4	5	16	73	8.7	7	13	101
Ag	1.4	3	17	140	0.75	7	19	270
Na	20	8	49	130	1380	8	9.8	95
Tl	6.7	4	22	260	5.0	7	20	180
V	1010	5	7.5	100	1.2	6	11	80
Zn	2.2	6	7.6	93	266	7	2.5	101

These performance values are independent of sample preparation because the labs analyzed portions of the same digests and are provided for illustrative purposes only.
n = Number of measurements.
Bias for the ICP-AES data is expressed as a percentage of atomic absorption spectroscopy (AA) data for the same digests.

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY



Appendix D
EPA Test Method 300

METHOD 300.1

DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY

Revision 1.0

John D. Pfaff (USEPA, ORD, NERL) - Method 300.0, (1993)

Daniel P. Hautman (USEPA, Office of Water) and David J. Munch (USEPA, Office of Water) - Method 300.1, (1997)

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ERRATA COVER SHEET TO U.S.EPA METHOD 300.1
April 27, 1999

The following were editorial changes which have been incorporated into U.S.EPA Method 300.1. These minor clarifications are incorporated into the body of this text as follows:

ERRATA #1 -
An additional sentence was added to Section 4.1.1 reiterating the analyst's responsibilities when incorporating any method change, including modifying eluent strength, or any other method parameter. The additional sentence states,

> "*...The analyst must verify that these changes do not negatively affect performance by repeating and passing all the QC criteria in Section 9.*"

On this same theme, section 11.9, was also further clarified and specific precautions were added as follows,

> "*...The analysts must verify that this dilution does not negatively affect performance by repeating and passing all the QC criteria in Section 9. As a specific precaution, upon dilution of the carbonate eluent, a peak for bicarbonate may be observed within the retention time window for bromate which will negatively impact the analysis.*"

ERRATA #2 -
An acronym in Section 9.3.2.2 for Laboratory Fortified Blank (LFB) was incorrectly identified as LRB. This typographical error was corrected.

ERRATA #3 -
Clarifications and corrections were made to Section 9.4.1.5, 9.4.3.2 and 9.4.3.3. These clarifications pertain to data reportability for Laboratory Fortified Sample Matrices (LFM) as well as to analysis continuation when Duplicate Sample QC acceptance criteria are not met.

Section 9.4.1.5 clarifies and now specifies how to report data when the LFM recovery falls outside the established control criteria by stating,

> "*...the recovery problem encountered with the LFM is judged to be matrix induced and the results for that sample and the LFM are reported with a "matrix induced bias" qualifier.*"

Section 9.4.3.2 required the correction of a typographical reference by removing "%Diff" in the duplicate sample acceptance criteria and replacing it with the defined RPD, indicating "relative percent difference".

Section 9.4.3.3, also had a "%Diff" reference corrected with RPD and included clarification regarding continuation of an analysis set when a duplicate analysis fails to meet the acceptance criteria. This section now reads,

> "*If the RPD fails to meet these criteria, the samples must be reported with a qualifier identifying the sample analysis result as yielding a poor duplicate analysis RPD. This should not be a chronic problem and if it frequently recurs, (>20% of duplicate analysis) it indicates a problem with the instrument or individual technique.*"

METHOD 300.1

DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY

1. SCOPE AND APPLICATION

1.1 This method covers the determination of the following inorganic anions in reagent water, surface water, ground water, and finished drinking water. As a result of different specified injection volumes (See conditions in Tables 1A and 1B), these anions are divided between the common anions listed in Part A and the inorganic disinfection by-products listed in Part B. These different injection volumes are required in order to compensate for the relative concentrations of these anions in drinking water and maintain good chromatographic peak shape throughout the expected dynamic range of the detector. Bromide is included in both Part A, due to its importance as a common anion, as well as Part B due to its critical role as a disinfection by-product precursor.

PART A.-- Common Anions

Bromide	Nitrite
Chloride	ortho-Phosphate-P
Fluoride	Sulfate
Nitrate	

PART B.-- Inorganic Disinfection By-products

Bromate	Chlorite
Bromide	Chlorate

1.2 The single laboratory Method Detection Limits (MDL, defined in Sect. 3.11) for the above analytes are listed in Tables 1A, 1B and 1C. The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample and the specific instrumentation employed.

1.2.1 In order to achieve comparable detection limits, an ion chromatographic system must utilize suppressed conductivity detection, be properly maintained and must be capable of yielding a baseline with no more than 5 nS noise/drift per minute of monitored response over the background conductivity.

1.3 This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

1.4 When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a fortified sample matrix covering the anions of interest. The fortification procedure is described in Sect. 9.4.1.

1.5 Users of the method data should state the data-quality objectives prior to analysis. Users of the method must demonstrate the ability to generate acceptable results with this method, using the procedures described in Sect. 9.0.

1.6 Bromide and nitrite react with most oxidants employed as disinfectants. The utility of measuring these anions in treated water should be considered prior to conducting the analysis.

2. SUMMARY OF METHOD

2.1 A small volume of sample, 10 uL for Part A and 50 uL for Part B, is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

2.2 The ONLY difference between Parts A and B is the volume of sample analyzed by the ion chromatographic system. The separator columns and guard columns as well as eluent conditions are identical.

3. DEFINITIONS

3.1 ANALYSIS BATCH -- A group of no more than 20 field samples (Field sample analyses include only those samples derived from a field sample matrix. These include the initial and duplicate field samples as well as all Laboratory Fortified Sample Matrices). The analysis batch must include an Initial Calibration Check Standard, an End Calibration Check Standard, Laboratory Reagent Blank, and a Laboratory Fortified Blank. Within an ANALYSIS BATCH, for every group of ten field samples, at least one Laboratory Fortified Matrix (LFM) and either a Field Duplicate, a Laboratory Duplicate or a duplicate of the LFM must be analyzed. When more than 10 field samples are analyzed, a Continuing Calibration Check Standard must be analyzed after the tenth field sample analysis.

3.2 CALIBRATION STANDARD (CAL) -- A solution prepared from the primary dilution standard solution or stock standard solutions and the surrogate analyte. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.2.1 INITIAL CALIBRATION STANDARDS -- A series of CAL solutions used to initially establish instrument calibration and develop calibration curves for individual target anions.

3.2.2 INITIAL CALIBRATION CHECK STANDARD -- An individual CAL solution, analyzed initially, prior to any sample analysis, which verifies previously established calibration curves.

3.2.3 CONTINUING CALIBRATION CHECK STANDARD -- An individual CAL solution which is analyzed after every tenth field sample analyses

which verifies the previously established calibration curves and confirms accurate analyte quantitation for the previous ten field samples analyzed.

3.2.4 END CALIBRATION CHECK STANDARD -- An individual CAL solution which is analyzed after the last field sample analyses which verifies the previously established calibration curves and confirms accurate analyte quantitation for all field samples analyzed since the last continuing calibration check.

3.3 FIELD DUPLICATES -- Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

3.4 INSTRUMENT PERFORMANCE CHECK SOLUTION (IPC) -- A solution of one or more method analytes, surrogates, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.5 LABORATORY DUPLICATE -- Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicate precision associated specifically with the laboratory procedures, removing any associated variables attributed by sample collection, preservation, or storage procedures.

3.6 LABORATORY FORTIFIED BLANK (LFB) -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.7 LABORATORY FORTIFIED SAMPLE MATRIX (LFM) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.8 LABORATORY REAGENT BLANK (LRB) -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.9 LINEAR CALIBRATION RANGE (LCR) -- The concentration range over which the instrument response is linear.

3.10 MATERIAL SAFETY DATA SHEET (MSDS) -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.11 METHOD DETECTION LIMIT (MDL) -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.12 MINIMUM REPORTING LEVEL (MRL) -- The minimum concentration that can be reported for an anion in a sample following analysis. This defined concentration can be no lower than the concentration of the lowest calibration standard and can only be used if acceptable quality control criteria for this standard are met.

3.13 PERFORMANCE EVALUATION SAMPLE (PE) -- A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst.

3.14 QUALITY CONTROL SAMPLE (QCS) -- A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

3.15 SURROGATE ANALYTE -- An analyte added to a sample, which is unlikely to be found in any sample at significant concentration, and which is added directly to a sample aliquot in known amounts before any sample processing procedures are conducted. It is measured with the same procedures used to measure other sample components. The purpose of the surrogate analyte is to monitor method performance with each sample.

3.16 STOCK STANDARD SOLUTION (SSS) -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

4. INTERFERENCES

4.1 Interferences can be divided into three different categories: direct chromatographic coelution, where an analyte response is observed at very nearly the same retention time as the target anion; concentration dependant coelution, which is observed when the response of higher than typical concentrations of the

neighboring peak overlap into the retention window of the target anion; and, ionic character displacement, where retention times may significantly shift due to the influence of high ionic strength matrices (high mineral content or hardness) overloading the exchange sites in the column and significantly shortening target analyte's retention times.

4.1.1 A direct chromatographic coelution may be solved by changing columns, eluent strength, modifying the eluent with organic solvents (if compatible with IC columns), changing the detection systems, or selective removal of the interference with pretreatment. Sample dilution will have little to no effect. The analyst must verify that these changes do not negatively affect performance by repeating and passing all the QC criteria in Section 9.

4.1.2 Sample dilution may resolve some of the difficulties if the interference is the result of either concentration dependant coelution or ionic character displacement, but it must be clarified that **sample dilution will alter your Minimum Reporting Limit (MRL)** by a proportion equivalent to that of the dilution. Therefore, careful consideration of project objectives should be given prior to performing such a dilution. An alternative to sample dilution, may be dilution of the eluent as outlined in 11.9.

4.1.3 Pretreatment cartridges can be effective as a means to eliminate certain matrix interferences. Prior to using any pretreatment, the analyst should be aware that all instrument calibration standards must be pretreated in exactly the same manner as the pretreated unknown field samples. The need for these cartridges have been greatly reduced with recent advances in high capacity anion exchange columns.

4.1.3.1 Extreme caution should be exercised in using these pretreatment cartridges. Artifacts are known to leach from certain cartridges which can foul the guard and analytical columns causing loss of column capacity indicated by shortened retention times and irreproducible results. Frequently compare your calibration standard chromatograms to those of the column test chromatogram (received when the column was purchased) to insure proper separation and similar response ratios between the target analytes is observed.

4.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in an ion chromatogram. These interferences can lead to false positive results for target analytes as well as reduced detection limits as a consequence of elevated baseline noise.

4.3 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

4.4 Any anion that is only weakly retained by the column may elute in the retention time window of fluoride and potentially interfere. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant, however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.5 Close attention should be given to the potential for carry over peaks from one analysis which will effect the proper detection of analytes of interest in a second, subsequent analysis. Normally, the elution of sulfate (retention time of 13.8 min.) indicates the end of a chromatographic run, but, in the ozonated and chlorine dioxide matrices, which were included as part of the single operator accuracy and bias study (See Table 2B), a small response (200 nS baseline rise) was observed for a very late eluting unknown peak at approximately 23 minutes. Consequently, a run time of 25 minutes is recommended to allow for the proper elution of any potentially interferant late peaks. It is the responsibility of the user to confirm that no late eluting peaks have carried over into a subsequent analysis thereby compromising the integrity of the analytical results.

4.6 Any residual chlorine dioxide present in the sample will result in the formation of additional chlorite prior to analysis. If any concentration of chlorine dioxide is suspected in the sample, the sample must be purged with an inert gas (helium, argon or nitrogen) for approximately five minutes or until no chlorine dioxide remains. This sparging must be conducted prior to ethylenediamine preservation and at time of sample collection.

5. SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

5.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS.

5.3.1 Sulfuric acid -- When used to prepared a 25 mN sulfuric acid regenerant solution for chemical suppression using a Dionex Anion Micro Membrane Suppressor (AMMS).

6. EQUIPMENT AND SUPPLIES

6.1 Ion chromatograph -- Analytical system complete with ion chromatograph and all required accessories including syringes, analytical columns, compressed gasses and a conductivity detector.

6.1.1 Anion guard column: Dionex AG9-HC, 2 mm (P/N 52248), or equivalent. This column functions as a protector of the separator column. If omitted from the system the retention times will be shorter.

6.1.2 Anion separator column: Dionex AS9-HC column, 2 mm (P/N 52244), or equivalent. The microbore (2 mm) was selected in the development of this method as a means to tighten the bromate elution band and thus reduce the detection limit. An optional column (2 mm or 4 mm) may be used if comparable resolution of peaks is obtained, and the requirements of Sect. 9.0 can be met. The AS9-HC, 2 mm column using the conditions outlined in Table 1A and 1B produced the separation shown in Figures 1 through 4.

6.1.2.1 If a 4 mm column is employed, the injection volume should be raised by a factor of four to 40 uL for Part A anions and 200 uL for Part B anions in order to attain comparable detection limits. A four fold increase in injection volume compensates for the four fold increase in cross sectional surface area of the 4 mm standard bore column over the 2 mm microbore column.

6.1.2.2 Comparable results can be attained using the Dionex, AS9-HC, 4 mm column. MDLs for the part B, inorganic disinfection by-products using this 4 mm column are displayed along with analysis conditions in Table 1C.

6.1.3 Anion suppressor device: The data presented in this method were generated using a Dionex Anion Self Regenerating Suppressor (ASRS, P/N 43187). An equivalent suppressor device may be utilized provided comparable detection limits are achieved and adequate baseline stability is attained as measured by a combined baseline drift/noise of no more than 5 nS per minute over the background conductivity.

6.1.3.1 The ASRS was set to perform electrolytic suppression at a current setting of 100 mA using an external source DI water mode. Insufficient baseline stability was observed using the ASRS in recycle mode.

6.1.4 Detector -- Conductivity cell (Dionex CD20, or equivalent) capable of providing data as required in Sect. 9.2.

6.2 The Dionex Peaknet Data Chromatography Software was used to generate all the data in the attached tables. Systems using a strip chart recorder and integrator or other computer based data system may achieve approximately the same MDL's but the user should demonstrate this by the procedure outlined in Sect. 9.2.

6.3 Analytical balance, ±0.1 mg sensitivity. Used to accurately weigh target analyte salts for stock standard preparation.

6.4 Top loading balance, ±10 mg sensitivity. Used to accurately weigh reagents to prepare eluents.

6.5 Weigh boats, plastic, disposable - for weighing eluent reagents.

6.6 Syringes, plastic, disposable, 10 mL - used during sample preparation.

6.7 Pipets, Pasteur, plastic or glass, disposable, graduated, 5 mL and 10 mL.

6.8 Bottles, high density polyethylene (HDPE), opaque or glass, amber, 30 mL, 125 mL, 250 mL. For sampling and storage of calibration solutions. Opaque or amber due to the photoreactivity of chlorite anion.

6.9 Micro beakers, plastic, disposable - used during sample preparation.

7. REAGENTS AND STANDARDS

7.1 Reagent water: Distilled or deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

7.2 Eluent solution : Sodium carbonate (CASRN 497-19-8) 9.0 mM. Dissolve 1.91 g sodium carbonate (Na_2CO_3) in reagent water and dilute to 2 L.

7.2.1 This eluent solution must be purged for 10 minutes with helium prior to use to remove dissolved gases which may form micro bubbles in the IC compromising system performance and adversely effecting the integrity of the data.

7.3 Stock standard solutions, 1000 mg/L (1 mg/mL): Stock standard solutions may be purchased as certified solutions or prepared from ACS reagent grade, potassium or sodium salts as listed below, for most analytes. Chlorite requires careful consideration as outline below in 7.3.5.1.

7.3.1 Bromide (Br^-) 1000 mg/L: Dissolve 0.1288 g sodium bromide (NaBr, CASRN 7647-15-6) in reagent water and dilute to 100 mL in a volumetric flask.

7.3.2 Bromate (BrO_3^-) 1000 mg/L: Dissolve 0.1180 g of sodium bromate ($NaBrO_3$, CASRN 7789-38-0) in reagent water and dilute to 100 mL in a volumetric flask.

7.3.3 Chlorate ($C10_3^-$) 1000 mg/L: Dissolve 0.1275 g of sodium chlorate ($NaC10_3$, CASRN 7775-09-9) in reagent water and dilute to 100 mL in a volumetric flask.

7.3.4 Chloride (Cl^-) 1000 mg/L: Dissolve 0.1649 g sodium chloride (NaCl, CASRN 7647-14-5) in reagent water and dilute to 100 mL in a volumetric flask.

7.3.5 Chlorite ($C10_2^-$) 1000 mg/L: Assuming an exact 80.0 % $NaC10_2$ is amperometrically titrated from technical grade $NaC10_2$ (See Sect. 7.3.5.1). Dissolve 0.1676 g of sodium chlorite ($NaC10_2$, CASRN 7758-19-2) in reagent water and dilute to 100 mL in a volumetric flask.

7.3.5.1 High purity sodium chlorite ($NaClO_2$) is not currently commercially available due to potential explosive instability. Recrystallization of the technical grade (approx. 80%) can be performed but it is labor intensive and time consuming. The simplest approach is to determine the exact % $NaClO_2$ using the iodometric titration procedure (Standard Methods, 19th Ed., 4500-ClO_2.C). Following titration, an individual component standard of chlorite must be analyzed to determine if there is any significant contamination (greater than 1% of the chlorite weight) in the technical grade chlorite standard from any of the Part B components. These contaminants will place a high bias on the calibration of the other anions if all four Part B components are mixed in an combined calibration solution. If these other anions are present as contaminants, a separate chlorite calibration needs to be performed.

7.3.6 Fluoride (F^-) 1000 mg/L: Dissolve 0.2210 g sodium fluoride (NaF, CASRN 7681-49-4) in reagent water and dilute to 100 mL in a volumetric flask.

7.3.7 Nitrate (NO^-_3-N) 1000 mg/L: Dissolve 0.6068 g sodium nitrate ($NaNO_3$, CASRN 7631-99-4) in reagent water and dilute to 100 mL in a volumetric flask.

7.3.8 Nitrite (NO^-_2-N) 1000 mg/L: Dissolve 0.4926 g sodium nitrite ($NaNO_2$, CASRN 7632-00-0) in reagent water and dilute to 100 mL in a volumetric flask.

7.3.9 Phosphate (PO_4^{3-}-P) 1000 mg/L: Dissolve 0.4394 g potassium dihydrogenphosphate (KH_2PO_4, CASRN 7778-77-0) in reagent water and dilute to 100 mL in a volumetric flask.

7.3.10 Sulfate (SO_4^{2-}) 1000 mg/L: Dissolve 0.1814 g potassium sulfate (K_2SO_4, CASRN 7778-80-5) in reagent water and dilute to 100 mL in a volumetric flask.

NOTE: Stability of standards: Stock standards (7.3) for most anions are stable for at least 6 months when stored at 4ºC. Except for the chlorite standard which is only stable for two weeks when stored protected from light at 4ºC, and nitrite and phosphate which are only stable for 1 month when stored at 4ºC. Dilute working standards should be prepared monthly, except those that contain chlorite, or nitrite and phosphate which should be prepared fresh daily.

7.4 Ethylenediamine (EDA) preservation solution, 100 mg/mL: Dilute 2.8 mL of ethylenediamine (99%) (CASRN 107-15-3) to 25 mL with reagent water. Prepare fresh monthly.

7.5 Surrogate Solution: 0.50 mg/mL dichloroacetate (DCA) prepared by dissolving 0.065 g dichloroacetic acid, potassium salt (Cl_2CHCO_2K, CASRN 19559-59-2) in reagent water and dilute to 100 mL in a volumetric flask.

7.5.1 Dichloroacetate is potentially present in treated drinking waters as the acetate of the organic disinfection by product, dichloroacetic acid (DCAA). Typical concentrations of DCAA rarely exceed 50 ug/L, which, for this worst case example, would represent only a five percent increase in the observed response over the fortified concentration of 1.00 mg/L. Consequently, the criteria for acceptable recovery (90% to 115%) for the surrogate is weighted to 115% to allow for this potential background.

7.5.2 Prepare this solution fresh every 3 months or sooner if signs of degradation are present.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

8.2 Special sampling requirements and precautions for chlorite.

8.2.1 Sample bottles used for chlorite analysis must be opaque to protect the sample from light.

8.2.2 When preparing the LFM, be aware that chlorite is an oxidant and may react with the natural organic matter in an untreated drinking water matrix as a result of oxidative demand. If untreated water is collected for chlorite analysis, and subsequently used for the LFM, EDA preservation will not control this demand and reduced chlorite recoveries may be observed.

8.3 Sample preservation and holding times for the anions that can be determined by this method are as follows:

PART A : Common Anions

Analyte	Preservation	Holding Time
Bromide	None required	28 days
Chloride	None required	28 days
Fluoride	None required	28 days
Nitrate-N	Cool to 4°C	48 hours
Nitrite-N	Cool to 4°C	48 hours
ortho-Phosphate-P	Cool to 4°C	48 hours
Sulfate	Cool to 4°C	28 days

PART B : Inorganic Disinfection By-products

Analyte	Preservation	Holding Time
Bromate	50 mg/L EDA	28 days
Bromide	None required	28 days
Chlorate	50 mg/L EDA	28 days
Chlorite	50 mg/L EDA, Cool to 4°C	14 days

8.4 When collecting a sample from a treatment plant employing chlorine dioxide, the sample must be sparged with an inert gas (helium, argon, nitrogen) prior to addition of the EDA preservative at time of sample collection.

8.5 All four anions, in Part B, can be analyzed in a sample matrix which has been preserved with EDA. Add a sufficient volume of the EDA preservation solution (Sect. 7.4) such that the final concentration is 50 mg/L in the sample. This would be equivalent to adding 0.5 mL of the EDA preservation solution to 1 L of sample.

8.6 EDA is primarily used as a preservative for chlorite. Chlorite is susceptible to degradation both through catalytic reactions with dissolved iron salts and reactivity towards free chlorine which exists as hypochlorous acid/hypochlorite ion in most drinking water as a residual disinfectant. EDA serves a dual purpose as a preservative for chlorite by chelating iron as well as any other catalytically destructive metal cations and removing hypochlorous acid/hypochlorite ion by forming an organochloroamine. EDA preservation of chlorite also preserves the integrity of chlorate which can increase in unpreserved samples as a result of chlorite degradation. EDA also preserves the integrity of bromate concentrations by binding with hypobromous acid/hypobromite which is an intermediate formed

as by-product of the reaction of either ozone or hypochlorous acid/hypochlorite with bromide ion. If hypobromous acid/hypobromite is not removed from the matrix further reactions may form bromate ion.

8.7 Degradation of ortho-phosphate has been observed in samples held at room temperature for over 16 hrs (see table 3A). Therefore, samples to be analyzed for ortho-phosphate must not be held at room temperature for more than 12 cumulative hours.

9. QUALITY CONTROL

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The requirements of this program consist of an initial demonstration of laboratory performance, and subsequent analysis in each analysis batch (Sect. 3.1) of a Laboratory Reagent Blank, Laboratory Fortified Blank, Instrument Performance Check Standard, calibration check standards, Laboratory Fortified Sample Matrices (LFM) and either Field, Laboratory or LFM duplicate sample analyses. This section details the specific requirements for each of these QC parameters. The laboratory is required to maintain performance records that define the quality of the data that are generated.

9.2 INITIAL DEMONSTRATION OF PERFORMANCE

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of accuracy through the analysis of the QCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method.

9.2.2 Quality Control Sample (QCS) -- When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within ± 15% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

9.2.3 Method Detection Limit (MDL) -- MDLs must be established for all analytes, using reagent water (blank) fortified at a concentration of three to five times the estimated instrument detection limit.[6] To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method over at least three separate days. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = (t) \times (S)$$

where, t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates].
S = standard deviation of the replicate analyses.

9.2.3.1 MDLs should be determined every 6 months, when a new operator begins work or whenever there is a significant change in the background, or instrument response.

9.3 ASSESSING LABORATORY PERFORMANCE

9.3.1 Laboratory Reagent Blank (LRB) -- The laboratory must analyze at least one LRB with each analysis batch (defined Sect 3.1). Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

9.3.1.1 If conducting analysis for the Part B anions, EDA must be added to the LRB at 50 mg/L. By including EDA in the LRB, any bias as a consequence of the EDA which may be observed in the field samples, particularly in terms of background contamination, will be identified.

9.3.2 Laboratory Fortified Blank (LFB) -- The LFB should be prepared at concentrations similar to those expected in the field samples and ideally at the same concentration used to prepare the LFM. Calculate accuracy as percent recovery (Sect. 9.4.1.3). If the recovery of any analyte falls outside the required concentration dependant control limits (Sect. 9.3.2.2), that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

9.3.2.1 If conducting analysis for the Part B anions, EDA must be added to the LFB at 50 mg/L. The addition of EDA to all reagent water prepared calibration and quality control samples is required not as a preservative but rather as a means to normalize any bias attributed by the presence of EDA in the field samples.

9.3.2.2 Control Limits for the LFB

Concentration range	Percent Recovery Limits
MRL to 10xMRL	75 - 125 %
10xMRL to highest calibration level	85 - 115 %

9.3.2.2.1 These control limits only apply if the MRL is established within a factor of 10 times the MDL. Otherwise, the limits are set at 85% to 115%.

9.3.2.3 The laboratory must use the LFB to assess laboratory performance against the required control limits listed in 9.3.2.2. When sufficient internal performance data become available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

UPPER CONTROL LIMIT = x + 3S
LOWER CONTROL LIMIT = x - 3S

The optional control limits must be equal to or better than those listed in 9.3.2.2. After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of concentrations monitored. These data must be kept on file and be available for review.

9.3.3 Instrument Performance Check Solution (IPC) -- The Initial Calibration Check Standard is to be evaluated as the instrument performance check solution in order to confirm proper instrument performance. Proper chromatographic performance must be demonstrated by calculating the Peak Gaussian Factor (PGF), which is a means to measure peak symmetry and monitoring retention time drift in the surrogate peak over time. Critically evaluate the surrogate peak in the initial calibration check standard, and calculate the PGF as follows,

$$PGF = \frac{1.83 \times W(1/2)}{W(1/10)}$$

where: W(1/2) is the peak width at half height
W(1/10) is the peak width at tenth height

9.3.3.1 The PGF must fall between 0.80 and 1.15 in order to demonstrate proper instrument performance.

9.3.3.2 The retention time for the surrogate in the IPC must be closely monitored on each day of analysis and throughout the lifetime of the analytical column. Small variations in retention time can be anticipated when a new solution of eluent is prepared but if shifts of more than 2% are observed in the surrogate retention time, some type of instrument problem is present. Potential

problems include improperly prepared eluent, erroneous method parameters programmed such as flow rate or some other system problem. The chromatographic profile (elution order) of the target anions following an ion chromatographic analysis should closely replicate the profile displayed in the test chromatogram that was shipped when the column was purchased. As a column ages, it is normal to see a gradual shift and shortening of retention times, but if after several years of use, extensive use over less than a year, or use with harsh samples, this retention time has noticeably shifted to any less than 80% of the original recorded value, the column may require cleaning or replacement. Particularly if resolution problems are beginning to become common between previously resolved peaks. A laboratory must retain a historic record of retention times for the surrogate and all the target anions to provide evidence of an analytical columns vitality.

9.4 ASSESSING ANALYTE RECOVERY AND DATA QUALITY

9.4.1 Laboratory Fortified Sample Matrix (LFM) -- The laboratory must add a known amount of analyte to a minimum of 10% of the field samples within an analysis batch. The LFM sample must be prepared from a sample matrix which has been analyzed prior to fortification. The analyte concentration must be high enough to be detected above the original sample and should adhere to the requirement of 9.4.1.2. It is recommended that the solutions used to fortify the LFM be prepared from the same stocks used to prepare the calibration standards and not from external source stocks. This will remove the bias contributed by an externally prepared stock and focus on any potential bias introduced by the field sample matrix.

9.4.1.1 If the fortified concentration is less than the observed background concentration of the unfortified matrix, the recovery should not be calculated. This is due to the difficulty in calculating accurate recoveries of the fortified concentration when the native sample concentration is so high.

9.4.1.2 The LFM should be prepared at concentrations no greater than five times the highest concentration observed in any field sample. If no analyte is observed in any field sample, the LFM must be fortified no greater than five times the lowest calibration level which as outlined in 12.2 is the minimum reported level (MRL). For example, if bromate is not detected in any field samples above the lowest calibrations standard concentration of 5.00 ug/L, the highest LFM fortified concentration allowed is 25.0 ug/L.

9.4.1.3 Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample. Percent recovery should be calculated using the following equation:

$$R = \frac{C_s - C}{s} \times 100$$

where, R = percent recovery.
C_s = fortified sample concentration
C = sample background concentration
s = concentration equivalent of analyte added to sample.

9.4.1.4 Until sufficient data becomes available (usually a minimum of 20 to 30 analysis), assess laboratory performance against recovery limits of 75 to 125%. When sufficient internal performance data becomes available develop control limits from percent mean recovery and the standard deviation of the mean recovery. The optional control limits must be equal to or better than the required control limits of 75-125%.

9.4.1.5 If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control (Sect. 9.3), the recovery problem encountered with the LFM is judged to be matrix induced and the results for that sample and the LFM are reported with a "matrix induced bias" qualifier.

9.4.2 SURROGATE RECOVERY -- Calculate the surrogate recovery from all analyses using the following formula

$$R = \frac{SRC}{SFC} \times 100$$

where, R = percent recovery.
SRC = Surrogate Recovered Concentration
SFC = Surrogate Fortified Concentration

9.4.2.1 Surrogate recoveries must fall between 90-115% for proper instrument performance and analyst technique to be verified. The recovery of the surrogate is slightly bias to 115% to allow for the potential contribution of trace levels of dichloroacetate as the halogenated organic disinfection by-product (DBP) dichloroacetic acid (DCAA) Background levels of this organic DBP are rarely observed above 50 ug/L (0.05 mg/L) which constitutes only 5% of the 1.00 mg/L recommended fortified concentration.

9.4.2.2 If the surrogate recovery falls outside the 90-115% recovery window, a analysis error is evident and sample reanalysis is

required. Poor recoveries could be the result of imprecise sample injection or analyst fortification errors.

9.4.3 FIELD OR LABORATORY DUPLICATES -- The laboratory must analyze either a field or a laboratory duplicate for a minimum of 10% of the collected field samples or at least one with every analysis batch, whichever is greater. The sample matrix selected for this duplicate analysis must contain measurable concentrations of the target anions in order to establish the precision of the analysis set and insure the quality of the data. If none of the samples within an analysis batch have measurable concentrations, the LFM should be employed as a laboratory duplicate.

9.4.3.1 Calculate the relative percent difference (RPD) of the initial quantitated concentration (I_C) and duplicate quantitated concentration (D_c) using the following formula,

$$RPD = \frac{(I_C - D_c)}{([I_C + D_c]/2)} \times 100$$

9.4.3.2 Duplicate analysis acceptance criteria

Concentration range	RPD Limits
MRL to 10xMRL	+/- 20 %
10xMRL to highest calibration level	+/- 10 %

9.4.3.3 If the RPD fails to meet these criteria, the samples must be reported with a qualifier identifying the sample analysis result as yielding a poor duplicate analysis RPD. This should not be a chronic problem and if it frequently recurs (>20% of duplicate analyses) it indicates a problem with the instrument or individual technique.

9.4.4 Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

9.4.5 In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of different columns, injection volumes, and/or eluents, to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in Sect. 9.2 and adhere to the condition of baseline stability found in Sect. 1.2.1.

9.4.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most

productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should perform analysis of quality control check samples and participate in relevant performance evaluation sample studies.

10. CALIBRATION AND STANDARDIZATION

10.1 Establish ion chromatographic operating parameters equivalent to those indicated in Tables 1A or 1B if employing a 2 mm column, Table 1C if employing a 4 mm column.

10.2 Estimate the Linear Calibration Range (LCR) -- The LCR should cover the expected concentration range of the field samples and should not extend over more than 2 orders of magnitude in concentration (For example, if quantitating nitrate in the expected range of 1.0 mg/L to 10 mg/L, 2 orders of magnitude would permit the minimum and maximum calibration standards of 0.20 mg/L and 20 mg/L, respectively.) The restriction of 2 orders of magnitude is prescribed since beyond this it is difficult to maintain linearity throughout the entire calibration range.

10.2.1 If quantification is desired over a larger range, then two separate calibration curves should be prepared.

10.2.2 For an individual calibration curve, a minimum of three calibration standards are required for a curve that extends over a single order of magnitude and a minimum of five calibration standards are required if the curve covers two orders of magnitude. (For example, using the nitrate example cited above in section 10.2, but in this case limit the curve to extend only from 1.0 mg/L to 10 mg/L or a single order of magnitude. A third standard is required somewhere in the middle of the range. For the calibration range of 0.20 mg/L to 20 mg/L, over two orders of magnitude, five calibrations standards should be employed, one each at the lower and upper concentration ranges and the other three proportionally divided throughout the middle of the curve.)

10.3 Prepare the calibration standards by carefully adding measured volumes of one or more stock standards (7.3) to a volumetric flask and diluting to volume with reagent water.

10.3.1 For the Part B anions, EDA must be added to the calibration standards at 50 mg/L. The addition of EDA to all reagent water prepared calibration and quality control samples is required not as a preservative but rather as a means to normalize any bias attributed by the presence of EDA in the field samples.

10.3.2 Prepare a 10.0 mL aliquot of surrogate fortified calibration solution which can be held for direct manual injection or used to fill an autosampler vial. Add 20 uL of the surrogate solution (7.5) to a 20 mL disposable plastic micro beaker. Using a 10.0 mL disposable pipet, place

exactly 10.0 mL of calibration standard into the micro beaker and mix. The calibration standard is now ready for analysis. The same surrogate solution that has been employed for the standards should also be used in the section 11.3.2 for the field samples.

10.4 Using a 2 mm column, inject 10 uL (Part A) or 50 uL (Part B) of each calibration standard. Using a 4 mm column, inject 50 uL (Part A) or 200 uL (Part B) of each calibration standard. Tabulate peak area responses against the concentration. The results are used to prepare calibration curves using a linear least squares fit for each analyte. Acceptable calibration curves are confirmed after reviewing the curves for linearity and passing the criteria for the initial calibration check standard in section 10.5.1. Alternately, if the ratio of response to concentration (response factor) is constant over the LCR (indicated by < 15% relative standard deviation (RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve,

10.4.1 Peak areas are strongly recommended since they have been found to be more consistent, in terms of quantitation, than peak heights. Peak height can tend to be suppressed as a result of high levels of common anions in a given matrix which can compete for exchange sites. Using peak areas, it is the analyst responsibility to review all chromatograms to insure accurate baseline integration of target analyte peaks since poorly drawn baselines will more significantly influence peak areas than peak heights.

10.5 Once the calibration curves have been established they must be verified prior to conducting any sample analysis using an initial calibration check standard (3.2.2). This verification must be performed on each analysis day or whenever fresh eluent has been prepared. A continuing calibration check standard (3.2.3) must be analyzed after every tenth sample and at the end of the analysis set as an end calibration check standard (3.2.4). The response for the initial, continuing and end calibration check must satisfy the criteria listed in 10.5.1. If during the analysis set, the response differs by more than the calibration verification criteria shown in 10.5.1., or the retention times shift more than ± 5% from the expected values for any analyte, the test must be repeated, using fresh calibration standards. If the results are still outside these criteria, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable calibration check standard must be reanalyzed.

10.5.1 Control limits for calibration verification

Concentration range	Percent Recovery Limits
MRL to 10xMRL	75 - 125 %
10xMRL to highest calibration level	85 - 115 %

10.5.1.1 These control limits only apply if the MRL is established within a factor of 10 times the MDL. Otherwise, the limits are set at 85% to 115%.

10.5.2 CALIBRATION VERIFICATION REQUIREMENT FOR PART B
As a mandatory requirement of calibration verification, the laboratory MUST verify calibration using the lowest calibration standard as the initial calibration check standard.

10.5.3 After satisfying the requirement of 10.5.2, the levels selected for the other calibration check standards should be varied between a middle calibration level and the highest calibration level.

11. PROCEDURE

11.1 Tables 1A and 1B summarize the recommended operating conditions for the ion chromatograph. Included in these tables are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the requirements of Sect. 9.2 are met.

11.2 Check system calibration daily and, if required, recalibrate as described in Sect. 10.

11.3 Sample Preparation

11.3.1 For refrigerated or samples arriving to the laboratory cold, ensure the samples have come to room temperature prior to conducting sample analysis by allowing the samples to warm on the bench for at least 1 hour.

11.3.2 Prepare a 10.0 mL aliquot of surrogate fortified sample which can be held for direct manual injection or used to fill an autosampler vial. Add 20 uL of the surrogate solution (7.5) to a 20 mL disposable plastic micro beaker. Using a 10.0 mL disposable pipet, place exactly 10.0 mL of sample into the micro beaker and mix. Sample is now ready for analysis.

11.3.2.1 The less than 1% dilution error introduced by the addition of the surrogate is considered insignificant.

11.4 Using a Luer lock, plastic 10 mL syringe, withdraw the sample from the micro beaker and attach a 0.45 um particulate filter (demonstrated to be free of ionic contaminants) directly to the syringe. Filter the sample into an autosampler vial (If vial is not designed to automatically filter) or manually load the injection loop injecting a fixed amount of well mixed sample. If using a manually loaded injection loop, flush the loop thoroughly between sample analysis using sufficient volumes of each new sample matrix.

11.5 Using a 2 mm column, inject 10 uL (Part A) or 50 uL (Part B) of each sample. Using a 4 mm column, inject 40 uL (Part A) or 200 uL (Part B) of each sample. Tabulate peak area responses against the concentration. During this procedure, retention times must be recorded. Use the same size loop for standards and

samples. Record the resulting peak size in area units. An automated constant volume injection system may also be used.

11.6 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11.7 If the response of a sample analyte exceeds the calibration range, the sample may be diluted with an appropriate amount of reagent water and reanalyzed. If this is not possible then three new calibration concentrations must be employed to create a separate high concentration curve, one standard near the estimated concentration and the other two bracketing around an interval equivalent to ± 25% the estimated concentration. The latter procedure involves significantly more time than a simple sample dilution therefore, it is advisable to collect sufficient sample to allow for sample dilution or sample reanalysis, if required.

11.8 Shifts in retention time are inversely proportional to concentration. Nitrate, phosphate and sulfate will exhibit the greatest degree of change, although all anions can be affected. In some cases this peak migration may produce poor resolution or make peak identification difficult.

11.9 Should more complete resolution be needed between any two coeluting peaks, the eluent (7.2) can be diluted. This will spread out the run, however, and will cause late eluting anions to be retained even longer. The analysts must verify that this dilution does not negatively affect performance by repeating and passing all the QC criteria in Section 9. As a specific precaution, upon dilution of the carbonate eluent, a peak for bicarbonate may be observed within the retention time window for bromate which will negatively impact the analysis.

11.9.1 Eluent dilution will reduce the overall response of an anion due to chromatographic band broadening which will be evident by shortened and broadened peaks. This will adversely effect the MDLs for each analyte.

12. DATA ANALYSIS AND CALCULATIONS

12.1 Prepare a calibration curve for each analyte by plotting instrument response, as peak area, against standard concentration. Compute sample concentration by comparing sample response with the standard curve. If a sample has been diluted, multiply the response by the appropriate dilution factor.

12.2 Report ONLY those values that fall between the lowest and the highest calibration standards. Samples with target analyte responses exceeding the highest standard should be diluted and reanalyzed. Samples with target analytes identified but quantitated below the concentration established by the lowest

calibration standard should be reported as below the minimum reporting limit (MRL).

12.3 Report results for Part A anions in mg/L and for Part B anions in ug/L.

12.4 Report NO_2^- as N
NO_3^- as N
$HPO_4^=$ as P
Br^- in mg/L when reported with Part A
Br^- in ug/L when reported with Part B

13. METHODS PERFORMANCE

13.1 Tables 1A, 1B, and 1C give the single laboratory (OW OGWDW TSC-Cincinnati) retention times, standard conditions and MDL determined for each anion included in the method. MDLs for the Part A anions were determined in reagent water on the 2 mm column (Table 1A). MDLs for the Part B anions were conducted not only in reagent water but also a simulated high ionic strength water (HIW) on the 2 mm column (Table 1B) and in reagent water on the 4 mm column (Table 1C). HIW is designed to simulate a high ionic strength field sample. It was prepared from reagent water which was fortified with the common anions of chloride at 100 mg/L, carbonate at 100 mg/L, nitrate at 10.0 mg/L as nitrogen, phosphate at 10.0 mg/L as phosphorous, and sulfate at 100 mg/L.

13.2 Tables 2A and 2B give the single laboratory (OW OGWDW TSC-Cincinnati) standard deviation for each anion included in the method in a variety of waters for the standard conditions identified in Table 1A and 1B, respectively.

13.3 Tables 3A and 3B shown stability data for the Part A and B anions, respectively. Each data point in these tables represent the mean percent recovery following triplicate analysis. These data were used to formulate the holding times shown in Sect. 8.3.

14. POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 Quantity of the chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

15. WASTE MANAGEMENT

15.1 The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Sect. 14.3.

16. REFERENCES

1. "Determination of Inorganic Disinfection By-Products by Ion Chromatography", J. Pfaff, C. Brockhoff. J. Am. Water Works Assoc., Vol 82, No. 4, pg 192.

2. Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Anions by Ion Chromatography", 18th Edition of Standard Methods (1992).

3. Dionex, System DX500 Operation and Maintenance Manual, Dionex Corp., Sunnyvale, California 94086, 1996.

4. Method Detection Limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, page 1426, December, 1981.

5. American Society for Testing and Materials. Test Method for Anions in Water by Chemically-Suppressed Ion Chromatography D4327-91. Annual Book of Standards, Vol 11.01 (1993).

6. Code of Federal Regulations 40, Ch. 1, Pt. 136, Appendix B.

7. Hautman, D.P. & Bolyard, M. Analysis of Oxyhalide Disinfection By-products and other Anions of Interest in Drinking Water by Ion Chromatography. Jour. of Chromatog., 602, (1992), 65-74.

8. Standard Methods for the Examination of Water and Wastewater, Method 4500-ClO_2,C "Amperometric Method I" (for the determination of Chlorine Dioxide), 19th Edition of Standard Methods (1995).

17. TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

TABLE 1A. CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS IN REAGENT WATER FOR THE COMMON ANIONS (PART A).

			MDL DETERMINATION		
ANALYTE	PEAK # [(1)]	RETENTION TIME (MIN.)	Fort Conc, mg/L	Number of Replicates	DI MDL mg/L
Fluoride	1	2.53	0.020	7	0.009
Chloride	2	4.67	0.020	7	0.004
Nitrite-N	3	6.01	0.010	7	0.001
Surrogate: DCA	4	7.03			
Bromide	5	8.21	0.040	7	0.014
Nitrate-N	6	9.84	0.010	7	0.008
ortho-Phosphate-P	7	11.98	0.040	7	0.019
Sulfate	8	13.49	0.040	7	0.019

Standard Conditions:

Ion Chromatograph: Dionex DX500
Columns : Dionex AG9-HC / AS9-HC, 2 mm
Detector: Suppressed Conductivity Detector, Dionex CD20
Suppressor: ASRS-I, external source electrolytic mode, 100 mA current
Eluent: 9.0 mM Na_2CO_3
Eluent Flow: 0.40 mL/min
Sample Loop: 10 uL

System Backpressure: 2800 psi
Background Conductivity: 22 uS

Recommended method total analysis time: 25 minutes

(1) See Figure 1

TABLE 1B. CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS IN BOTH REAGENT WATER AND HIGH IONIC STRENGTH WATER FOR THE INORGANIC DISINFECTION BY-PRODUCTS (PART B).

ANALYTE	PEAK # [1]	RETENTION TIME (MIN.)	MDL DETERMINATION			
			Fort Conc, ug/L	Number of Replicates	DI MDL ug/L	HIW[2] MDL ug/L
Chlorite	1	3.63	2.00	7	0.89	0.45
Bromate	2	4.19	2.00	7	1.44	1.28
Surrogate: DCA	4	7.28				
Bromide	5	8.48	2.00	7	1.44	2.51
Chlorate	6	9.28	2.00	7	1.31	0.78

Standard Conditions:

Ion Chromatograph: Dionex DX500
Columns : Dionex AG9-HC / AS9-HC, 2 mm
Detector: Suppressed Conductivity Detector, Dionex CD20
Suppressor: ASRS-I, external source electrolytic mode, 100 mA current
Eluent: 9.0 mM Na_2CO_3
Eluent Flow: 0.40 mL/min
Sample Loop: 50 uL

System Backpressure: 2800 psi
Background Conductivity: 22 uS

Recommended method total analysis time: 25 minutes

(1) See Figure 2 and 3

(2) HIW indicates High Ionic Strength Water which is a simulated drinking water prepared from reagent water and fortified with chloride at 100 mg/L, carbonate at 100 mg/L, nitrate at 10.0 mg/L as nitrogen, phosphate at 10.0 mg/L as phosphorous, and sulfate at 100 mg/L.

TABLE 1C. CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS IN REAGENT WATER FOR THE INORGANIC DISINFECTION BY-PRODUCTS USING AN ALTERNATE 4 mm AS9-HC COLUMN (PART B).

ANALYTE	PEAK #	RETENTION TIME (MIN.)	MDL DETERMINATION		
			Fort Conc, ug/L	Number of Replicates	DI MDL ug/L
Chlorite	1	4.43	2.00	7	1.44
Bromate	2	5.10	2.00	7	1.32
Surrogate: DCA	4	8.82			
Bromide	5	10.11	2.00	7	0.98
Chlorate	6	10.94	2.00	7	2.55

Standard Conditions:

Ion Chromatograph: Dionex DX500
Columns : Dionex AG9-HC / AS9-HC, 4 mm
Detector: Suppressed Conductivity Detector, Dionex CD20
Suppressor: ASRS-I, external source electrolytic mode, 300 mA current
Eluent: 9.0 mM Na_2CO_3
Eluent Flow: 1.25 mL/min
Sample Loop: 200 uL

System Backpressure: 1900 psi
Background Conductivity: 21 uS

Recommended method total analysis time: 25 minutes

TABLE 2A. SINGLE-OPERATOR PRECISION AND RECOVERY FOR THE COMMON ANIONS (PART A).

ANALYTE	MATRIX	UNFORT MATRIX CONC., mg/L	FORT CONC mg/L	# OF REPLC	MEAN mg/L	MEAN %REC	SD(n-1)	%RSD
Fluoride	RW	<MDL[1]	2.00	9	1.79	89.7	0.02	1.18
	SW	0.139	2.00	9	1.75	80.4	0.01	0.56
	GW	0.280	2.00	9	1.97	84.3	0.02	0.85
	CDW	0.807	2.00	9	2.59	89.0	0.01	0.46
Chloride	RW	0.029	50.0	9	49.4	98.7	0.03	0.10
	SW	12.1	50.0	9	58.7	93.3	0.04	0.10
	GW	56.6	50.0	9	100.	---[2]	0.22	0.22
	CDW	16.0	50.0	9	64.9	97.8	0.11	0.16
Nitrite-N	RW	<MDL	1.00	9	0.851	85.1	0.00	0.51
	SW	<MDL	1.00	9	0.780	78.0	0.00	0.40
	GW	0.013	1.00	9	0.879	86.6	0.01	0.77
	CDW	<MDL	1.00	9	0.720	72.0	0.00	0.55
Bromide	RW	<MDL	0.500	9	0.480	96.1	0.00	0.92
	SW	0.028	0.500	9	0.469	88.1	0.00	0.94
	GW	0.153	0.500	9	0.634	96.3	0.00	0.52
	CDW	<MDL	0.500	9	0.431	86.2	0.01	1.28
Nitrate-N	RW	<MDL	10.0	9	9.50	95.0	0.01	0.14
	SW	2.12	10.0	9	10.9	87.7	0.03	0.30
	GW	0.016	10.0	9	9.64	96.3	0.03	0.27
	CDW	1.64	10.0	9	10.9	92.4	0.04	0.41
Phosphate-P	RW	<MDL	10.0	9	9.62	96.2	0.01	0.14
	SW	<MDL	10.0	9	8.70	87.0	0.02	0.18
	GW	<MDL	10.0	9	6.12	61.2	0.28	4.66
	CDW	<MDL	10.0	9	9.15	91.5	0.04	0.42
Sulfate	RW	<MDL	50.0	9	44.8	89.5	0.05	0.11
	SW	47.8	50.0	9	92.1	88.6	0.21	0.23
	GW	105	50.0	9	154	---[2]	0.60	0.39
	CDW	57.8	50.0	9	105	---[2]	0.33	0.32
Surrogate:	RW	----	5.00	9	5.12	102.3	0.50	0.49
	SW	----	5.00	9	5.09	102.3	1.12	1.09
	GW	----	5.00	9	5.16	101.8	0.67	0.66
	CDW	----	5.00	9	5.17	103.1	1.36	1.32

RW = Reagent Water GW = Ground Water
SW = Surface Water CDW = chlorine dioxide treated finished drinking water

(1) <MDL indicates less than method detection limit.
(2) Not calculated since amount fortified was less than unfortified native matrix concentration (See 9.4.1.1.).

TABLE 2B. SINGLE-OPERATOR PRECISION AND RECOVERY FOR THE INORGANIC DISINFECTION BY-PRODUCTS (PART B).

ANALYTE	MATRIX	UNFORT CONC. ug/L	FORT CONC ug/L	# OF REPLC	MEAN ug/L	MEAN %REC	SD(n-1)	%RSD
Chlorite	RW	<MDL[1]	100	9	96.2	96.2	0.95	0.99
			500	9	523	105	3.13	0.60
	HIW	<MDL	100	9	102	102	2.19	2.15
			500	9	520	104	3.64	0.70
	SW	<MDL	100	9	91.4	91.4	1.22	1.33
			500	9	495	99.0	7.54	1.52
	GW	<MDL	100	9	92.9	92.9	1.65	1.77
			500	9	490	98.1	3.40	0.69
	ClW	<MDL	100	9	87.4	87.4	0.59	0.68
			500	9	485	97.1	6.36	1.31
	CDW	292	100	9	396	---[2]	1.64	0.41
			500	9	811	104	4.00	0.49
	O3W	<MDL	100	9	84.4	84.4	0.46	0.54
			500	9	481	96.1	3.24	0.67
Bromate	RW	<MDL	5.00	9	5.04	101	0.45	8.86
			25.0	9	26.5	106	1.71	6.47
	HIW	<MDL	5.00	9	4.88	97.5	0.95	19.5
			25.0	9	25.6	102	1.37	5.37
	SW	<MDL	5.00	9	4.46	89.2	0.58	13.0
			25.0	9	26.3	105	1.10	4.18
	GW	<MDL	5.00	9	5.10	102	0.50	9.75
			25.0	9	22.2	88.9	1.29	5.81
	ClW	<MDL	5.00	9	4.63	92.6	0.77	16.7
			25.0	9	25.1	100	1.64	6.55
	CDW	<MDL	5.00	9	4.14	82.7	0.62	15.1
			25.0	9	25.1	101	1.28	5.09
	O3W	1.45	5.00	9	5.49	80.9	0.61	11.1
			25.0	9	24.1	90.6	1.13	4.69

RW = Reagent Water
HIW = High Ionic strength Water [see note (2) in Table 1B]
SW = Surface Water
GW = Groundwater
ClW = Chlorinated drinking water
CDW = Chlorine dioxide treated drinking water
O3W = Ozonated drinking water

(1) <MDL indicates less than method detection limit.
(2) Not calculated since amount fortified was less than unfortified native matrix concentration (See 9.4.1.1.).

TABLE 2B. SINGLE-OPERATOR PRECISION AND RECOVERY FOR THE INORGANIC DISINFECTION BY-PRODUCTS (PART B) (contd.).

ANALYTE	MATRIX	UNFORT CONC. ug/L	FORT CONC ug/L	# OF REPLC	MEAN ug/L	MEAN %REC	SD(n-1)	%RSD
Bromide	RW	<MDL[1]	20.0	9	20.9	104	0.80	3.82
			100	9	107	107	0.60	0.56
	HIW	3.24	20.0	9	21.8	92.5	0.79	3.63
			100	9	105	102	1.05	1
	SW	31.0	20.0	9	51.3	---[2]	0.97	1.9
			100	9	140.	109	1.88	1.35
	GW	151	20.0	9	172	---[2]	0.78	0.45
			100	9	265	---[2]	2.18	0.82
	ClW	16.3	20.0	9	39.3	115	0.64	1.62
			100	9	125	109	2.00	1.6
	CDW	11.5	20.0	9	34.4	115	0.76	2.22
			100	9	125	113	1.24	0.99
	O3W	39.8	20.0	9	65.4	---[2]	3.67	5.61
			100	9	153	113	1.00	0.65
Chlorate	RW	<MDL	100	9	98.3	98.3	0.80	0.82
			500	9	520	104	4.15	0.8
	HIW	<MDL	100	9	86.1	86.1	1.47	1.7
			500	9	502	100.	4.52	0.9
	SW	3.18	100	9	102	98.3	1.57	1.55
			500	9	513	102	7.11	1.39
	GW	<MDL	100	9	93.5	93.5	2.00	2.14
			500	9	510	102	3.84	0.75
	ClW	34.4	100	9	136	102	1.01	0.74
			500	9	549	103	3.11	0.57
	CDW	121	100	9	223	---[2]	3.20	1.44
			500	9	651	106	3.50	0.54
	O3W	6.15	100	9	106	100	1.20	1.13
			500	9	523	103	2.45	0.47

RW = Reagent Water
HIW = High Ionic strength Water [see note (2) in Table 1B]
SW = Surface Water
GW = Groundwater
ClW = Chlorinated drinking water
CDW = Chlorine dioxide treated drinking water
O3W = Ozonated drinking water

(1) <MDL indicates less than method detection limit.
(2) Not calculated since amount fortified was less than unfortified native matrix concentration (See 9.4.1.1.).

TABLE 2B. SINGLE-OPERATOR PRECISION AND RECOVERY FOR THE INORGANIC DISINFECTION BY-PRODUCTS (PART B)(contd.).

ANALYTE	MATRIX	FORT CONC mg/L	# OF REPLC	MEAN mg/L	MEAN %REC	SD(n-1)	%RSD
Surrogate: DCA	RW	5.00	9	5.11	102	0.93	0.91
(see NOTE below)				4.98	99.5	0.69	0.69
	HIW	5.00	9	5.00	100	0.79	0.79
				4.96	99.2	1.76	1.78
	SW	5.00	9	4.95	98.9	0.70	0.7
				4.99	99.8	1.60	1.61
	GW	5.00	9	5.12	102	0.50	0.49
				5.13	103	0.50	0.49
	ClW	5.00	9	5.15	103	1.73	1.68
				5.13	103	1.12	1.09
	CDW	5.00	9	5.01	100	1.02	1.02
				5.04	101	1.08	1.07
	O3W	5.00	9	4.99	99.8	0.70	0.7
				5.11	101	0.53	0.52

RW = Reagent Water
HIW = High Ionic strength Water [see note (2) in Table 1B]
SW = Surface Water
GW = Groundwater
ClW = Chlorinated drinking water
CDW = Chlorine dioxide treated drinking water
O3W = Ozonated drinking water

NOTE: The surrogate DCA was fortified at 5 mg/L but due to concerns about measuring trace concentrations of bromide with such high concentration of the neighboring surrogate peak, the recommended fortified concentration for the surrogate has been reduced to 1.00 mg/L.

TABLE 3A. STABILITY STUDY RESULTS FOR THE COMMON ANIONS (PART A).

ANALYTE	Preservative	Matrix	UNFORT CONC. mg/L	FORT CONC mg/L	Analyte % Recovery Day 0	Day 14	Day 28	See Note
Fluoride	None	RW	<MDL	2.00	89.8	88.3	88.4	
		SW	0.140	2.00	79.9	80.2	80.0	
		GW	0.280	2.00	84.7	87.8	87.0	
		CDW	0.929	2.00	82.9	83.6	81.6	
Chloride	None	RW	<MDL	50.0	98.8	99.1	98.1	
		SW	12.0	50.0	93.4	93.5	92.8	
		GW	56.6	50.0	87.6	87.6	86.5	
		CDW	16.0	50.0	97.9	98.4	97.8	
Nitrite-N	None	RW	<MDL	1.00	85.2	85.5	83.6	
		SW	<MDL	1.00	77.8	76.6	11.9	(1)
		GW	<MDL	1.00	88.2	85.4	56.1	(1)
		CDW	<MDL	1.00	71.9	71.7	73.9	(2)
Bromide	None	RW	<MDL	0.500	95.5	97.0	96.2	
		SW	0.028	0.500	87.5	88.3	86.7	
		GW	0.153	0.500	96.9	96.0	96.1	
		CDW	<MDL	0.500	85.7	87.1	89.2	(2)
Nitrate-N	None	RW	<MDL	10.0	94.9	94.7	94.2	
		SW	2.12	10.0	87.6	87.0	88.7	
		GW	<MDL	10.0	96.5	96.5	95.5	
		CDW	1.64	10.0	92.3	93.3	91.9	
Phosphate-P	None	RW	<MDL	10.0	96.3	95.8	95.2	
		SW	<MDL	10.0	86.9	86.4	85.1	
		GW	<MDL	10.0	62.8	93.1	89.5	(3)
		CDW	<MDL	10.0	91.6	91.4	90.8	
Sulfate	None	RW	<MDL	50.0	89.6	89.3	89.1	
		SW	47.8	50.0	89.0	89.0	88.1	
		GW	105	50.0	97.5	97.3	96.5	
		CDW	57.8	50.0	94.3	94.9	93.8	

NOTES:

(1) Degradation apparent.

(2) Analyte recovery will be adversely effected by reactions with free chlorine.

(3) Phosphate recovery on day 0 is believed to have been adversely effected by biological degradation since the sample sat in the autosampler for 18 hrs prior to analysis

TABLE 3B STABILITY STUDY RESULTS FOR THE INORGANIC DISINFECTION BY-PRODUCTS (PART B).

ANALYTE	Preservative	Matrix	UNFORT CONC. ug/L	FORT CONC ug/L	Analyte % Recovery Day 0	Day 3	Day 10	Day 30	See Note
Chlorite	None	RW	<MDL	500	99.8	100	104	94.3	
		HIW	<MDL	500	99.3	98.5	106	89.3	
		SW	<MDL	500	92	88.5	82.	75.1	(1)
		GW	<MDL	500	93.9	94.5	96.	91.7	
		ClW	<MDL	500	93.7	NA(1)	90.	84.7	(2,3)
		CDW	286	500	98.6	101	91.	77.5	(1,3)
		O3W	<MDL	500	10	NA	82.	90.5	(2)
Chlorite	EDA	RW	<MDL	500	101	101	104	95.3	
		HIW	<MDL	500	98.4	98.7	104	95.4	
		SW	<MDL	500	98.3	97.3	97.	92.7	
		GW	<MDL	500	97.7	97.1	97.	92.6	
		ClW	<MDL	500	98.9	NA	96.	92.6	(2)
		CDW	297	500	103	107	102	94.5	
		O3W	<MDL	500	105	NA	96.	91.9	(2)
Bromate	None	RW	<MDL	25.0	93.6	94.1	110	96.1	
		HIW	<MDL	25.0	100	86.0	105	87.7	
		SW	<MDL	25.0	98.7	95.1	105	102	
		GW	<MDL	25.0	79.4	92.4	77.	82.2	
		ClW	<MDL	25.0	102	NA	101	103	(2)
		CDW	<MDL	25.0	104	96.8	98.	92.1	
		O3W	2.27	25.0	87.3	NA	84.	99.9	(2)
Bromate	EDA	RW	<MDL	25.0	97.3	95.3	99.	102	
		HIW	<MDL	25.0	86.9	86.1	107	91.2	
		SW	<MDL	25.0	100	104	103	94.9	
		GW	<MDL	25.0	83.2	101	88.	88.3	
		ClW	<MDL	25.0	105	NA	101	102	(2)
		CDW	<MDL	25.0	117	97.3	98.	83.9	
		O3W	2.32	25.0	92.6	NA	84.	88.9	(2)

See bottom of next page for explanation of notes

TABLE 3B. STABILITY STUDY RESULTS FOR THE INORGANIC DISINFECTION BY-PRODUCTS (PART B)(contd.)

ANALYTE	Preservative	Matrix	UNFORT CONC. ug/L	FORT CONC ug/L	Analyte % Recovery Day 0	Day 3	Day 10	Day 30	See Note
Bromide	None	RW	<MDL	100	99.4	97.2	107	101	
		HIW	<MDL	100	102	103	105	105	
		SW	30.6	100	102	97.1	107	99.1	
		GW	149	100	97.7	95.3	109	100	
		ClW	4.73	100	8.9	NA(1)	37.	11.4	(2,3)
		CDW	<MDL	100	5.78	23.1	38.	51.3	(3)
		O3W	30.4	100	98.3	NA	120	108	(2)
Bromide	EDA	RW	<MDL	100	98.4	98.6	107	100	
		HIW	<MDL	100	104	103	106	105	
		SW	30.5	100	99.5	98.2	107	100	
		GW	149	100	100	97	114	97.7	
		ClW	11.9	100	101	NA	115	97.4	(2,3)
		CDW	6.14	100	101	96.5	119	110	(3)
		O3W	31.0	100	97.3	NA	122	102	(2)
Chlorate	None	RW	<MDL	500	102	102	105	97.4	
		HIW	<MDL	500	96.5	97.8	101	95.4	
		SW	5.84	500	99.8	97.8	100	96	
		GW	<MDL	500	99.5	98.7	101	99.8	
		ClW	37.8	500	102	NA	104	98.2	(2)
		CDW	125	500	102	99.9	104	99.6	
		O3W	8.34	500	100	NA	103	97.3	(2)
Chlorate	EDA	RW	<MDL	500	104	98.6	103	97.3	
		HIW	<MDL	500	97.3	103	100	95	
		SW	6.70	500	99.7	98.2	99.	95.6	
		GW	<MDL	500	102	97	101	99.3	
		ClW	38.2	500	101	NA	102	96.1	(2)
		CDW	123	500	102	96.5	105	97.7	
		O3W	8.62	500	98.4	NA	103	96.4	(2)

NOTES:

(1) Degradation in the unpreserved matrix is apparent.

(2) NA indicates "NOT ANALYZED"

(3) Analyte recovery will be adversely effected by reactions with free chlorine.

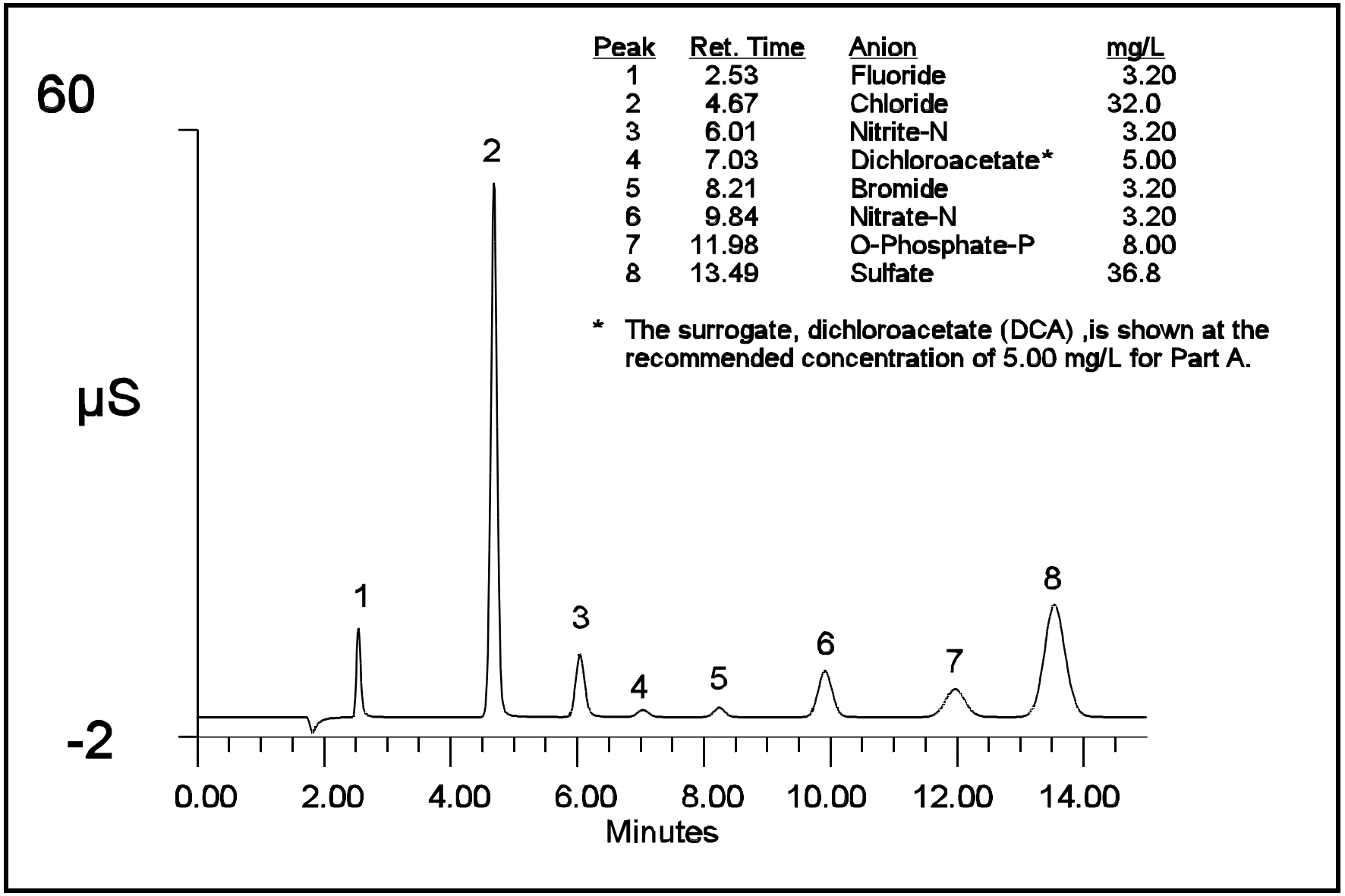

Figure 1. Chromatogram showing separation of the Part A common anions on the AS9-HC column. See Table 1A for analysis conditions.

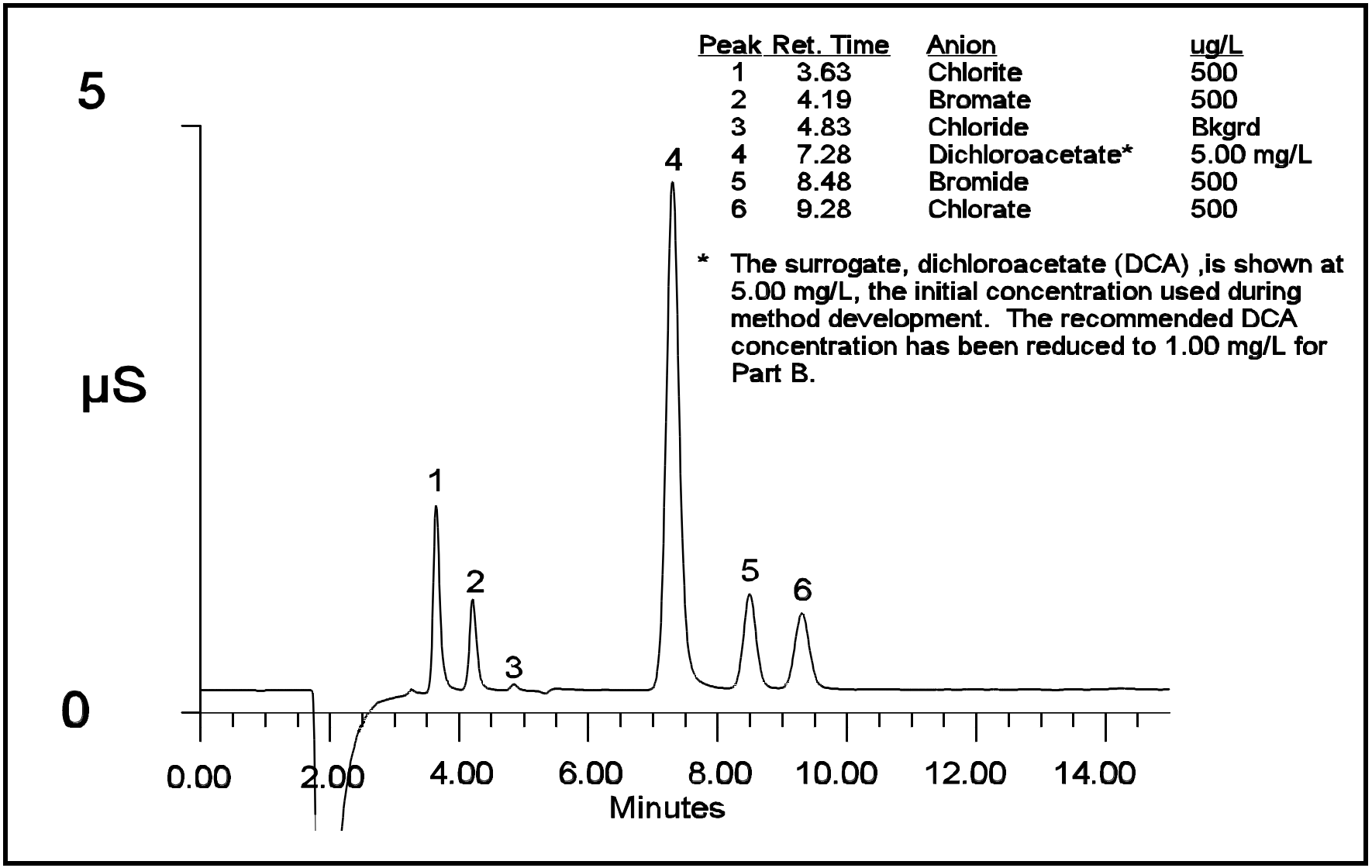

Figure 2 Chromatogram showing separation of the Part B inorganic DBPs and bromide on the AS9-HC column. See Table 1B for analysis conditions.

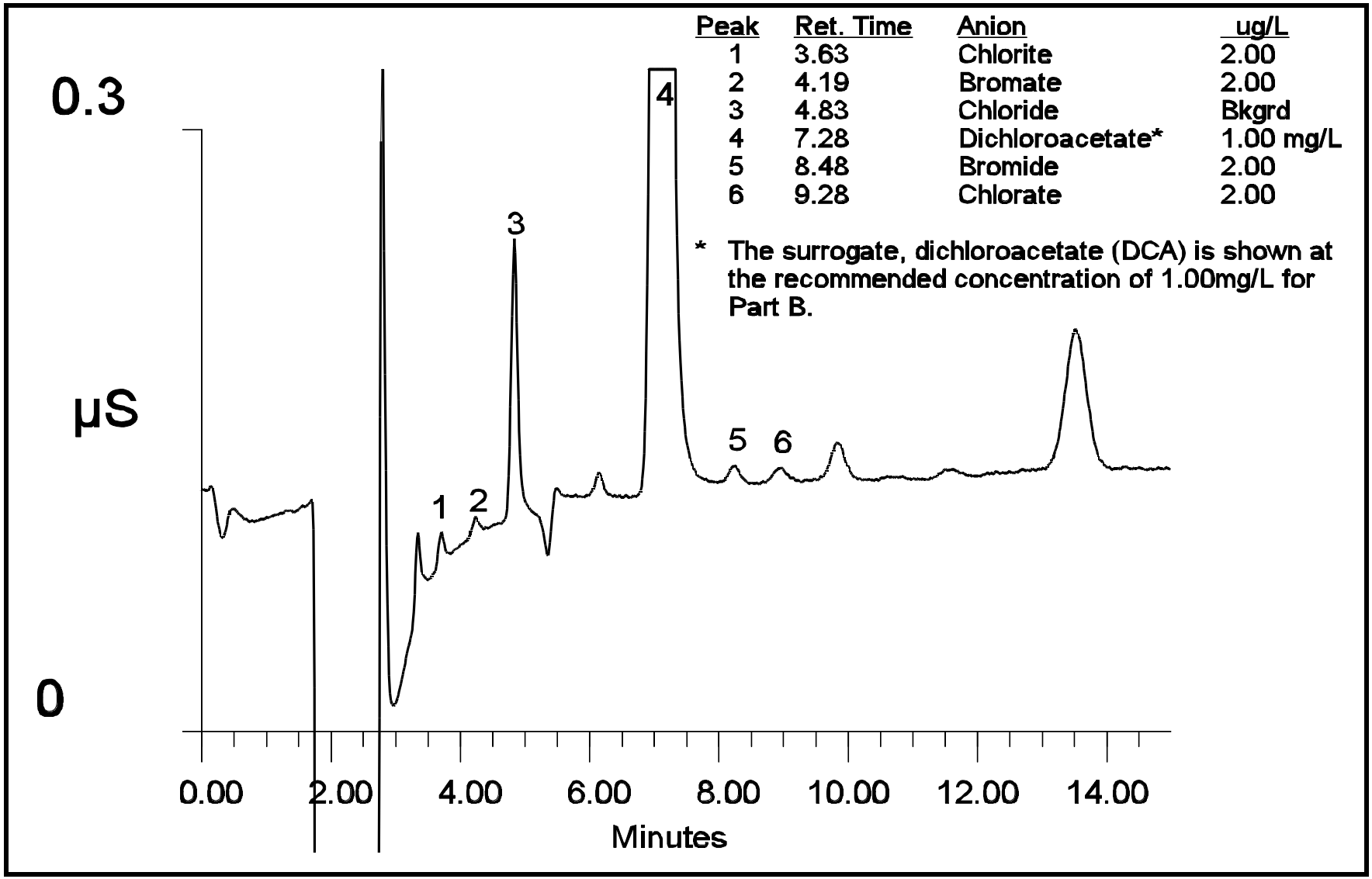

Figure 3. Chromatogram of the inorganic DBPs and bromide (Part B) during the MDL determination in reagent water. See Table 1B for analysis conditions.

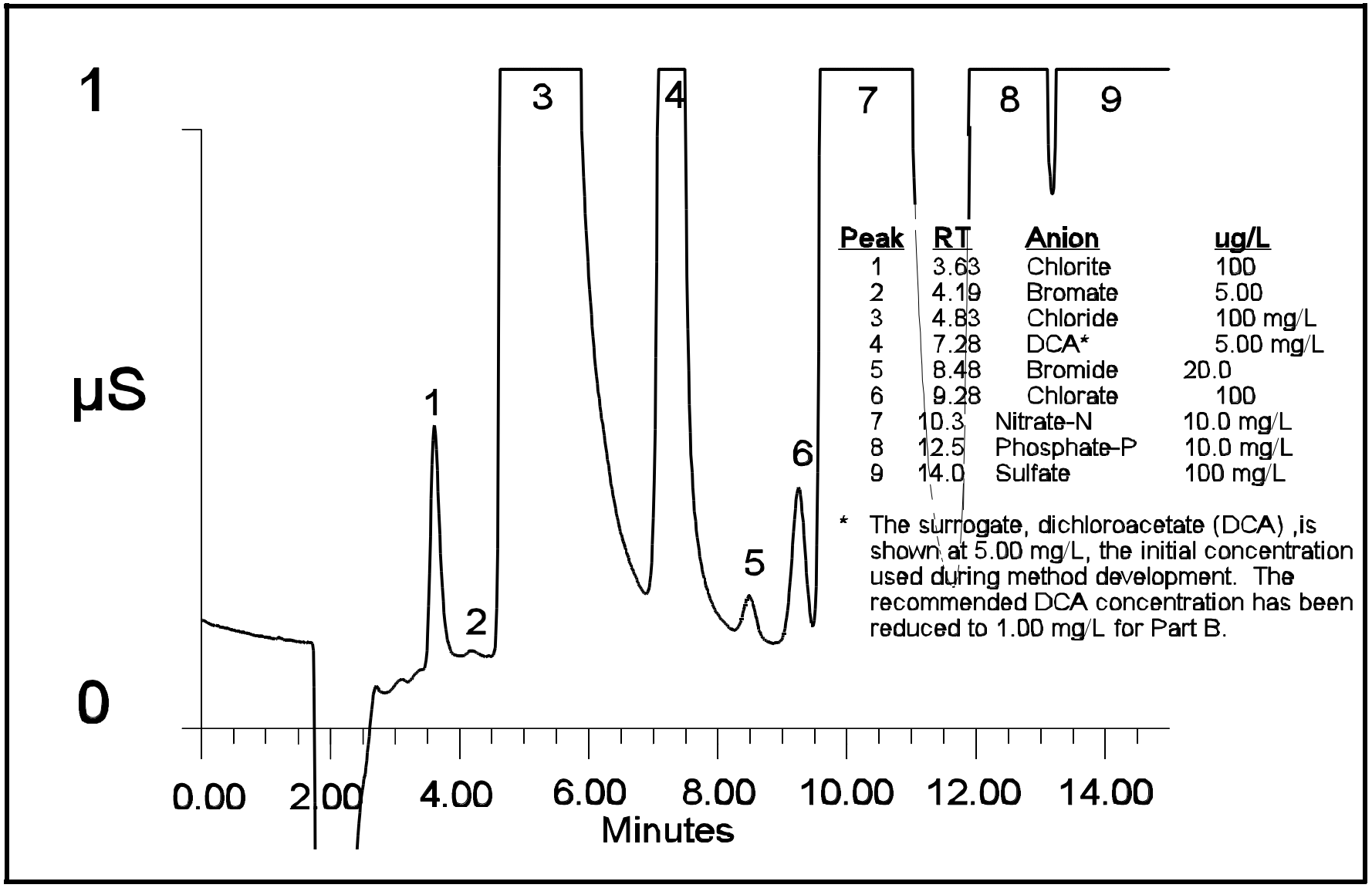

Figure 4. Chromatogram of the inorganic DBPs and bromide (Part B) in high ionic strength water (HIW). See Table 1B for analysis conditions.

Appendix E
EPA Test Method TO-15

EPA/625/R-96/010b

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air

Second Edition

Compendium Method TO-15

Determination Of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry (GC/MS)

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U.S. Environmental Protection Agency
Cincinnati, OH 45268

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Method TO-15
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DISCLAIMER

This Compendium has been subjected to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

METHOD TO-15

Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry (GC/MS)

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METHOD TO-15

Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry (GC/MS)

1. Scope

1.1 This method documents sampling and analytical procedures for the measurement of subsets of the 97 volatile organic compounds (VOCs) that are included in the 189 hazardous air pollutants (HAPs) listed in Title III of the Clean Air Act Amendments of 1990. VOCs are defined here as organic compounds having a vapor pressure greater than 10^{-1} Torr at 25°C and 760 mm Hg. Table 1 is the list of the target VOCs along with their CAS number, boiling point, vapor pressure and an indication of their membership in both the list of VOCs covered by Compendium Method TO-14A (1) and the list of VOCs in EPA's Contract Laboratory Program (CLP) document entitled: *Statement-of-Work (SOW) for the Analysis of Air Toxics from Superfund Sites (2)*.

Many of these compounds have been tested for stability in concentration when stored in specially-prepared canisters (see Section 8) under conditions typical of those encountered in routine ambient air analysis. The stability of these compounds under all possible conditions is not known. However, a model to predict compound losses due to physical adsorption of VOCs on canister walls and to dissolution of VOCs in water condensed in the canisters has been developed (3). Losses due to physical adsorption require only the establishment of equilibrium between the condensed and gas phases and are generally considered short term losses, (i.e., losses occurring over minutes to hours). Losses due to chemical reactions of the VOCs with cocollected ozone or other gas phase species also account for some short term losses. Chemical reactions between VOCs and substances inside the canister are generally assumed to cause the gradual decrease of concentration over time (i.e., long term losses over days to weeks). Loss mechanisms such as aqueous hydrolysis and biological degradation (4) also exist. No models are currently known to be available to estimate and characterize all these potential losses, although a number of experimental observations are referenced in Section 8. Some of the VOCs listed in Title III have short atmospheric lifetimes and may not be present except near sources.

1.2 This method applies to ambient concentrations of VOCs above 0.5 ppbv and typically requires VOC enrichment by concentrating up to one liter of a sample volume. The VOC concentration range for ambient air in many cases includes the concentration at which continuous exposure over a lifetime is estimated to constitute a 10^{-6} or higher lifetime risk of developing cancer in humans. Under circumstances in which many hazardous VOCs are present at 10^{-6} risk concentrations, the total risk may be significantly greater.

1.3 This method applies under most conditions encountered in sampling of ambient air into canisters. However, the composition of a gas mixture in a canister, under unique or unusual conditions, will change so that the sample is known not to be a true representation of the ambient air from which it was taken. For example, low humidity conditions in the sample may lead to losses of certain VOCs on the canister walls, losses that would not happen if the humidity were higher. If the canister is pressurized, then condensation of water from high humidity samples may cause fractional losses of water-soluble compounds. Since the canister surface area is limited, all gases are in competition for the available active sites. Hence an absolute storage stability cannot be assigned to a specific gas. Fortunately, under conditions of normal usage for sampling ambient air, most VOCs can be recovered from canisters near their original concentrations after storage times of up to thirty days (see Section 8).

1.4 Use of the Compendium Method TO-15 for many of the VOCs listed in Table 1 is likely to present two difficulties: (1) what calibration standard to use for establishing a basis for testing and quantitation, and (2) how

to obtain an audit standard. In certain cases a chemical similarity exists between a thoroughly tested compound and others on the Title III list. In this case, what works for one is likely to work for the other in terms of making standards. However, this is not always the case and some compound standards will be troublesome. The reader is referred to the Section 9.2 on standards for guidance. Calibration of compounds such as formaldehyde, diazomethane, and many of the others represents a challenge.

1.5 Compendium Method TO-15 should be considered for use when a subset of the 97 Title III VOCs constitute the target list. Typical situations involve ambient air testing associated with the permitting procedures for emission sources. In this case sampling and analysis of VOCs is performed to determine the impact of dispersing source emissions in the surrounding areas. Other important applications are prevalence and trend monitoring for hazardous VOCs in urban areas and risk assessments downwind of industrialized or source-impacted areas.

1.6 Solid adsorbents can be used in lieu of canisters for sampling of VOCs, provided the solid adsorbent packings, usually multisorbent packings in metal or glass tubes, can meet the performance criteria specified in Compendium Method TO-17 which specifically addresses the use of multisorbent packings. The two sample collection techniques are different but become the same upon movement of the sample from the collection medium (canister or multisorbent tubes) onto the sample concentrator. Sample collection directly from the atmosphere by automated gas chromatographs can be used in lieu of collection in canisters or on solid adsorbents.

2. Summary of Method

2.1 The atmosphere is sampled by introduction of air into a specially-prepared stainless steel canister. Both subatmospheric pressure and pressurized sampling modes use an initially evacuated canister. A pump ventilated sampling line is used during sample collection with most commercially available samplers. Pressurized sampling requires an additional pump to provide positive pressure to the sample canister. A sample of air is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into the pre-evacuated and passivated canister.

2.2 After the air sample is collected, the canister valve is closed, an identification tag is attached to the canister, and the canister is transported to the laboratory for analysis.

2.3 Upon receipt at the laboratory, the canister tag data is recorded and the canister is stored until analysis. Storage times of up to thirty days have been demonstrated for many of the VOCs (5).

2.4 To analyze the sample, a known volume of sample is directed from the canister through a solid multisorbent concentrator. A portion of the water vapor in the sample breaks through the concentrator during sampling, to a degree depending on the multisorbent composition, duration of sampling, and other factors. Water content of the sample can be further reduced by dry purging the concentrator with helium while retaining target compounds. After the concentration and drying steps are completed, the VOCs are thermally desorbed, entrained in a carrier gas stream, and then focused in a small volume by trapping on a reduced temperature trap or small volume multisorbent trap. The sample is then released by thermal desorption and carried onto a gas chromatographic column for separation.

As a simple alternative to the multisorbent/dry purge water management technique, the amount of water vapor in the sample can be reduced below any threshold for affecting the proper operation of the analytical system by

reducing the sample size. For example, a small sample can be concentrated on a cold trap and released directly to the gas chromatographic column. The reduction in sample volume may require an enhancement of detector sensitivity.

Other water management approaches are also acceptable as long as their use does not compromise the attainment of the performance criteria listed in Section 11. A listing of some commercial water management systems is provided in Appendix A. One of the alternative ways to dry the sample is to separate VOCs from condensate on a low temperature trap by heating and purging the trap.

2.5 The analytical strategy for Compendium Method TO-15 involves using a high resolution gas chromatograph (GC) coupled to a mass spectrometer. If the mass spectrometer is a linear quadrupole system, it is operated either by continuously scanning a wide range of mass to charge ratios (SCAN mode) or by monitoring select ion monitoring mode (SIM) of compounds on the target list. If the mass spectrometer is based on a standard ion trap design, only a scanning mode is used (note however, that the Selected Ion Storage (SIS) mode for the ion trap has features of the SIM mode). Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with stored spectra taken under similar conditions, in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the system response to the primary fragment for known amounts of the compound. This establishes the compound concentration that exists in the sample.

Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification. If the technique is supported by a comprehensive mass spectral database and a knowledgeable operator, then the correct identification and quantification of VOCs is further enhanced.

3. Significance

3.1 Compendium Method TO-15 is significant in that it extends the Compendium Method TO-14A description for using canister-based sampling and gas chromatographic analysis in the following ways:

- Compendium Method TO-15 incorporates a multisorbent/dry purge technique or equivalent (see Appendix A) for water management thereby addressing a more extensive set of compounds (the VOCs mentioned in Title III of the CAAA of 1990) than addressed by Compendium Method TO-14A. Compendium Method TO-14A approach to water management alters the structure or reduces the sample stream concentration of some VOCs, especially water-soluble VOCs.
- Compendium Method TO-15 uses the GC/MS technique as the only means to identify and quantitate target compounds. The GC/MS approach provides a more scientifically-defensible detection scheme which is generally more desirable than the use of single or even multiple specific detectors.
- In addition, Compendium Method TO-15 establishes method performance criteria for acceptance of data, allowing the use of alternate but equivalent sampling and analytical equipment. There are several new and viable commercial approaches for water management as noted in Appendix A of this method on which to base a VOC monitoring technique as well as other approaches to sampling (i.e., autoGCs and solid

adsorbents) that are often used. This method lists performance criteria that these alternatives must meet to be acceptable alternatives for monitoring ambient VOCs.
- Finally, Compendium Method TO-15 includes enhanced provisions for inherent quality control. The method uses internal analytical standards and frequent verification of analytical system performance to assure control of the analytical system. This more formal and better documented approach to quality control guarantees a higher percentage of good data.

3.2 With these features, Compendium Method TO-15 is a more general yet better defined method for VOCs than Compendium Method TO-14A. As such, the method can be applied with a higher confidence to reduce the uncertainty in risk assessments in environments where the hazardous volatile gases listed in the Title III of the Clean Air Act Amendments of 1990 are being monitored. An emphasis on risk assessments for human health and effects on the ecology is a current goal for the U.S. EPA.

4. Applicable Documents

4.1 ASTM Standards

- **Method D1356** *Definitions of Terms Relating to Atmospheric Sampling and Analysis*.
- **Method E260** *Recommended Practice for General Gas Chromatography Procedures*.
- **Method E355** *Practice for Gas Chromatography Terms and Relationships*.
- **Method D5466** *Standard Test Method of Determination of Volatile Organic Compounds in Atmospheres (Canister Sampling Methodology)*.

4.2 EPA Documents

- *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II*, U. S. Environmental Protection Agency, EPA-600/R-94-038b, May 1994.
- *Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air*, U. S. Environmental Protection Agency, EPA-600/4-83-027, June 1983.
- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air: Method TO-14, Second Supplement*, U. S. Environmental Protection Agency, EPA-600/4-89-018, March 1989.
- *Statement-of-Work (SOW) for the Analysis of Air Toxics from Superfund Sites*, U. S. Environmental Protection Agency, Office of Solid Waste, Washington, D.C., Draft Report, June 1990.
- *Clean Air Act Amendments of 1990*, U. S. Congress, Washington, D.C., November 1990.

5. Definitions

[Note: Definitions used in this document and any user-prepared standard operating procedures (SOPs) should be consistent with ASTM Methods D1356, E260, and E355. Aside from the definitions given below, all pertinent abbreviations and symbols are defined within this document at point of use.]

5.1 Gauge Pressure—pressure measured with reference to the surrounding atmospheric pressure, usually expressed in units of kPa or psi. Zero gauge pressure is equal to atmospheric (barometric) pressure.

5.2 Absolute Pressure—pressure measured with reference to absolute zero pressure, usually expressed in units of kPa, or psi.

5.3 Cryogen—a refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or on front of the analytical column. Typical cryogens are liquid nitrogen (bp -195.8°C), liquid argon (bp -185.7°C), and liquid CO_2 (bp -79.5°C).

5.4 Dynamic Calibration—calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed and by introducing such standards into the inlet of the sampling or analytical system from a manifold through which the gas standards are flowing.

5.5 Dynamic Dilution—means of preparing calibration mixtures in which standard gas(es) from pressurized cylinders are continuously blended with humidified zero air in a manifold so that a flowing stream of calibration mixture is available at the inlet of the analytical system.

5.6 MS-SCAN—mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.

5.7 MS-SIM—mass spectrometric mode of operation in which the GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly [i.e., selected ion monitoring (SIM) mode].

5.8 Qualitative Accuracy—the degree of measurement accuracy required to correctly identify compounds with an analytical system.

5.9 Quantitative Accuracy—the degree of measurement accuracy required to correctly measure the concentration of an identified compound with an analytical system with known uncertainty.

5.10 Replicate Precision—precision determined from two canisters filled from the same air mass over the same time period and determined as the absolute value of the difference between the analyses of canisters divided by their average value and expressed as a percentage (see Section 11 for performance criteria for replicate precision).

5.11 Duplicate Precision—precision determined from the analysis of two samples taken from the same canister. The duplicate precision is determined as the absolute value of the difference between the canister analyses divided by their average value and expressed as a percentage.

5.12 Audit Accuracy—the difference between the analysis of a sample provided in an audit canister and the nominal value as determined by the audit authority, divided by the audit value and expressed as a percentage (see Section 11 for performance criteria for audit accuracy).

6. Interferences and Contamination

6.1 Very volatile compounds, such as chloromethane and vinyl chloride can display peak broadening and co-elution with other species if the compounds are not delivered to the GC column in a small volume of carrier gas. Refocusing of the sample after collection on the primary trap, either on a separate focusing trap or at the head of the gas chromatographic column, mitigates this problem.

6.2 Interferences in canister samples may result from improper use or from contamination of: (1) the canisters due to poor manufacturing practices, (2) the canister cleaning apparatus, and (3) the sampling or analytical system. Attention to the following details will help to minimize the possibility of contamination of canisters.

6.2.1 Canisters should be manufactured using high quality welding and cleaning techniques, and new canisters should be filled with humidified zero air and then analyzed, after "aging" for 24 hours, to determine cleanliness. The cleaning apparatus, sampling system, and analytical system should be assembled of clean, high quality components and each system should be shown to be free of contamination.

6.2.2 Canisters should be stored in a contaminant-free location and should be capped tightly during shipment to prevent leakage and minimize any compromise of the sample.

6.2.3 Impurities in the calibration dilution gas (if applicable) and carrier gas, organic compounds out-gassing from the system components ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with Buna-N rubber components must be avoided.

6.2.4 Significant contamination of the analytical equipment can occur whenever samples containing high VOC concentrations are analyzed. This in turn can result in carryover contamination in subsequent analyses. Whenever a high concentration (>25 ppbv of a trace species) sample is encountered, it should be followed by an analysis of humid zero air to check for carry-over contamination.

6.2.5 In cases when solid sorbents are used to concentrate the sample prior to analysis, the sorbents should be tested to identify artifact formation (see Compendium Method TO-17 for more information on artifacts).

7. Apparatus and Reagents

[Note: Compendium Method To-14A list more specific requirements for sampling and analysis apparatus which may be of help in identifying options. The listings below are generic.]

7.1 Sampling Apparatus

[Note: Subatmospheric pressure and pressurized canister sampling systems are commercially available and have been used as part of U.S. Environmental Protection Agency's Toxic Air Monitoring Stations (TAMS), Urban Air Toxic Monitoring Program (UATMP), the non-methane organic compound (NMOC) sampling and analysis program, and the Photochemical Assessment Monitoring Stations (PAMS).]

7.1.1 Subatmospheric Pressure (see Figure 1, without metal bellows type pump).

7.1.1.1 Sampling Inlet Line. Stainless steel tubing to connect the sampler to the sample inlet.

7.1.1.2 Sample Canister. Leak-free stainless steel pressure vessels of desired volume (e.g., 6 L), with valve and specially prepared interior surfaces (see Appendix B for a listing of known manufacturers/resellers of canisters).

7.1.1.3 Stainless Steel Vacuum/Pressure Gauges. Two types are required, one capable of measuring vacuum (–100 to 0 kPa or 0 to - 30 in Hg) and pressure (0–206 kPa or 0–30 psig) in the sampling system and a second type (for checking the vacuum of canisters during cleaning) capable of measuring at 0.05 mm Hg (see Appendix B) within 20%. Gauges should be tested clean and leak tight.

7.1.1.4 Electronic Mass Flow Controller. Capable of maintaining a constant flow rate (± 10%) over a sampling period of up to 24 hours and under conditions of changing temperature (20–40°C) and humidity.

7.1.1.5 Particulate Matter Filter. 2-μm sintered stainless steel in-line filter.

7.1.1.6 Electronic Timer. For unattended sample collection.

7.1.1.7 Solenoid Valve. Electrically-operated, bi-stable solenoid valve with Viton® seat and O-rings. A Skinner Magnelatch valve is used for purposes of illustration in the text (see Figure 2).

7.1.1.8 Chromatographic Grade Stainless Steel Tubing and Fittings. For interconnections. All such materials in contact with sample, analyte, and support gases prior to analysis should be chromatographic grade stainless steel or equivalent.

7.1.1.9 Thermostatically Controlled Heater. To maintain above ambient temperature inside insulated sampler enclosure.

7.1.1.10 Heater Thermostat. Automatically regulates heater temperature.

7.1.1.11 Fan. For cooling sampling system.

7.1.1.12 Fan Thermostat. Automatically regulates fan operation.

7.1.1.13 Maximum-Minimum Thermometer. Records highest and lowest temperatures during sampling period.

7.1.1.14 Stainless Steel Shut-off Valve. Leak free, for vacuum/pressure gauge.

7.1.1.15 Auxiliary Vacuum Pump. Continuously draws air through the inlet manifold at 10 L/min. or higher flow rate. Sample is extracted from the manifold at a lower rate, and excess air is exhausted.

[Note: The use of higher inlet flow rates dilutes any contamination present in the inlet and reduces the possibility of sample contamination as a result of contact with active adsorption sites on inlet walls.]

7.1.1.16 Elapsed Time Meter. Measures duration of sampling.

7.1.1.17 Optional Fixed Orifice, Capillary, or Adjustable Micrometering Valve. May be used in lieu of the electronic flow controller for grab samples or short duration time-integrated samples. Usually appropriate only in situations where screening samples are taken to assess future sampling activity.

7.1.2 Pressurized (see Figure 1 with metal bellows type pump and Figure 3).

7.1.2.1 Sample Pump. Stainless steel, metal bellows type, capable of 2 atmospheres output pressure. Pump must be free of leaks, clean, and uncontaminated by oil or organic compounds.

[Note: An alternative sampling system has been developed by Dr. R. Rasmussen, The Oregon Graduate Institute of Science and Technology, 20000 N.W. Walker Rd., Beaverton, Oregon 97006, 503-690-1077, and is illustrated in Figure 3. This flow system uses, in order, a pump, a mechanical flow regulator, and a mechanical compensation flow restrictive device. In this configuration the pump is purged with a large sample flow, thereby eliminating the need for an auxiliary vacuum pump to flush the sample inlet.]

7.1.2.2 Other Supporting Materials. All other components of the pressurized sampling system are similar to components discussed in Sections 7.1.1.1 through 7.1.1.17.

7.2 Analytical Apparatus

7.2.1 Sampling/Concentrator System (many commercial alternatives are available).

7.2.1.1 Electronic Mass Flow Controllers. Used to maintain constant flow (for purge gas, carrier gas and sample gas) and to provide an analog output to monitor flow anomalies.

7.2.1.2 Vacuum Pump. General purpose laboratory pump, capable of reducing the downstream pressure of the flow controller to provide the pressure differential necessary to maintain controlled flow rates of sample air.

7.2.1.3 Stainless Steel Tubing and Stainless Steel Fittings. Coated with fused silica to minimize active adsorption sites.

7.2.1.4 Stainless Steel Cylinder Pressure Regulators. Standard, two-stage cylinder regulators with pressure gauges.

7.2.1.5 Gas Purifiers. Used to remove organic impurities and moisture from gas streams.

7.2.1.6 Six-port Gas Chromatographic Valve. For routing sample and carrier gas flows.

7.2.1.7 Multisorbent Concentrator. Solid adsorbent packing with various retentive properties for adsorbing trace gases are commercially available from several sources. The packing contains more than one type of adsorbent packed in series.

7.2.1.7.1A pre-packed adsorbent trap (Supelco 2-0321) containing 200 mg Carbopack B (60/80 mesh) and 50 mg Carbosieve S-III (60/80 mesh) has been found to retain VOCs and allow some water vapor to pass through (6). The addition of a dry purging step allows for further water removal from the adsorbent trap. The steps constituting the dry purge technique that are normally used with multisorbent traps are illustrated in Figure 4. The optimum trapping and dry purging procedure for the Supelco trap consists of a sample volume of 320 mL and a dry nitrogen purge of 1300 mL. Sample trapping and drying is carried out at 25°C. The trap is back-flushed with helium and heated to 220°C to transfer material onto the GC column. A trap bake-out at 260°C for 5 minutes is conducted after each run.

7.2.1.7.2 An example of the effectiveness of dry purging is shown in Figure 5. The multisorbent used in this case is Tenax/Ambersorb 340/Charcoal (7). Approximately 20% of the initial water content in the sample remains after sampling 500 mL of air. The detector response to water vapor (hydrogen atoms detected by atomic emission detection) is plotted versus purge gas volume. Additional water reduction by a factor of 8 is indicated at temperatures of 45°C or higher. Still further water reduction is possible using a two-stage concentration/dryer system.

7.2.1.8 Cryogenic Concentrator. Complete units are commercially available from several vendor sources. The characteristics of the latest concentrators include a rapid, "ballistic" heating of the concentrator to release any trapped VOCs into a small carrier gas volume. This facilitates the separation of compounds on the gas chromatographic column.

7.2.2 Gas Chromatographic/Mass Spectrometric (GC/MS) System.

7.2.2.1 Gas Chromatograph. The gas chromatographic (GC) system must be capable of temperature programming. The column oven can be cooled to subambient temperature (e.g., -50°C) at the start of the gas chromatographic run to effect a resolution of the very volatile organic compounds. In other designs, the rate of release of compounds from the focusing trap in a two stage system obviates the need for retrapping of compounds on the column. The system must include or be interfaced to a concentrator and have all required accessories including analytical columns and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants or flow controllers with Buna-N rubber components must not be used.

7.2.2.2 Chromatographic Columns. 100% methyl silicone or 5% phenyl, 95% methyl silicone fused silica capillary columns of 0.25- to 0.53-mm I.D. of varying lengths are recommended for separation of many of the possible subsets of target compounds involving nonpolar compounds. However, considering the diversity of the target list, the choice is left to the operator subject to the performance standards given in Section 11.

7.2.2.3 Mass Spectrometer. Either a linear quadrupole or ion trap mass spectrometer can be used as long as it is capable of scanning from 35 to 300 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the instrument performance acceptance criteria when 50 ng or less of p-bromofluorobenzene (BFB) is analyzed.

7.2.2.3.1Linear Quadrupole Technology. A simplified diagram of the heart of the quadrupole mass spectrometer is shown in Figure 6. The quadrupole consists of a parallel set of four rod electrodes mounted in a square configuration. The field within the analyzer is created by coupling opposite pairs of rods together and applying radiofrequency (RF) and direct current (DC) potentials between the pairs of rods. Ions created in the ion source from the reaction of column eluates with electrons from the electron source are moved through the

parallel array of rods under the influence of the generated field. Ions which are successfully transmitted through the quadrupole are said to possess stable trajectories and are subsequently recorded with the detection system. When the DC potential is zero, a wide band of m/z values is transmitted through the quadrupole. This "RF only" mode is referred to as the "total-ion" mode. In this mode, the quadrupole acts as a strong focusing lens analogous to a high pass filter. The amplitude of the RF determines the low mass cutoff. A mass spectrum is generated by scanning the DC and RF voltages using a fixed DC/RF ratio and a constant drive frequency or by scanning the frequency and holding the DC and RF constant. With the quadrupole system only 0.1 to 0.2 percent of the ions formed in the ion source actually reach the detector.

7.2.2.3.2Ion Trap Technology. An ion-trap mass spectrometer consists of a chamber formed between two metal surfaces in the shape of a hyperboloid of one sheet (ring electrode) and a hyperboloid of two sheets (the two end-cap electrodes). Ions are created within the chamber by electron impact from an electron beam admitted through a small aperture in one of the end caps. Radio frequency (RF) (and sometimes direct current voltage offsets) are applied between the ring electrode and the two end-cap electrodes establishing a quadrupole electric field. This field is uncoupled in three directions so that ion motion can be considered independently in each direction; the force acting upon an ion increases with the displacement of the ion from the center of the field but the direction of the force depends on the instantaneous voltage applied to the ring electrode. A restoring force along one coordinate (such as the distance, r, from the ion-trap's axis of radial symmetry) will exist concurrently with a repelling force along another coordinate (such as the distance, z, along the ion traps axis), and if the field were static the ions would eventually strike an electrode. However, in an RF field the force along each coordinate alternates direction so that a stable trajectory may be possible in which the ions do not strike a surface. In practice, ions of appropriate mass-to-charge ratios may be trapped within the device for periods of milliseconds to hours. A diagram of a typical ion trap is illustrated in Figure 7. Analysis of stored ions is performed by increasing the RF voltage, which makes the ions successively unstable. The effect of the RF voltage on the ring electrode is to "squeeze" the ions in the xy plane so that they move along the z axis. Half the ions are lost to the top cap (held at ground potential); the remaining ions exit the lower end cap to be detected by the electron multiplier. As the energy applied to the ring electrode is increased, the ions are collected in order of increasing mass to produce a conventional mass spectrum. With the ion trap, approximately 50 percent of the generated ions are detected. As a result, a significant increase in sensitivity can be achieved when compared to a full scan linear quadrupole system.

7.2.2.4 GC/MS Interface. Any gas chromatograph to mass spectrometer interface that gives acceptable calibration points for each of the analytes of interest and can be used to achieve all acceptable performance criteria may be used. Gas chromatograph to mass spectrometer interfaces constructed of all-glass, glass-lined, or fused silica-lined materials are recommended. Glass and fused silica should be deactivated.

7.2.2.5 Data System. The computer system that is interfaced to the mass spectrometer must allow the continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as a Selected Ion Current Profile (SICP). Software must also be available that allows integrating the abundance in any SICP between specified time or scan number limits. Also, software must be available that allows for the comparison of sample spectra with reference library spectra. The National Institute of Standards and Technology (NIST) or Wiley Libraries or equivalent are recommended as reference libraries.

7.2.2.6 Off-line Data Storage Device. Device must be capable of rapid recording and retrieval of data and must be suitable for long-term, off-line data storage.

7.3 Calibration System and Manifold Apparatus (see Figure 8)

7.3.1 Calibration Manifold. Stainless steel, glass, or high purity quartz manifold, (e.g.,1.25-cm I.D. x 66-cm) with sampling ports and internal baffles for flow disturbance to ensure proper mixing. The manifold should be heated to ~50°C.

7.3.2 Humidifier. 500-mL impinger flask containing HPLC grade deionized water.

7.3.3 Electronic Mass Flow Controllers. One 0 to 5 L/min unit and one or more 0 to 100 mL/min units for air, depending on number of cylinders in use for calibration.

7.3.4 Teflon Filter(s). 47-mm Teflon® filter for particulate collection.

7.4 Reagents

7.4.1 Neat Materials or Manufacturer-Certified Solutions/Mixtures. Best source (see Section 9).

7.4.2 Helium and Air. Ultra-high purity grade in gas cylinders. He is used as carrier gas in the GC.

7.4.3 Liquid Nitrogen or Liquid Carbon Dioxide. Used to cool secondary trap.

7.4.4 Deionized Water. High performance liquid chromatography (HPLC) grade, ultra-high purity (for humidifier).

8. Collection of Samples in Canisters

8.1 Introduction

8.1.1 Canister samplers, sampling procedures, and canister cleaning procedures have not changed very much from the description given in the original Compendium Method TO-14. Much of the material in this section is therefore simply a restatement of the material given in Compendium Method TO-14, repeated here in order to have all the relevant information in one place.

8.1.2 Recent notable additions to the canister technology has been in the application of canister-based systems for example, to microenvironmental monitoring (8), the capture of breath samples (9), and sector sampling to identify emission sources of VOCs (10).

8.1.3 EPA has also sponsored the development of a mathematical model to predict the storage stability of arbitrary mixtures of trace gases in humidified air (3), and the investigation of the SilcoSteel™ process of coating the canister interior with a film of fused silica to reduce surface activity (11). A recent summary of storage stability data for VOCs in canisters is given in the open literature (5).

8.2 Sampling System Description

8.2.1 Subatmospheric Pressure Sampling [see Figure 1 (without metal bellows type pump)].

8.2.1.1 In preparation for subatmospheric sample collection in a canister, the canister is evacuated to 0.05 mm Hg (see Appendix C for discussion of evacuation pressure). When the canister is opened to the atmosphere containing the VOCs to be sampled, the differential pressure causes the sample to flow into the canister. This technique may be used to collect grab samples (duration of 10 to 30 seconds) or time-weighted-average (TWA) samples (duration of 1-24 hours) taken through a flow-restrictive inlet (e.g., mass flow controller, critical orifice).

8.2.1.2 With a critical orifice flow restrictor, there will be a decrease in the flow rate as the pressure approaches atmospheric. However, with a mass flow controller, the subatmospheric sampling system can maintain a constant flow rate from full vacuum to within about 7 kPa (1.0 psi) or less below ambient pressure.

8.2.2 Pressurized Sampling [see Figure 1 (with metal bellows type pump)].

8.2.2.1 Pressurized sampling is used when longer-term integrated samples or higher volume samples are required. The sample is collected in a canister using a pump and flow control arrangement to achieve a typical 101-202 kPa (15-30 psig) final canister pressure. For example, a 6-liter evacuated canister can be filled at 10 mL/min for 24 hours to achieve a final pressure of 144 kPa (21 psig).

8.2.2.2 In pressurized canister sampling, a metal bellows type pump draws in air from the sampling manifold to fill and pressurize the sample canister.

8.2.3 All Samplers.

8.2.3.1 A flow control device is chosen to maintain a constant flow into the canister over the desired sample period. This flow rate is determined so the canister is filled (to about 88.1 kPa for subatmospheric pressure sampling or to about one atmosphere above ambient pressure for pressurized sampling) over the desired sample period. The flow rate can be calculated by:

$$F = \frac{P \times V}{T \times 60}$$

where:

F = flow rate, mL/min.
P = final canister pressure, atmospheres absolute. P is approximately equal to

$$\frac{\text{kPa gauge}}{101.2} + 1$$

V = volume of the canister, mL.
T = sample period, hours.

For example, if a 6-L canister is to be filled to 202 kPa (2 atmospheres) absolute pressure in 24 hours, the flow rate can be calculated by:

$$F = \frac{2 \times 6000}{24 \times 60} = 8.3 \text{ mL/min}$$

8.2.3.2 For automatic operation, the timer is designed to start and stop the pump at appropriate times for the desired sample period. The timer must also control the solenoid valve, to open the valve when starting the pump and to close the valve when stopping the pump.

8.2.3.3 The use of the Skinner Magnelatch valve (see Figure 2) avoids any substantial temperature rise that would occur with a conventional, normally closed solenoid valve that would have to be energized during the entire sample period. The temperature rise in the valve could cause outgassing of organic compounds from the Viton® valve seat material. The Skinner Magnelatch valve requires only a brief electrical pulse to open or close at the appropriate start and stop times and therefore experiences no temperature increase. The pulses may be obtained either with an electronic timer that can be programmed for short (5 to 60 seconds) ON periods, or with a conventional mechanical timer and a special pulse circuit. A simple electrical pulse circuit for operating the Skinner Magnelatch solenoid valve with a conventional mechanical timer is illustrated in Figure 2(a). However, with this simple circuit, the valve may operate unreliably during brief power interruptions or if the timer is manually switched on and off too fast. A better circuit incorporating a time-delay relay to provide more reliable valve operation is shown in Figure 2(b).

8.2.3.4 The connecting lines between the sample inlet and the canister should be as short as possible to minimize their volume. The flow rate into the canister should remain relatively constant over the entire sampling period.

8.2.3.5 As an option, a second electronic timer may be used to start the auxiliary pump several hours prior to the sampling period to flush and condition the inlet line.

8.2.3.6 Prior to field use, each sampling system must pass a humid zero air certification (see Section 8.4.3). All plumbing should be checked carefully for leaks. The canisters must also pass a humid zero air certification before use (see Section 8.4.1).

8.3 Sampling Procedure

8.3.1 The sample canister should be cleaned and tested according to the procedure in Section 8.4.1.

8.3.2 A sample collection system is assembled as shown in Figures 1 and 3 and must be cleaned according to the procedure outlined in Sections 8.4.2 and 8.4.4.

[Note: The sampling system should be contained in an appropriate enclosure.]

8.3.3 Prior to locating the sampling system, the user may want to perform "screening analyses" using a portable GC system, as outlined in Appendix B of Compendium Method TO-14A, to determine potential volatile organics present and potential "hot spots." The information gathered from the portable GC screening analysis would be used in developing a monitoring protocol, which includes the sampling system location, based upon the "screening analysis" results.

8.3.4 After "screening analysis," the sampling system is located. Temperatures of ambient air and sampler box interior are recorded on the canister sampling field test data sheet (FTDS), as documented in Figure 9.

[Note: The following discussion is related to Figure 1]

8.3.5 To verify correct sample flow, a "practice" (evacuated) canister is used in the sampling system.

[Note: For a subatmospheric sampler, a flow meter and practice canister are needed. For the pump-driven system, the practice canister is not needed, as the flow can be measured at the outlet of the system.]

A certified mass flow meter is attached to the inlet line of the manifold, just in front of the filter. The canister is opened. The sampler is turned on and the reading of the certified mass flow meter is compared to the sampler mass flow controller. The values should agree within ±10%. If not, the sampler mass flow meter needs to be recalibrated or there is a leak in the system. This should be investigated and corrected.

[Note: Mass flow meter readings may drift. Check the zero reading carefully and add or subtract the zero reading when reading or adjusting the sampler flow rate to compensate for any zero drift.]

After 2 minutes, the desired canister flow rate is adjusted to the proper value (as indicated by the certified mass flow meter) by the sampler flow control unit controller (e.g., 3.5 mL/min for 24 hr, 7.0 mL/min for 12 hr). Record final flow under "CANISTER FLOW RATE" on the FTDS.

8.3.6 The sampler is turned off and the elapsed time meter is reset to 000.0.

[Note: Whenever the sampler is turned off, wait at least 30 seconds to turn the sampler back on.]

8.3.7 The "practice" canister and certified mass flow meter are disconnected and a clean certified (see Section 8.4.1) canister is attached to the system.

8.3.8 The canister valve and vacuum/pressure gauge valve are opened.

8.3.9 Pressure/vacuum in the canister is recorded on the canister FTDS (see Figure 9) as indicated by the sampler vacuum/pressure gauge.

8.3.10 The vacuum/pressure gauge valve is closed and the maximum-minimum thermometer is reset to current temperature. Time of day and elapsed time meter readings are recorded on the canister FTDS.

8.3.11 The electronic timer is set to start and stop the sampling period at the appropriate times. Sampling starts and stops by the programmed electronic timer.

8.3.12 After the desired sampling period, the maximum, minimum, current interior temperature and current ambient temperature are recorded on the FTDS. The current reading from the flow controller is recorded.

8.3.13 At the end of the sampling period, the vacuum/pressure gauge valve on the sampler is briefly opened and closed and the pressure/vacuum is recorded on the FTDS. Pressure should be close to desired pressure.

[Note: For a subatmospheric sampling system, if the canister is at atmospheric pressure when the field final pressure check is performed, the sampling period may be suspect. This information should be noted on the sampling field data sheet.]

Time of day and elapsed time meter readings are also recorded.

8.3.14 The canister valve is closed. The sampling line is disconnected from the canister and the canister is removed from the system. For a subatmospheric system, a certified mass flow meter is once again connected to the inlet manifold in front of the in-line filter and a "practice" canister is attached to the Magnelatch valve of the sampling system. The final flow rate is recorded on the canister FTDS (see Figure 9).

[Note: For a pressurized system, the final flow may be measured directly.]

The sampler is turned off.

8.3.15 An identification tag is attached to the canister. Canister serial number, sample number, location, and date, as a minimum, are recorded on the tag. The canister is routinely transported back to the analytical laboratory with other canisters in a canister shipping case.

8.4 Cleaning and Certification Program

8.4.1 Canister Cleaning and Certification.

8.4.1.1 All canisters must be clean and free of any contaminants before sample collection.

8.4.1.2 All canisters are leak tested by pressurizing them to approximately 206 kPa (30 psig) with zero air.

[Note: The canister cleaning system in Figure 10 can be used for this task.]

The initial pressure is measured, the canister valve is closed, and the final pressure is checked after 24 hours. If acceptable, the pressure should not vary more than ± 13.8 kPa (± 2 psig) over the 24 hour period.

8.4.1.3 A canister cleaning system may be assembled as illustrated in Figure 10. Cryogen is added to both the vacuum pump and zero air supply traps. The canister(s) are connected to the manifold. The vent shut-off valve and the canister valve(s) are opened to release any remaining pressure in the canister(s). The vacuum pump is started and the vent shut-off valve is then closed and the vacuum shut-off valve is opened. The canister(s) are evacuated to <0.05 mm Hg (see Appendix B) for at least 1 hour.

[Note: On a daily basis or more often if necessary, the cryogenic traps should be purged with zero air to remove any trapped water from previous canister cleaning cycles.]

Air released/evacuated from canisters should be diverted to a fume hood.

8.4.1.4 The vacuum and vacuum/pressure gauge shut-off valves are closed and the zero air shut-off valve is opened to pressurize the canister(s) with humid zero air to approximately 206 kPa (30 psig). If a zero gas generator system is used, the flow rate may need to be limited to maintain the zero air quality.

8.4.1.5 The zero air shut-off valve is closed and the canister(s) is allowed to vent down to atmospheric pressure through the vent shut-off valve. The vent shut-off valve is closed. Repeat Sections 8.4.1.3 through 8.4.1.5 two additional times for a total of three (3) evacuation/pressurization cycles for each set of canisters.

8.4.1.6 At the end of the evacuation/pressurization cycle, the canister is pressurized to 206 kPa (30 psig) with humid zero air. The canister is then analyzed by a GC/MS analytical system. Any canister that has not tested clean (compared to direct analysis of humidified zero air of less than 0.2 ppbv of targeted VOCs) should not be used. As a "blank" check of the canister(s) and cleanup procedure, the final humid zero air fill of 100% of the canisters is analyzed until the cleanup system and canisters are proven reliable (less than 0.2 ppbv of any target VOCs). The check can then be reduced to a lower percentage of canisters.

8.4.1.7 The canister is reattached to the cleaning manifold and is then reevacuated to <0.05 mm Hg (see Appendix B) and remains in this condition until used. The canister valve is closed. The canister is removed from the cleaning system and the canister connection is capped with a stainless steel fitting. The canister is now ready for collection of an air sample. An identification tag is attached to the inlet of each canister for field notes and chain-of-custody purposes. An alternative to evacuating the canister at this point is to store the canisters and reevacuate them just prior to the next use.

8.4.1.8 As an option to the humid zero air cleaning procedures, the canisters are heated in an isothermal oven not to exceed 100°C during evacuation of the canister to ensure that higher molecular weight compounds are not retained on the walls of the canister.

[Note: For sampling more complex VOC mixtures the canisters should be heated to higher temperatures during the cleaning procedure although a special high temperature valve would be needed].

Once heated, the canisters are evacuated to <0.05 mm Hg (see Appendix B) and maintained there for 1 hour. At the end of the heated/evacuated cycle, the canisters are pressurized with humid zero air and analyzed by a GC/MS system after a minimum of 12 hrs of "aging." Any canister that has not tested clean (less than 0.2 ppbv each of targeted compounds) should not be used. Once tested clean, the canisters are reevacuated to <0.05 mm Hg (see Appendix B) and remain in the evacuated state until used. As noted in Section 8.4.1.7, reevacuation can occur just prior to the next use.

8.4.2 Cleaning Sampling System Components.

8.4.2.1 Sample components are disassembled and cleaned before the sampler is assembled. Nonmetallic parts are rinsed with HPLC grade deionized water and dried in a vacuum oven at 50°C. Typically, stainless steel parts and fittings are cleaned by placing them in a beaker of methanol in an ultrasonic bath for 15 minutes. This procedure is repeated with hexane as the solvent.

8.4.2.2 The parts are then rinsed with HPLC grade deionized water and dried in a vacuum oven at 100°C for 12 to 24 hours.

8.4.2.3 Once the sampler is assembled, the entire system is purged with humid zero air for 24 hours.

8.4.3 Zero Air Certification.

[Note: In the following sections, "certification" is defined as evaluating the sampling system with humid zero air and humid calibration gases that pass through all active components of the sampling system. The system is "certified" if no significant additions or deletions (less than 0.2 ppbv each of target compounds) have occurred when challenged with the test gas stream.]

8.4.3.1 The cleanliness of the sampling system is determined by testing the sampler with humid zero air without an evacuated gas sampling canister, as follows.

8.4.3.2 The calibration system and manifold are assembled, as illustrated in Figure 8. The sampler (without an evacuated gas canister) is connected to the manifold and the zero air cylinder is activated to generate a humid gas stream (2 L/min) to the calibration manifold [see Figure 8(b)].

8.4.3.3 The humid zero gas stream passes through the calibration manifold, through the sampling system (without an evacuated canister) to the water management system/VOC preconcentrator of an analytical system.

[Note: The exit of the sampling system (without the canister) replaces the canister in Figure 11.]

After the sample volume (e.g., 500 mL) is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed and refocussed on a cold trap. This trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. The VOCs are refocussed prior to gas chromatographic separation. Then, the oven temperature (programmed) increases and the VOCs begin to elute and are detected by a GC/MS (see Section 10) system. The analytical system should not detect greater than 0.2 ppbv of any targeted VOCs in order for the sampling system to pass the humid zero air certification test. Chromatograms (using an FID) of a certified sampler and contaminated sampler are illustrated in Figures 12(a) and 12(b), respectively. If the sampler passes the humid zero air test, it is then tested with humid calibration gas standards containing selected VOCs at concentration levels expected in field sampling (e.g., 0.5 to 2 ppbv) as outlined in Section 8.4.4.

8.4.4 Sampler System Certification with Humid Calibration Gas Standards from a Dynamic Calibration System

8.4.4.1 Assemble the dynamic calibration system and manifold as illustrated in Figure 8.

8.4.4.2 Verify that the calibration system is clean (less than 0.2 ppbv of any target compounds) by sampling a humidified gas stream, *without* gas calibration standards, with a previously certified clean canister (see Section 8.1).

8.4.4.3 The assembled dynamic calibration system is certified clean if less than 0.2 ppbv of any targeted compounds is found.

8.4.4.4 For generating the humidified calibration standards, the calibration gas cylinder(s) containing nominal concentrations of 10 ppmv in nitrogen of selected VOCs is attached to the calibration system as illustrated in Figure 8. The gas cylinders are opened and the gas mixtures are passed through 0 to 10 mL/min certified mass flow controllers to generate ppb levels of calibration standards.

8.4.4.5 After the appropriate equilibrium period, attach the sampling system (containing a certified evacuated canister) to the manifold, as illustrated in Figure 8(b).

8.4.4.6 Sample the dynamic calibration gas stream with the sampling system.

8.4.4.7 Concurrent with the sampling system operation, realtime monitoring of the calibration gas stream is accomplished by the on-line GC/MS analytical system [Figure 8(a)] to provide reference concentrations of generated VOCs.

8.4.4.8 At the end of the sampling period (normally the same time period used for experiments), the sampling system canister is analyzed and compared to the reference GC/MS analytical system to determine if the concentration of the targeted VOCs was increased or decreased by the sampling system.

8.4.4.9 A recovery of between 90% and 110% is expected for all targeted VOCs.

8.4.5 Sampler System Certification without Compressed Gas Cylinder Standards.

8.4.5.1 Not all the gases on the Title III list are available/compatible with compressed gas standards. In these cases sampler certification must be approached by different means.

8.4.5.2 Definitive guidance is not currently available in these cases; however, Section 9.2 lists several ways to generate gas standards. In general, Compendium Method TO-14A compounds (see Table 1) are available commercially as compressed gas standards.

9. GC/MS Analysis of Volatiles from Canisters

9.1 Introduction

9.1.1 The analysis of canister samples is accomplished with a GC/MS system. Fused silica capillary columns are used to achieve high temporal resolution of target compounds. Linear quadrupole or ion trap mass spectrometers are employed for compound detection. The heart of the system is composed of the sample inlet concentrating device that is needed to increase sample loading into a detectable range. Two examples of concentrating systems are discussed. Other approaches are acceptable as long as they are compatible with achieving the system performance criteria given in Section 11.

9.1.2 With the first technique, a whole air sample from the canister is passed through a multisorbent packing (including single adsorbent packings) contained within a metal or glass tube maintained at or above the surrounding air temperature. Depending on the water retention properties of the packing, some or most of the water vapor passes completely through the trap during sampling. Additional drying of the sample is accomplished after the sample concentration is completed by forward purging the trap with clean, dry helium or another inert gas (air is not used). The sample is then thermally desorbed from the packing and backflushed from the trap onto a gas chromatographic column. In some systems a "refocusing" trap is placed between the primary trap and the gas chromatographic column. The specific system design downstream of the primary trap depends on technical factors such as the rate of thermal desorption and sampled volume, but the objective in most cases is to enhance chromatographic resolution of the individual sample components before detection on a mass spectrometer.

9.1.3 Sample drying strategies depend on the target list of compounds. For some target compound lists, the multisorbent packing of the concentrator can be selected from hydrophobic adsorbents which allow a high percentage of water vapor in the sample to pass through the concentrator during sampling and without significant loss of the target compounds. However, if very volatile organic compounds are on the target list, the adsorbents required for their retention may also strongly retain water vapor and a more lengthy dry purge is necessary prior to analysis.

9.1.4 With the second technique, a whole air sample is passed through a concentrator where the VOCs are condensed on a reduced temperature surface (cold trap). Subsequently, the condensed gases are thermally desorbed and backflushed from the trap with an inert gas onto a gas chromatographic column. This concentration technique is similar to that discussed in Compendium Method TO-14, although a membrane dryer is not used. The sample size is reduced in volume to limit the amount of water vapor that is also collected (100 mL or less may be necessary). The attendant reduction in sensitivity is offset by enhancing the sensitivity of detection, for example by using an ion trap detector.

9.2 Preparation of Standards

9.2.1 Introduction.

9.2.1.1 When available, standard mixtures of target gases in high pressure cylinders must be certified traceable to a NIST Standard Reference Material (SRM) or to a NIST/EPA approved Certified Reference Material (CRM). Manufacturer's certificates of analysis must be retained to track the expiration date.

9.2.1.2 The neat standards that are used for making trace gas standards must be of high purity; generally a purity of 98 percent or better is commercially available.

9.2.1.3 Cylinder(s) containing approximately 10 ppmv of each of the target compounds are typically used as primary stock standards. The components may be purchased in one cylinder or in separate cylinders depending on compatibility of the compounds and the pressure of the mixture in the cylinder. Refer to manufacturer's specifications for guidance on purchasing and mixing VOCs in gas cylinders.

9.2.2 Preparing Working Standards.

9.2.2.1 Instrument Performance Check Standard. Prepare a standard solution of BFB in humidified zero air at a concentration which will allow collection of 50 ng of BFB or less under the optimized concentration parameters.

9.2.2.2 Calibration Standards. Prepare five working calibration standards in humidified zero air at a concentration which will allow collection at the 2, 5, 10, 20, and 50 ppbv level for each component under the optimized concentration parameters.

9.2.2.3 Internal Standard Spiking Mixture. Prepare an internal spiking mixture containing bromochloromethane, chlorobenzene-d_5, and 1,4-difluorobenzene at 10 ppmv each in humidified zero air to be added to the sample or calibration standard. 500 µL of this mixture spiked into 500 mL of sample will result in a concentration of 10 ppbv. The internal standard is introduced into the trap during the collection time for all calibration, blank, and sample analyses using the apparatus shown in Figure 13 or by equivalent means. The volume of internal standard spiking mixture added for each analysis must be the same from run to run.

9.2.3 Standard Preparation by Dynamic Dilution Technique.

9.2.3.1 Standards may be prepared by dynamic dilution of the gaseous contents of a cylinder(s) containing the gas calibration stock standards with humidified zero air using mass flow controllers and a calibration manifold. The working standard may be delivered from the manifold to a clean, evacuated canister using a pump and mass flow controller.

9.2.3.2 Alternatively, the analytical system may be calibrated by sampling directly from the manifold if the flow rates are optimized to provide the desired amount of calibration standards. However, the use of the canister as a reservoir prior to introduction into the concentration system resembles the procedure normally used to collect samples and is preferred. Flow rates of the dilution air and cylinder standards (all expressed in the same units) are measured using a bubble meter or calibrated electronic flow measuring device, and the concentrations of target compounds in the manifold are then calculated using the dilution ratio and the original concentration of each compound.

$$\text{Manifold Conc.} = \frac{(\text{Original Conc.})\ (\text{Std. Gas Flowrate})}{(\text{Air Flowrate}) + (\text{Std. Gas Flowrate})}$$

9.2.3.3 Consider the example of 1 mL/min flow of 10 ppmv standard diluted with 1,000 mL/min of humid air provides a nominal 10 ppbv mixture, as calculated below:

$$\text{Manifold Conc.} = \frac{(10\ \text{ppm})(1\ \text{mL/min})(1000\ \text{ppb/1 ppm})}{(1000\ \text{mL/min}) + (1\ \text{mL/min})} = 10\ \text{ppb}$$

9.2.4 Standard Preparation by Static Dilution Bottle Technique

[Note: Standards may be prepared in canisters by spiking the canister with a mixture of components prepared in a static dilution bottle (12). This technique is used specifically for liquid standards.]

9.2.4.1 The volume of a clean 2-liter round-bottom flask, modified with a threaded glass neck to accept a Mininert septum cap, is determined by weighing the amount of water required to completely fill up the flask. Assuming a density for the water of 1 g/mL, the weight of the water in grams is taken as the volume of the flask in milliliters.

9.2.4.2 The flask is flushed with helium by attaching a tubing into the glass neck to deliver the helium. After a few minutes, the tubing is removed and the glass neck is immediately closed with a Mininert septum cap.

9.2.4.3 The flask is placed in a 60°C oven and allowed to equilibrate at that temperature for about 15 minutes. Predetermined aliquots of liquid standards are injected into the flask making sure to keep the flask temperature constant at 60°C.

9.2.4.4 The contents are allowed to equilibrate in the oven for at least 30 minutes. To avoid condensation, syringes must be preheated in the oven at the same temperature prior to withdrawal of aliquots to avoid condensation.

9.2.4.5 Sample aliquots may then be taken for introduction into the analytical system or for further dilution. An aliquot or aliquots totaling greater than 1 percent of the flask volume should be avoided.

9.2.4.6 Standards prepared by this method are stable for one week. The septum must be replaced with each freshly prepared standard.

9.2.4.7 The concentration of each component in the flask is calculated using the following equation:

$$\text{Concentration, mg/L} = \frac{(V_a)(d)}{V_f}$$

where: V_a = Volume of liquid neat standard injected into the flask, µL.

d = Density of the liquid neat standard, mg/µL.

V_f = Volume of the flask, L.

9.2.4.8 To obtain concentrations in ppbv, the equation given in Section 9.2.5.7 can be used.

[Note: In the preparation of standards by this technique, the analyst should make sure that the volume of neat standard injected into the flask does not result in an overpressure due to the higher partial pressure produced by the standard compared to the vapor pressure in the flask. Precautions should also be taken to avoid a significant decrease in pressure inside the flask after withdrawal of aliquot(s).]

9.2.5 Standard Preparation Procedure in High Pressure Cylinders

[Note: Standards may be prepared in high pressure cylinders (13). A modified summary of the procedure is provided below.]

9.2.5.1 The standard compounds are obtained as gases or neat liquids (greater than 98 percent purity).

9.2.5.2 An aluminum cylinder is flushed with high-purity nitrogen gas and then evacuated to better than 25 in. Hg.

9.2.5.3 Predetermined amounts of each neat standard compound are measured using a microliter or gastight syringe and injected into the cylinder. The cylinder is equipped with a heated injection port and nitrogen flow to facilitate sample transfer.

9.2.5.4 The cylinder is pressurized to 1000 psig with zero nitrogen.

[Note: User should read all SOPs associated with generating standards in high pressure cylinders. Follow all safety requirements to minimize danger from high pressure cylinders.]

9.2.5.5 The contents of the cylinder are allowed to equilibrate (~24 hrs) prior to withdrawal of aliquots into the GC system.

9.2.5.6 If the neat standard is a gas, the cylinder concentration is determined using the following equation:

$$\text{Concentration, ppbv} = \frac{\text{Volume}_{\text{standard}}}{\text{Volume}_{\text{dilution gas}}} \times 10^9$$

[Note: Both values must be expressed in the same units.]

9.2.5.7 If the neat standard is a liquid, the gaseous concentration can be determined using the following equations:

$$V = \frac{nRT}{P}$$

and:

$$n = \frac{(mL)(d)}{MW}$$

where:

V = Gaseous volume of injected compound at EPA standard temperature (25°C) and pressure (760 mm Hg), L.
n = Moles.
R = Gas constant, 0.08206 L-atm/mole °K.
T = 298°K (standard temperature).
P = 1 standard pressure, 760 mm Hg (1 atm).
mL = Volume of liquid injected, mL.
d = Density of the neat standard, g/mL.
MW = Molecular weight of the neat standard expressed, g/g-mole.

The gaseous volume of the injected compound is divided by the cylinder volume at STP and then multiplied by 10^9 to obtain the component concentration in ppb units.

9.2.6 Standard Preparation by Water Methods.

[Note: Standards may be prepared by a water purge and trap method (14) and summarized as follows].

9.2.6.1 A previously cleaned and evacuated canister is pressurized to 760 mm Hg absolute (1 atm) with zero grade air.

9.2.6.2 The air gauge is removed from the canister and the sparging vessel is connected to the canister with the short length of 1/16 in. stainless steel tubing.

[Note: Extra effort should be made to minimize possible areas of dead volume to maximize transfer of analytes from the water to the canister.]

9.2.6.3 A measured amount of the stock standard solution and the internal standard solution is spiked into 5 mL of water.

9.2.6.4 This water is transferred into the sparge vessel and purged with nitrogen for 10 mins at 100 mL/min. The sparging vessel is maintained at 40°C.

9.2.6.5 At the end of 10 mins, the sparge vessel is removed and the air gauge is re-installed, to further pressurize the canister with pure nitrogen to 1500 mm Hg absolute pressure (approximately 29 psia).

9.2.6.6 The canister is allowed to equilibrate overnight before use.

9.2.6.7 A schematic of this approach is shown in Figure 14.

9.2.7 Preparation of Standards by Permeation Tubes.

9.2.7.1 Permeation tubes can be used to provide standard concentration of a trace gas or gases. The permeation of the gas can occur from inside a permeation tube containing the trace species of interest to an air stream outside. Permeation can also occur from outside a permeable membrane tube to an air stream passing through the tube (e.g., a tube of permeable material immersed in a liquid).

9.2.7.2 The permeation system is usually held at a constant temperature to generate a constant concentration of trace gas. Commercial suppliers provide systems for generation and dilution of over 250 compounds. Some commercial suppliers of permeation tube equipment are listed in Appendix D.

9.2.8 Storage of Standards.

9.2.8.1 Working standards prepared in canisters may be stored for thirty days in an atmosphere free of potential contaminants.

9.2.8.2 It is imperative that a storage logbook be kept to document storage time.

10. GC/MS Operating Conditions

10.1 Preconcentrator

The following are typical cryogenic and adsorbent preconcentrator analytical conditions which, however, depend on the specific combination of solid sorbent and must be selected carefully by the operator. The reader is referred to Tables 1 and 2 of Compendium Method TO-17 for guidance on selection of sorbents. An example of a system using a solid adsorbent preconcentrator with a cryofocusing trap is discussed in the literature (15). Oven temperature programming starts above ambient.

10.1.1 Sample Collection Conditions

Cryogenic Trap	**Adsorbent Trap**

Set point	-150°C	Set point	27°C
Sample volume	- up to 100 mL	Sample volume	- up to 1,000 mL
Carrier gas purge flow	- none	Carrier gas purge flow	- selectable

[Note: The analyst should optimize the flow rate, duration of sampling, and absolute sample volume to be used. Other preconcentration systems may be used provided performance standards (see Section 11) are realized.]

10.1.2 Desorption Conditions

Cryogenic Trap		**Adsorbent Trap**	
Desorb Temperature	120°C	Desorb Temperature	Variable
Desorb Flow Rate	~ 3 mL/min He	Desorb Flow Rate	~3 mL/min He
Desorb Time	<60 sec	Desorb Time	<60 sec

The adsorbent trap conditions depend on the specific solid adsorbents chosen (see manufacturers' specifications).

10.1.3 Trap Reconditioning Conditions.

Cryogenic Trap		**Adsorbent Trap**	
Initial bakeout	120°C (24 hrs)	Initial bakeout	
Variable (24 hrs)			
After each run	120°C (5 min)	After each run	Variable (5 min)

10.2 GC/MS System

10.2.1 Optimize GC conditions for compound separation and sensitivity. Baseline separation of benzene and carbon tetrachloride on a 100% methyl polysiloxane stationary phase is an indication of acceptable chromatographic performance.

10.2.2 The following are the recommended gas chromatographic analytical conditions when using a 50-meter by 0.3-mm I.D., 1 µm film thickness fused silica column with refocusing on the column.

Item	**Condition**	
Carrier Gas:	Helium	
Flow Rate:	Generally 1-3 mL/min as recommended by manufacturer	
Temperature Program:	Initial Temperature:	-50°C
	Initial Hold Time:	2 min
	Ramp Rate:	8° C/min
	Final Temperature:	200°C
	Final Hold Time:	Until all target compounds elute.

10.2.3 The following are the recommended mass spectrometer conditions:

Item	**Condition**

Electron Energy:	70 Volts (nominal)
Mass Range:	35-300 amu [the choice of 35 amu excludes the detection of some target compounds such as methanol and formaldehyde, and the quantitation of others such as ethylene oxide, ethyl carbamate, etc. (see Table 2). Lowering the mass range and using special programming features available on modern gas chromatographs will be necessary in these cases, but are not considered here.
Scan Time:	To give at least 10 scans per peak, not to exceed 1 second per scan].

A schematic for a typical GC/MS analytical system is illustrated in Figure 15.

10.3 Analytical Sequence

10.3.1 Introduction. The recommended GC/MS analytical sequence for samples during each 24-hour time period is as follows:

- Perform instrument performance check using bromofluorobenzene (BFB).
- Initiate multi-point calibration or daily calibration checks.
- Perform a laboratory method blank.
- Complete this sequence for analysis of $\leq$20 field samples.

10.4 Instrument Performance Check

10.4.1 Summary. It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. The GC/MS system is set up according to the manufacturer's specifications, and the mass calibration and resolution of the GC/MS system are then verified by the analysis of the instrument performance check standard, bromofluorobenzene (BFB).

10.4.2 Frequency. Prior to the analyses of any samples, blanks, or calibration standards, the Laboratory must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check standard containing BFB. The instrument performance check solution must be analyzed initially and once per 24-hour time period of operation.

The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or daily calibration check criteria) begins at the injection of the BFB which the laboratory records as documentation of a compliance tune.

10.4.3 Procedure. The analysis of the instrument performance check standard is performed by trapping 50 ng of BFB under the optimized preconcentration parameters. The BFB is introduced from a cylinder into the GC/MS via a sample loop valve injection system similar to that shown in Figure 13.

The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is conducted using a single scan prior to the elution of BFB.

10.4.4 Technical Acceptance Criteria. Prior to the analysis of any samples, blanks, or calibration standards, the analyst must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check standard as specified in Table 3.

10.4.5 Corrective Action. If the BFB acceptance criteria are not met, the MS must be retuned. It may be necessary to clean the ion source, or quadrupoles, or take other necessary actions to achieve the acceptance criteria.

10.4.6 Documentation. Results of the BFB tuning are to be recorded and maintained as part of the instrumentation log.

10.5 Initial Calibration

10.5.1 Summary. Prior to the analysis of samples and blanks but after the instrument performance check standard criteria have been met, each GC/MS system must be calibrated at five concentrations that span the monitoring range of interest in an initial calibration sequence to determine instrument sensitivity and the linearity of GC/MS response for the target compounds. For example, the range of interest may be 2 to 20 ppbv, in which case the five concentrations would be 1, 2, 5, 10 and 25 ppbv.

One of the calibration points from the initial calibration curve must be at the same concentration as the daily calibration standard (e.g., 10 ppbv).

10.5.2 Frequency. Each GC/MS system must be recalibrated following corrective action (e.g., ion source cleaning or repair, column replacement, etc.) which may change or affect the initial calibration criteria or if the daily calibration acceptance criteria have not been met.

If time remains in the 24-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed.

If time does not remain in the 24-hour period after meeting the acceptance criteria for the initial calibration, a new analytical sequence shall commence with the analysis of the instrument performance check standard followed by analysis of a daily calibration standard.

10.5.3 Procedure. Verify that the GC/MS system meets the instrument performance criteria in Section 10.4.

The GC must be operated using temperature and flow rate parameters equivalent to those in Section 10.2.2. Calibrate the preconcentration-GC/MS system by drawing the standard into the system. Use one of the standards preparation techniques described under Section 9.2 or equivalent.

A minimum of five concentration levels are needed to determine the instrument sensitivity and linearity. One of the calibration levels should be near the detection level for the compounds of interest. The calibration range should be chosen so that linear results are obtained as defined in Sections 10.5.1 and 10.5.5.

Quantitation ions for the target compounds are shown in Table 2. The primary ion should be used unless interferences are present, in which case a secondary ion is used.

10.5.4 Calculations.

[Note: In the following calculations, an internal standard approach is used to calculate response factors. The area response used is that of the primary quantitation ion unless otherwise stated.]

10.5.4.1 Relative Response Factor (RRF). Calculate the relative response factors for each target compound relative to the appropriate internal standard (i.e., standard with the nearest retention time) using the following equation:

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where: RRF = Relative response factor.
A_x = Area of the primary ion for the compound to be measured, counts.
A_{is} = Area of the primary ion for the internal standard, counts.
C_{is} = Concentration of internal standard spiking mixture, ppbv.
C_x = Concentration of the compound in the calibration standard, ppbv.

[Note: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume of field and QC sample introduced into the trap is the same for each analysis. C_{is} and C_x must be in the same units.]

10.5.4.2 Mean Relative Response Factor. Calculate the mean RRF for each compound by averaging the values obtained at the five concentrations using the following equation:

$$\overline{RRF} = \sum_{i=1}^{n} \frac{x_i}{n}$$

where: $\overline{RRF}$ = Mean relative response factor.
x_i = RRF of the compound at concentration i.
n = Number of concentration values, in this case 5.

10.5.4.3 Percent Relative Standard Deviation (%RSD). Using the RRFs from the initial calibration, calculate the %RSD for all target compounds using the following equations:

$$\%RSD = \frac{SD_{RRF}}{\overline{RRF}} \times 100$$

and

$$SD_{RRF} = \sqrt{\sum_{i=1}^{N} \frac{(RRF_i - \overline{RRF})^2}{N - 1}}$$

where: SD_{RRF} = Standard deviation of initial response factors (per compound).
RRF_i = Relative response factor at a concentration level i.
$\overline{RRF}$ = Mean of initial relative response factors (per compound).

10.5.4.4 Relative Retention Times (RRT). Calculate the RRTs for each target compound over the initial calibration range using the following equation:

$$RRT = \frac{RT_c}{RT_{is}}$$

where: RT_c = Retention time of the target compound, seconds
RT_{is} = Retention time of the internal standard, seconds.

10.5.4.5 Mean of the Relative Retention Times ($\overline{RRT}$). Calculate the mean of the relative retention times ($\overline{RRT}$) for each analyte target compound over the initial calibration range using the following equation:

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT}{n}$$

where: $\overline{RRT}$ = Mean relative retention time for the target compound for each initial calibration standard.

RRT = Relative retention time for the target compound at each calibration level.

10.5.4.6 Tabulate Primary Ion Area Response (Y) for Internal Standard. Tabulate the area response (Y) of the primary ions (see Table 2) and the corresponding concentration for each compound and internal standard.

10.5.4.7 Mean Area Response ($\overline{Y}$) for Internal Standard. Calculate the mean area response ($\overline{Y}$) for each internal standard compound over the initial calibration range using the following equation:

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$$

where: $\overline{Y}$ = Mean area response.

Y = Area response for the primary quantitation ion for the internal standard for each initial calibration standard.

10.5.4.8 Mean Retention Times ($\overline{RT}$). Calculate the mean of the retention times ($\overline{RT}$) for each internal standard over the initial calibration range using the following equation:

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$

where: $\overline{RT}$ = Mean retention time, seconds

RT = Retention time for the internal standard for each initial calibration standard, seconds.

10.5.5 Technical Acceptance Criteria for the Initial Calibration.

10.5.5.1 The calculated %RSD for the RRF for each compound in the calibration table must be less than 30% with at most two exceptions up to a limit of 40%.

[Note: This exception may not be acceptable for all projects. Many projects may have a specific target list of compounds which would require the lower limit for all compounds.]

10.5.5.2 The RRT for each target compound at each calibration level must be withiin 0.06 RRT units of the mean RRT for the compound.

10.5.5.3 The area response Y of at each calibration level must be within 40% of the mean area response $\overline{Y}$ over the initial calibration range for each internal standard.

10.5.5.4 The retention time shift for each of the internal standards at each calibration level must be within 20 s of the mean retention time over the initial calibration range for each internal standard.

10.5.6 Corrective Action.

10.5.6.1 Criteria. If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to meet the initial calibration technical acceptance criteria.

10.5.6.2 Schedule. Initial calibration acceptance criteria ***must*** be met before any field samples, performance evaluation (PE) samples, or blanks are analyzed.

10.6 Daily Calibration

10.6.1 Summary. Prior to the analysis of samples and blanks but after tuning criteria have been met, the initial calibration of each GC/MS system must be routinely checked by analyzing a daily calibration standard to ensure that the instrument continues to remain under control. The daily calibration standard, which is the nominal 10 ppbv level calibration standard, should contain all the target compounds.

10.6.2 Frequency. A check of the calibration curve must be performed once every 24 hours on a GC/MS system that has met the tuning criteria. The daily calibration sequence starts with the injection of the BFB. If the BFB analysis meets the ion abundance criteria for BFB, then a daily calibration standard may be analyzed.

10.6.3 Procedure. The mid-level calibration standard (10 ppbv) is analyzed in a GC/MS system that has met the tuning and mass calibration criteria following the same procedure in Section 10.5.

10.6.4 Calculations. Perform the following calculations.

[Note: As indicated earlier, the area response of the primary quantitation ion is used unless otherwise stated.]

10.6.4.1 Relative Response Factor (RRF). Calculate a relative response factor (RRF) for each target compound using the equation in Section 10.5.4.1.

10.6.4.2 Percent Difference (%D). Calculate the percent difference in the RRF of the daily RRF (24-hour) compared to the mean RRF in the most recent initial calibration. Calculate the %D for each target compound using the following equation:

$$\%D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

where: RRF_c = RRF of the compound in the continuing calibration standard.

$\overline{RRF_i}$ = Mean RRF of the compound in the most recent initial calibration.

10.6.5 Technical Acceptance Criteria. The daily calibration standard must be analyzed at the concentration level and frequency described in this Section 10.6 and on a GC/MS system meeting the BFB instrument performance check criteria (see Section 10.4).

The %D for each target compound in a daily calibration sequence must be within ±30 percent in order to proceed with the analysis of samples and blanks. A control chart showing %D values should be maintained.

10.6.6 Corrective Action. If the daily calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to meet the daily calibration technical acceptance criteria.

Daily calibration acceptance criteria must be met before any field samples, performance evaluation (PE) samples, or blanks are analyzed. If the % D criteria are not met, it will be necessary to rerun the daily calibration sample.

10.7 Blank Analyses

10.7.1 Summary. To monitor for possible laboratory contamination, laboratory method blanks are analyzed at least once in a 24-hour analytical sequence. All steps in the analytical procedure are performed on the blank

using all reagents, standards, equipment, apparatus, glassware, and solvents that would be used for a sample analysis.

A laboratory method blank (LMB) is an unused, certified canister that has not left the laboratory. The blank canister is pressurized with humidified, ultra-pure zero air and carried through the same analytical procedure as a field sample. The injected aliquot of the blank must contain the same amount of internal standards that are added to each sample.

10.7.2 Frequency. The laboratory method blank must be analyzed after the calibration standard(s) and before any samples are analyzed.

Whenever a high concentration sample is encountered (i.e., outside the calibration range), a blank analysis should be performed immediately after the sample is completed to check for carryover effects.

10.7.3 Procedure. Fill a cleaned and evacuated canister with humidified zero air (RH >20 percent, at 25°C). Pressurize the contents to 2 atm.

The blank sample should be analyzed using the same procedure outlined under Section 10.8.

10.7.4 Calculations. The blanks are analyzed similar to a field sample and the equations in Section 10.5.4 apply.

10.7.5 Technical Acceptance Criteria. A blank canister should be analyzed daily.

The area response for each internal standard (IS) in the blank must be within ±40 percent of the mean area response of the IS in the most recent valid calibration.

The retention time for each of the internal standards must be within ±0.33 minutes between the blank and the most recent valid calibration.

The blank should not contain any target analyte at a concentration greater than its quantitation level (three times the MDL as defined in Section 11.2) and should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.

10.7.6 Corrective Action. If the blanks do not meet the technical acceptance criteria, the analyst should consider the analytical system to be out of control. It is the responsibility of the analyst to ensure that contaminants in solvents, reagents, glassware, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated. If contamination is a problem, the source of the contamination must be investigated and appropriate corrective measures need to be taken and documented before further sample analysis proceeds.

If an analyte in the blank is found to be out of control (i.e., contaminated) and the analyte is also found in associated samples, those sample results should be "flagged" as possibly contaminated.

10.8 Sample Analysis

10.8.1 Summary. An aliquot of the air sample from a canister (e.g., 500 mL) is preconcentrated and analyzed by GC/MS under conditions stated in Sections 10.1 and 10.2. If using the multisorbent/dry purge approach, adjust the dry purge volume to reduce water effects in the analytical system to manageable levels.

[Note: The analyst should be aware that pressurized samples of high humidity samples will contain condensed water. As a result, the humidity of the sample released from the canister during analysis will vary

in humidity, being lower at the higher canister pressures and increasing in humidity as the canister pressures decreases. Storage integrity of water soluble compounds may also be affected.]

10.8.2 Frequency. If time remains in the 24-hour period in which an initial calibration is performed, samples may be analyzed without analysis of a daily calibration standard.

If time does not remain in the 24-hour period since the injection of the instrument performance check standard in which an initial calibration is performed, both the instrument performance check standard and the daily calibration standard should be analyzed before sample analysis may begin.

10.8.3 Procedure for Instrumental Analysis. Perform the following procedure for analysis.

10.8.3.1 All canister samples should be at temperature equilibrium with the laboratory.

10.8.3.2 Check and adjust the mass flow controllers to provide correct flow rates for the system.

10.8.3.3 Connect the sample canister to the inlet of the GC/MS analytical system, as shown in Figure 15 [Figure 16 shows an alternate two stage concentrator using multisorbent traps followed by a trap cooled by a closed cycle cooler (15)]. The desired sample flow is established through the six-port chromatographic valve and the preconcentrator to the downstream flow controller. The absolute volume of sample being pulled through the trap must be consistent from run to run.

10.8.3.4 Heat/cool the GC oven and cryogenic or adsorbent trap to their set points. Assuming a six-port value is being used, as soon as the trap reaches its lower set point, the six-port chromatographic valve is cycled to the trap position to begin sample collection. Utilize the sample collection time which has been optimized by the analyst.

10.8.3.5 Use the arrangement shown in Figure 13, (i.e., a gastight syringe or some alternate method) introduce an internal standard during the sample collection period. Add sufficient internal standard equivalent to 10 ppbv in the sample. For example, a 0.5 mL volume of a mixture of internal standard compounds, each at 10 ppmv concentration, added to a sample volume of 500 mL, will result in 10 ppbv of each internal standard in the sample.

10.8.3.6 After the sample and internal standards are preconcentrated on the trap, the GC sampling valve is cycled to the inject position and the trap is swept with helium and heated. Assuming a focusing trap is being used, the trapped analytes are thermally desorbed onto a focusing trap and then onto the head of the capillary column and are separated on the column using the GC oven temperature program. The canister valve is closed and the canister is disconnected from the mass flow controller and capped. The trap is maintained at elevated temperature until the beginning of the next analysis.

10.8.3.7 Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak should be acquired. Scanning also allows identification of unknown compounds in the sample through searching of library spectra.

10.8.3.8 Each analytical run must be checked for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound.

10.8.3.9 Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the laboratory record book.

10.8.4 Calculations. The equation below is used for calculating concentrations.

$$C_x = \frac{A_x C_{is} DF}{A_{is} \overline{RRF}}$$

where: C_x = Compound concentration, ppbv.

A_x = Area of the characteristic ion for the compound to be measured, counts.

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

C_{is} = Concentration of the internal standard spiking mixture, ppbv

.

$\overline{RRF}$ = Mean relative response factor from the initial calibration.

DF = Dilution factor calculated as described in section 2. If no dilution is performed, DF = 1.

[Note: The equation above is valid under the condition that the volume (~500 µL) of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume (~500 mL) of field and QC sample introduced into the trap is the same for each analysis.]

10.8.5 Technical Acceptance Criteria.

[Note: If the most recent valid calibration is an initial calibration, internal standard area responses and RTs in the sample are evaluated against the corresponding internal standard area responses and RTs in the mid level standard (10 ppbv) of the initial calibration.]

10.8.5.1 The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, and continuing calibration technical acceptance criteria at the frequency described in Sections 10.4, 10.5 and 10.6.

10.8.5.2 The field samples must be analyzed along with a laboratory method blank that met the blank technical acceptance criteria.

10.8.5.3 All of the target analyte peaks should be within the initial calibration range.

10.8.5.4 The retention time for each internal standard must be within ±0.33 minutes of the retention time of the internal standard in the most recent valid calibration.

10.8.6 Corrective Action. If the on-column concentration of any compound in any sample exceeds the initial calibration range, an aliquot of the original sample must be diluted and reanalyzed. Guidance in performing dilutions and exceptions to this requirement are given below.

- Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.
- The dilution factor chosen should keep the response of the largest analyte peak for a target compound in the upper half of the initial calibration range of the instrument.

[Note: Analysis involving dilution should be reported with a dilution factor and nature of the dilution gas.]

10.8.6.1 Internal standard responses and retention times must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 20 sec from the latest daily (24-hour) calibration standard (or mean retention time over the initial calibration range), the GC/MS system must be inspected for malfunctions, and corrections made as required.

10.8.6.2 If the area response for any internal standard changes by more than ±40 percent between the sample and the most recent valid calibration, the GC/MS system must be inspected for malfunction and

corrections made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

10.8.6.3 If, after reanalysis, the area responses or the RTs for all internal standards are inside the control limits, then the problem with the first analysis is considered to have been within the control of the Laboratory. Therefore, submit only data from the analysis with SICPs within the limits. This is considered the initial analysis and should be reported as such on all data deliverables.

11. Requirements for Demonstrating Method Acceptability for VOC Analysis from Canisters

11.1 Introduction

11.1.1 There are three performance criteria which must be met for a system to qualify under Compendium Method TO-15. These criteria are: the method detection limit of ≤0.5 ppbv, replicate precision within 25 percent, and audit accuracy within 30 percent for concentrations normally expected in contaminated ambient air (0.5 to 25 ppbv).

11.1.2 Either SIM or SCAN modes of operation can be used to achieve these criteria, and the choice of mode will depend on the number of target compounds, the decision of whether or not to determine tentatively identified compounds along with other VOCs on the target list, as well as on the analytical system characteristics.

11.1.3 Specific criteria for each Title III compound on the target compound list must be met by the analytical system. These criteria were established by examining summary data from EPA's Toxics Air Monitoring System Network and the Urban Air Toxics Monitoring Program network. Details for the determination of each of the criteria follow.

11.2 Method Detection Limit

11.2.1 The procedure chosen to define the method detection limit is that given in the *Code of Federal Regulations* (40 CFR 136 Appendix B).

11.2.2 The method detection limit is defined for each system by making seven replicate measurements of the compound of interest at a concentration near (within a factor of five) the expected detection limit, computing the standard deviation for the seven replicate concentrations, and multiplying this value by 3.14 (i.e., the Student's t value for 99 percent confidence for seven values). Employing this approach, the detection limits given in Table 4 were obtained for some of the VOCs of interest.

11.3 Replicate Precision

11.3.1 The measure of replicate precision used for this program is the absolute value of the difference between replicate measurements of the sample divided by the average value and expressed as a percentage as follows:

$$\text{percent difference} = \frac{|x_1 - x_2|}{\bar{x}} \times 100$$

where: x_1 = First measurement value.

x_2 = Second measurement value.

$\bar{x}$ = Average of the two values.

11.3.2 There are several factors which may affect the precision of the measurement. The nature of the compound of interest itself such as molecular weight, water solubility, polarizability, etc., each have some effect on the precision, for a given sampling and analytical system. For example, styrene, which is classified as a polar VOC, generally shows slightly poorer precision than the bulk of nonpolar VOCs. A primary influence on precision is the concentration level of the compound of interest in the sample, i.e., the precision degrades as the concentration approaches the detection limit. A conservative measure was obtained from replicate analysis of "real world" canister samples from the TAMS and UATMP networks. These data are summarized in Table 5 and suggest that a replicate precision value of 25 percent can be achieved for each of the target compounds.

11.4 Audit Accuracy

11.4.1 A measure of analytical accuracy is the degree of agreement with audit standards. Audit accuracy is defined as the difference between the nominal concentration of the audit compound and the measured value divided by the audit value and expressed as a percentage, as illustrated in the following equation:

$$\text{Audit Accuracy, \%} = \frac{\text{Spiked Value} - \text{Observed Value}}{\text{Spiked Value}} \times 100$$

11.4.2 Audit accuracy results for TAMS and UATMP analyses are summarized in Table 6 and were used to form the basis for a selection of 30 percent as the performance criterion for audit accuracy.

12. References

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APPENDIX A.

LISTING OF SOME COMMERCIAL WATER MANAGEMENT SYSTEMS USED WITH AUTOGC SYSTEMS

Tekmar Dohrman Company
7143 East Kemper Road
Post Office Box 429576
Cincinnati, Ohio 45242-9576
(513) 247-7000
(513) 247-7050 (Fax)
(800) 543-4461
[Moisture control module]

Entech Laboratory Automation
950 Enchanted Way No. 101
Simi Valley, California 93065
(805) 527-5939
(805) 527-5687 (Fax)
[Microscale Purge and Trap]

Dynatherm Analytical Instruments
Post Office Box 159
Kelton, Pennsylvania 19346
(215) 869-8702
(215) 869-3885 (Fax)
[Thermal Desorption System]

XonTech Inc.
6862 Hayenhurst Avenue
Van Nuys, CA 91406
(818) 787-7380
(818) 787-4275 (Fax)
[Multi-adsorbent trap/dry purge]

Graseby
500 Technology Ct.
Smyrna, Georgia 30082
(770) 319-9999
(770) 319-0336 (Fax)
(800) 241-6898
[Controlled Desorption Trap]

Varian Chromatography System
2700 Mitchell Drive
Walnut Creek, California 94898
(510) 945-2196
(510) 945-2335 (FAX)
[Variable Temperature Adsorption Trap]

APPENDIX B.

COMMENT ON CANISTER CLEANING PROCEDURES

The canister cleaning procedures given in Section 8.4 require that canister pressure be reduced to <0.05mm Hg before the cleaning process is complete. Depending on the vacuum system design (diameter of connecting tubing, valve restrictions, etc.) and the placement of the vacuum gauge, the achievement of this value may take several hours. In any case, the pressure gauge should be placed near the canisters to determine pressure. The objective of requiring a low pressure evacuation during canister cleaning is to reduce contaminants. If canisters can be routinely certified (<0.2 ppbv for target compounds) while using a higher vacuum, then this criteria can be relaxed. However, the ultimate vacuum achieved during cleaning should always be <0.2mm Hg.

Canister cleaning as described in Section 8.4 and illustrated in Figure 10 requires components with special features. The vacuum gauge shown in Figure 10 must be capable of measuring 0.05mm Hg with less than a 20% error. The vacuum pump used for evacuating the canister must be noncontaminating while being capable of achieving the 0.05 mm Hg vacuum as monitored near the canisters. Thermoelectric vacuum gauges and turbomolecular drag pumps are typically being used for these two components.

An alternate to achieving the canister certification requirement of <0.2 ppbv for all target compounds is the criteria used in Compendium Method TO-12 that the total carbon count be <10ppbC. This check is less expensive and typically more exacting than the current certification requirement and can be used if proven to be equivalent to the original requirement. This equivalency must be established by comparing the total nonmethane organic carbon (TNMOC) expressed in ppbC to the requirement that individual target compounds be <0.2 ppbv for a series of analytical runs.

APPENDIX C.

LISTING OF COMMERCIAL MANUFACTURERS AND RE-SUPPLIERS OF SPECIALLY-PREPARED CANISTERS

BRC/Rasmussen
17010 NW Skyline Blvd.
Portland, Oregon 97321
(503) 621-1435

Meriter
1790 Potrero Drive
San Jose, CA 95124
(408) 265-6482

Restek Corporation
110 Benner Circle
Bellefonte, PA 16823-8812
(814) 353-1300
(800) 356-1688

Scientific Instrumentation Specialists
P.O. Box 8941
815 Courtney Street
Moscow, ID 83843
(208) 882-3860

Graseby
500 Technology Ct.
Smyrna, Georgia 30082
(404) 319-9999
(800) 241-6898

XonTech Inc.
6862 Hayenhurst Avenue
Van Nuys, CA 91406
(818) 787-7380

APPENDIX D.

LISTING OF COMMERCIAL SUPPLIERS OF PERMEATION TUBES AND SYSTEMS

Kin-Tek
504 Laurel St.
Lamarque, Texas 77568
(409) 938-3627
(800) 326-3627

Vici Metronics, Inc.
2991 Corvin Drive
Santa Clara, CA 95051
(408) 737-0550

Analytical Instrument Development, Inc.
Rt. 41 and Newark Rd.
Avondale, PA 19311
(215) 268-3181

Ecology Board, Inc.
9257 Independence Ave.
Chatsworth, CA 91311
(213) 882-6795

Tracor, Inc.
6500 Tracor Land
Austin, TX
(512) 926-2800

Metronics Associates, Inc.
3201 Porter Drive
Standford Industrial Park
Palo Alto, CA 94304
(415) 493-5632

TABLE 1. VOLATILE ORGANIC COMPOUNDS ON THE TITLE III CLEAN AIR AMENDMENT LIST--MEMBERSHIP IN COMPENDIUM METHOD TO-14A LIST AND THE SOW-CLP LIST OF VOCs

Compound	CAS No.	BP (°C)	v.p. (mmHg)[1]	MW[1]	TO-14A	CLP-SOW
Methyl chloride (chloromethane); CH3Cl	74-87-3	-23.7	3.8 x 10	50.5	X	X
Carbonyl sulfide; COS	463-58-1	-50.0	3.7 x 10	60.1		
Vinyl chloride (chloroethene); C2H3Cl	75-01-4	-14.0	3.2 x 10	62.5	X	X
Diazomethane; CH2N2	334-88-3	-23.0	2.8 x 10	42.1		
Formaldehyde; CH2O	50-00-0	-19.5	2.7 x 10	30		
1,3-Butadiene; C4H6	106-99-0	-4.5	2.0 x 10	54		X
Methyl bromide (bromomethane); CH3Br	74-83-9	3.6	1.8 x 10	94.9	X	X
Phosgene; CCl2O	75-44-5	8.2	1.2 x 10	99		
Vinyl bromide (bromoethene); C2H3Br	593-60-2	15.8	l.l x 10	107		
Ethylene oxide; C2H4O	75-21-8	10.7	1.1 x 10	44		
Ethyl chloride (chloroethane); C2H5Cl	75-00-3	12.5	1.0 x 10	64.5	X	X
Acetaldehyde (ethanal); C2H4O	75-07-0	21.0	952	44		
Vinylidene chloride (1,1-dichloroethylene); C2H2Cl2	75-35-4	31.7	500	97	X	X
Propylene oxide; C3H6O	75-56-9	34.2	445	58		
Methyl iodide (iodomethane); CH3I	74-88-4	42.4	400	141.9		
Methylene chloride; CH2Cl2	75-09-2	40.0	349	84.9	X	X
Methyl isocyanate; C2H3NO	624-83-9	59.6	348	57.1		
Allyl chloride (3-chloropropene); C3H5Cl	107-05-1	44.5	340	76.5	X	X
Carbon disulfide; CS2	75-15-0	46.5	260	76		
Methyl tert-butyl ether; C5H12O	1634-04-4	55.2	249	86		
Propionaldehyde; C2H5CHO	123-38-6	49.0	235	58.1		
Ethylidene dichloride (1,1-dichloroethane); C2H4Cl2	75-34-3	57.0	230	99	X	

TABLE 1. (continued)

Compound	CAS No.	BP (°C)	v.p. (mmHg)[1]	MW[1]	TO-14A	CLP-SOW
Chloroprene (2-chloro-1,3-butadiene); C4H5Cl	126-99-8	59.4	226	88.5		
Chloromethyl methyl ether; C2H5ClO	107-30-2	59.0	224	80.5		
Acrolein (2-propenal); C3H4O	107-02-8	52.5	220	56		X
1,2-Epoxybutane (1,2-butylene oxide); C4H8O	106-88-7	63.0	163	72		
Chloroform; CHCl3	67-66-3	61.2	160	119	X	X
Ethyleneimine (aziridine); C2H5N	151-56-4	56	160.0	43		
1,1-Dimethylhydrazine; C2H8N2	57-14-7	63	157.0	60.0		
Hexane; C6H14	110-54-3	69.0	120	86.2	X	
1,2-Propyleneimine (2-methylaziridine); C3H7N	75-55-8	66.0	112	57.1		
Acrylonitrile (2-propenenitrile); C3H3N	107-13-1	77.3	100	53	X	
Methyl chloroform (1,1,1-trichloroethane); C2H3Cl3	71-55-6	74.1	100	133.4	X	X
Methanol; CH4O	67-56-1	65.0	92.0	32		X
Carbon tetrachloride; CCl4	56-23-5	76.7	90.0	153.8	X	X
Vinyl acetate; C4H6O2	108-05-4	72.2	83.0	86		X
Methyl ethyl ketone (2-butanone); C4H8O	78-93-3	79.6	77.5	72		X
Benzene; C6H6	71-43-2	80.1	76.0	78	X	X
Acetonitrile (cyanomethane); C2H3N	75-05-8	82	74.0	41.0		X
Ethylene dichloride (1,2-dichloroethane); C2H4Cl2	107-06-2	83.5	61.5	99	X	X
Triethylamine; C6H15N	121-44-8	89.5	54.0	101.2		
Methylhydrazine; CH6N2	60-34-4	87. 8	49.6	46.1		
Propylene dichloride (1,2-dichloropropane); C3H6Cl2	78-87-5	97.0	42.0	113	X	X
2,2,4-Trimethyl pentane C8H18	540-84-l	99.2	40.6	114		
1,4-Dioxane (1,4-Diethylene oxide); C4H8O2	123-91-1	101	37.0	88		
Bis(chloromethyl) ether; C2H4Cl2O	542-88-1	104	30.0	115		
Ethyl acrylate; C5H8O2	140-88-5	100	29.3	100		
Methyl methacrylate; C5H8O2	80-62-6	101	28.0	100.1		

TABLE 1. (continued)

Compound	CAS No.	BP (°C)	v.p. (mmgHg)[1]	MW[1]	TO-14A	CLP-SOW
Methyl methacrylate; C5H8O2	80-62-101	101	28.0	100.1		
1,3-Dichloropropene; C3H4Cl2 (cis)	542-75-6	112	27.8	111	X	X
Toluene; C7H8	108-88-3	111	22.0	92	X	X
Trichloroethylene; C2HCl3	79-01-6	87.0	20.0	131.4	X	X
1,1,2-Trichloroethane; C2H3Cl3	79-00-5	114	19.0	133.4	X	X
Tetrachloroethylene; C2Cl4	127-18-4	121	14.0	165.8	X	X
Epichlorohydrin (1-chloro-2,3-epoxy propane); C3H5ClO	106-89-8	117	12.0	92.5		
Ethylene dibromide (1,2-dibromoethane); C2H4Br2	106-93-4	132	11.0	187.9	X	X
N-Nitroso-N-methylurea; C2H5N3O2	684-93-5	124	10.0	103		
2-Nitropropane; C3H7NO2	79-46-9	120	10.0	89		
Chlorobenzene; C6H5Cl	108-90-7	132	8.8	112.6	X	X
Ethylbenzene; C8H10	100-41-4	136	7.0	106	X	X
Xylenes (isomer & mixtures); C8H10	1330-20-7	142	6.7	106.2	X	X
Styrene; C8H8	100-42-5	145	6.6	104	X	X
p-Xylene; C8H10	106-42-3	138	6.5	106.2	X	X
m-Xylene; C8H10	108-38-3	139	6.0	106.2	X	X
Methyl isobutyl ketone (hexone); C6H12O	108-10-1	117	6.0	100.2		
Bromoform (tribromomethane); CHBr3	75-25-2	149	5.6	252.8		
l,l,2,2-Tetrachloroethane; C2H2Cl4	79-34-5	146	5.0	167.9	X	X
o-Xylene; C8H10	95-47-6	144	5.0	106.2	X	X
Dimethylcarbamyl chloride; C3H6ClNO	79-44-7	166	4.9	107.6		
N-Nitrosodimethylamine; C2H6N2O	62-75-9	152	3.7	74		
Beta-Propiolactone; C3H4O2	57-57-8	Decomposes at 162	3.4	72		
Cumene (isopropylbenzene); C9Hl2	98-82-8	153	3.2	120		

[1] 1

TABLE 1. (continued)

Compound	CAS No.	BP (°C)	v.p. (mmHg)[1]	MW[1]	TO-14A	CLP-SOW
Cumene (isopropylbenzene); C9H12	98-82-8	153	3.2	120		
Acrylic acid; C3H4O2	79-10-7	141	3.2	72		
N,N-Dimethylformamide; C3H7NO	68-12-2	153	2.7	73		
1,3-Propane sultone; C3H6O3S	1120-71-4	180/30mm	2.0	122.1		
Acetophenone; C8H8O	98-86-2	202	1.0	120		
Dimethyl sulfate; C2H6O4S	77-78-1	188	1.0	126.1		
Benzyl chloride (a-chlorotoluene); C7H7Cl	100-44-7	179	1.0	126.6	X	X
1,2-Dibromo-3-chloropropane; C3H5Br2Cl	96-12-8	196	0.80	236.4		
Bis(2-Chloroethyl)ether; C4H8Cl2O	111-44-4	178	0.71	143		
Chloroacetic acid; C2H3ClO2	79-11-8	189	0.69	94.5		
Aniline (aminobenzene); C6H7N	62-53-3	184	0.67	93		
1,4-Dichlorobenzene (p-); C6H4Cl2	106-46-7	173	0.60	147	X	X
Ethyl carbamate (urethane); C3H7NO2	51-79-6	183	0.54	89		
Acrylamide; C3H5NO	79-06-1	125/25 mm	0.53	71		
N,N-Dimethylaniline; C8H11N	121-69-7	192	0.50	121		
Hexachloroethane; C2Cl6	67-72-1	Sublimes at 186	0.40	236.7		
Hexachlorobutadiene; C4Cl6	87-68-3	215	0.40	260.8	X	X
Isophorone; C9H14O	78-59-1	215	0.38	138.2		
N-Nitrosomorpholine; C4H8N2O2	59-89-2	225	0.32	116.1		
Styrene oxide; C8H8O	96-09-3	194	0.30	120.2		
Diethyl sulfate; C4H10O4S	64-67-5	208	0.29	154		
Cresylic acid (cresol isomer mixture);C7H8O	1319-77-3	202	0.26	108		
o-Cresol; C7H8O	95-48-7	191	0.24	108		
Catechol (o-hydroxyphenol); C6H6O2	120-80-9	240	0.22	110		
Phenol; C6H6O	108-95-2	182	0.20	94		

TABLE 1. (continued)

Compound	CAS No.	BP (°C)	v.p. (mmHg)[1]	MW[1]	TO-14A	CLP-SOW
Catechol (o-hydroxyphenol); C6H6O2	120-80-9	240	0.22	110		
Phenol; C6H6O	108-95-2	182	0.20	94		
1,2,4-Trichlorobenzene; C6H3Cl3	120-82-1	213	0.18	181.5	X	X
nitrobenzene; C6H5NO2	98-95-3	211	0.15	123		

[1]Vapor pressure (v.p.), boiling point (BP) and molecularweight (MW) data from:
(a)D. L. Jones and J. bursey, "Simultaneous Control of PM-10 and Hazardous Air Pollutants II: Rationale for Selection of Hazardous Air Pollutants as Potential Particulate Matter," Report EPA-452/R-93/013, U. S. Environmental Protection Agency, Research Triangle Park, NC. October 1992;
(b)R. C. Weber, P. A. Parker, and M. Bowser. Vapor Pressure Distribution of Selected Organic Chemicals, Report EPA-600/2-81-021, U. S. Environmental Protection Agency, Cincinnati, OH, February 1981; and
(c)R. C. Weast, ed., "CRC Handbook of Chemistry and Physics," 59th edition, CRC Press, Boca Raton, 1979.

TABLE 2. CHARACTERISTIC MASSES (M/Z) USED FOR QUANTIFYING THE TITLE III CLEAN AIR ACT AMENDMENT COMPOUNDS

Compound	CAS No.	Primary Ion	Secondary Ion
Methyl chloride (chloromethane); CH3Cl	74-87-3	50	52
Carbonyl sulfide; COS	463-S8-1	60	62
Vinyl chloride (chloroethene); C2H3Cl	7S-01-4	62	64
Diazomethane; CH2N2	334-88-3	42	41
Formaldehyde; CH2O	50-00-0	29	30
1,3-Butadiene; C4H6	106-99-0	39	54
Methyl bromide (bromomethane); CH3Br	74-83-9	94	96
Phosgene; CCl2O	75-44-5	63	65
Vinyl bromide (bromoethene); C2H3Br	593-60-2	106	108
Ethylene oxide; C2H4O	75-21-8	29	44
Ethyl chloride (chloroethane); C2H5Cl	75-00-3	64	66
Acetaldehyde (ethanal); C2H4O	75-07-0	44	29, 43
Vinylidene chloride (1,1-dichloroethylene); C2H2Cl2	75-35-4	61	96
Propylene oxide; C3H6O	75-56-9	58	57
Methyl iodide (iodomethane); CH3I	74-88-4	142	127
Methylene chloride; CH2Cl2	75-09-2	49	84, 86
Methyl isocyanate; C2H3NO	624-83-9	57	56
Allyl chloride (3-chloropropene); C3H5Cl	107-05-1	76	41, 78
Carbon disulfide; CS2	75-15-0	76	44, 78
Methyl tert-butyl ether; C5H12O	1634-04-4	73	41, 53
Propionaldehyde; C2H5CHO	123-38-6	58	29, 57
Ethylidene dichloride (1,1-dichloroethane); C2H4Cl2	75-34-3	63	65, 27
Chloroprene (2-chloro-1,3-butadiene); C4H5Cl	126-99-8	88	53, 90
Chloromethyl methyl ether; C2H5ClO	107-30-2	45	29, 49
Acrolein (2-propenal); C3H4O	107-02-8	56	55
1,2-Epoxybutane (1,2-butylene oxide); C4H8O	106-88-7	42	41, 72
Chloroform; CHCl3	67-66-3	83	85, 47
Ethyleneimine (aziridine); C2H5N	151-56-4	42	43
1,1-Dimethylhydrazine; C2H8N2	57-14-7	60	45, 59
Hexane; C6H14	110-54-3	57	41, 43
1,2-Propyleneimine (2-methylazindine); C3H7N	75-55-8	56	57, 42
Acrylonitrile (2-propenenitrile); C3H3N	107-13-1	53	52
Methyl chloroform (1,1,1 trichloroethane); C2H3Cl3	71-55-6	97	99, 61
Methanol; CH4O	67-56-1	31	29
Carbon tetrachloride; CCl4	56-23-5	117	119
Vinyl acetate; C4H6O2	108-05-4	43	86
Methyl ethyl ketone (2-butanone); C4H8O	78-93-3	43	72

TABLE 2. (continued)

Compound	CAS No.	Primary Ion	Secondary Ion
Benzene; C6H6	71-43-2	78	77,50
Acetonitrile (cyanomethane); C2H3N	75-05-8	41	40
Ethylene dichloride (1,2-dichloroethane); C2H4Cl2	107-06-2	62	64, 27
Triethylamine; C6H15N	121-44-8	86	58, 101
Methylhydrazine; CH6N2	60-34-4	46	31, 45
Propylene dichloride (1,2-dichloropropane); C3H6Cl2	78-87-5	63	41, 62
2,2,4-Trimethyl pentane; C8H18	540-84-1	57	41, 56
1,4-Dioxane (1,4 Diethylene oxide); C4H8O2	123-91-1	88	58
Bis(chloromethyl) ether; C2H4Cl2O	542-88-1	79	49, 81
Ethyl acrylate; C5H8O2	140-88-5	55	73
Methyl methacrylate; C5H8O2	80-62-6	41	69, 100
1,3-Dichloropropene; C3H4Cl2 (cis)	542-75-6	75	39, 77
Toluene; C7H8	108-88-3	91	92
Trichloethylene; C2HCl3	79-01-6	130	132, 95
1,1,2-Trichloroethane; C2H3Cl3	79-00-5	97	83, 61
Tetrachloroethylene; C2Cl4	127-18-4	166	164, 131
Epichlorohydrin (l-chloro-2,3-epoxy propane); C3H5ClO	106-89-8	57	49, 62
Ethylene dibromide (1,2-dibromoethane); C2H4Br2	106-93-4	107	109
N-Nitrso-N-methylurea; C2H5N3O2	684-93-5	60	44, 103
2-Nitropropane; C3H7NO2	79-46-9	43	41
Chlorobenzene; C6H5Cl	108-90-7	112	77, 114
Ethylbenzene; C8H10	100-41-4	91	106
Xylenes (isomer & mixtures); C8H10	1330-20-7	91	106
Styrene; C8H8	100-42-5	104	78, 103
p-Xylene; C8H10	106-42-3	91	106
m-Xylene; C8H10	108-38-3	91	106
Methyl isobutyl ketone (hexone); C6H12O	108-10-1	43	58, 100
Bromoform (tribromomethane); CHBr3	75-25-2	173	171, 175
1,1,2,2-Tetrachloroethane; C2H2Cl4	79-34-5	83	85
o-Xylene; C8H10	95-47-6	91	106
Dimethylcarbamyl chloride; C3H6ClNO	79-44-7	72	107
N-Nitrosodimethylamine; C2H6N2O	62-75-9	74	42
Beta-Propiolactone; C3H4O2	57-57-8	42	43
Cumene (isopropylbenzene); C9H12	98-82-8	105	120
Acrylic acid; C3H4O2	79-10-7	72	45, 55
N,N-Dimethylformamide; C3H7NO	68-12-2	73	42, 44
1,3-Propane sultone; C3H6O3S	1120-71-4	58	65, 122

TABLE 2. (continued)

Compound	CAS No.	Primary Ion	Secondary Ion
Acetophenone; C8H8O	98-86-2	105	77,120
Dimethyl sulfate; C2H6O4S	77-78-1	95	66,96
Benzyl chloride (a-chlorotoluene); C7H7Cl	100-44-7	91	126
1,2-Dibromo-3-chloropropane; C3H5Br2Cl	96-12-8	57	155, 157
Bis(2-Chloroethyl)ether; C4H8Cl2O	111-44-4	93	63, 95
Chloroacetic acid; C2H3ClO2	79-11-8	50	45, 60
Aniline (aminobenzene); C6H7N	62-53-3	93	66
1,4-Dichlorobenzene (p-); C6H4Cl2	106-46-7	146	148, 111
Ethyl carbamate (urethane); C3H7NO2	51-79-6	31	44, 62
Acrylamide; C3H5NO	79-06-1	44	55, 71
N,N-Dimethylaniline; C8H11N	121-69-7	120	77, 121
Hexachloroethane; C2Cl6	67-72-1	201	199, 203
Hexachlorobutadiene; C4Cl6	87-68-3	225	227, 223
Isophorone; C9H14O	78-59-1	82	138
N-Nitrosomorpholine; C4H8N2O2	59-89-2	56	86, 116
Styrene oxide; C8H8O	96-09-3	91	120
Diethyl sulfate; C4H10O4S	64-67-5	45	59, 139
Cresylic acid (cresol isomer mixture); C7H8O	1319-77-3		
o-Cresol; C7H8O	95-48-7	108	107
Catechol (o-hydroxyphenol); C6H6O2	120-80-9	110	64
Phenol; C6H6O	108-95-2	94	66
1,2,4-Trichlorobenzene; C6H3Cl3	120-82-1	180	182, 184
Nitrobenzene; C6H5NO2	98-95-3	77	51, 123

TABLE 3. REQUIRED BFB KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria[1]
50	8.0 to 40.0 Percent of m/e 95
75	30.0 to 66.0 Percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/e 95 (See note)
173	Less than 2.0 Percent of m/e 174
174	50.0 to 120.0 Percent of m/e 95
175	4.0 to 9.0 Percent of m/e 174
176	93.0 to 101.0 Percent of m/e 174
177	5.0 to 9.0 Percent of m/e 176

[1]All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

TABLE 4. METHOD DETECTION LIMITS (MDL)[1]

TO-14A List	Lab #1, SCAN	Lab #2, SIM
Benzene	0.34	0.29
Benzyl Chloride	--	--
Carbon tetrachloride	0.42	0.15
Chlorobenzene	0.34	0.02
Chloroform	0.25	0.07
1,3-Dichlorobenzene	0.36	0.07
1,2-Dibromoethane	--	0.05
1,4-Dichlorobenzene	0.70	0.12
1,2-Dichlorobenzene	0.44	--
1,1-Dichloroethane	0.27	0.05
1,2-Dichloroethane	0.24	--
1,1-Dichloroethene	--	0.22
cis-1,2-Dichloroethene	--	0.06
Methylene chloride	1.38	0.84
1,2-Dichloropropane	0.21	--
cis-1,3-Dichloropropene	0.36	--
trans-1,3-Dichloropropene	0.22	--
Ethylbenzene	0.27	0.05
Chloroethane	0.19	--
Trichlorofluoromethane	--	--
1,1,2-Trichloro-1,2,2-trifluoroethane		--
1,2-Dichloro-1,1,2,2-tetrafluoroethane	--	--
Dichlorodifluoromethane	--	--
Hexachlorobutadiene	--	--
Bromomethane	0.53	--
Chloromethane	0.40	--
Styrene	1.64	0.06
1,1,2,2-Tetrachloroethane	0.28	0.09
Tetrachloroethene	0.75	0.10
Toluene	0.99	0.20
1,2,4-Trichlorobenzene	--	--
1,1,1-Trichloroethane	0.62	0.21
1,1,2-Trichloroethane	0.50	--
Trichloroethene	0.45	0.07
1,2,4-Trimethylbenzene	--	--
1,3,5-Trimethylbenzene	--	--
Vinyl Chloride	0.33	0.48
m,p-Xylene	0.76	0.08
o-Xylene	0.57	0.28

[1]Method Detection Limits (MDLs) are defined as the product of the standard deviation of seven replicate analyses and the student's "t" test value for 99% confidence. For Lab #2, the MDLs represent an average over four studies. MDLs are for MS/SCAN for Lab #1 and for MS/SIM for Lab #2.

TABLE 5. SUMMARY OF EPA DATA ON REPLICATE PRECISION (RP) FROM EPA NETWORK OPERATIONS[1]

Monitoring Compound Identification	EPA's Urban Air Toxics Monitoring Program (UATMP)			EPA's Toxics Air Monitoring Stations (TAMS)		
	%RP	#	ppbv	%RP	#	ppbv
Dichlorodifluoromethane	--		--	13.9	47	0.9
Methylene chloride	16.3	07	4.3	19.4	47	0.6
1,2-Dichloroethane	36.2	31	1.6	--	--	--
1,1,1-Trichloroethane	14.1	44	1.0	10.6	47	2.0
Benzene	12.3	56	1.6	4.4	47	1.5
Trichloroethene	12.8	08	1.3	--	--	--
Toluene	14.7	76	3.1	3.4	47	3.1
Tetrachloroethene	36.2	12	0.8	--	--	--
Chlorobenzene	20.3	21	0.9	--	--	--
Ethylbenzene	14.6	32	0.7	5.4	47	0.5
m-Xylene	14.7	75	4.0	5.3	47	1.5
Styrene	22.8	59[2]	1.1	8.7	47	0.2[2]
o-Xylene	--		--	6.0	47	0.5
p-Xylene	--					
1,3-Dichlorobenzene	49.1	06	0.6	--	--	--
1,4-Dichlorobenzene	14.7	14	6.5	--	--	--

[1]Denotes the number of replicate or duplicate analysis used to generate the statistic. The replicate precision is defined as the mean ratio of absolute difference to the average value.
[2]Styrene and o-xylene coelute from the GC column used in UATMP. For the TAMS entries, both values were below detection limits for 18 of 47 replicates and were not included in the calculation.

TABLE 6. AUDIT ACCURACY (AA) VALUES[1] FOR SELECTED COMPENDIUM METHOD TO-14A COMPOUNDS

Selected Compounds From TO-14A List	FY-88 TAMS AA(%), N=30	FY-88 UATMP AA(%), N=3
Vinyl chloride	4.6	17.9
Bromomethane	--	6.4
Trichlorofluoromethane	6.4	--
Methylene chloride	8.6	31.4
Chloroform	--	4.2
1,2-Dichloroethane	6.8	11.4
1,1,1-Trichloroethane	18.6	11.3
Benzene	10.3	10.1
Carbon tetrachloride	12.4	9.4
1,2-Dichloropropane	--	6.2
Trichloroethene	8.8	5.2
Toluene	8.3	12.5
Tetrachloroethene	6.2	--
Chlorobenzene	10.5	11.7
Ethylbenzene	12.4	12.4
o-Xylene	16.2	21.2

[1]Audit accuracy is defined as the relative difference between the audit measurement result and its nominal value divided by the nominal value. N denotes the number of audits averaged to obtain the audit accuracy value. Information is not available for other TO-14A compounds because they were not present in the audit materials.

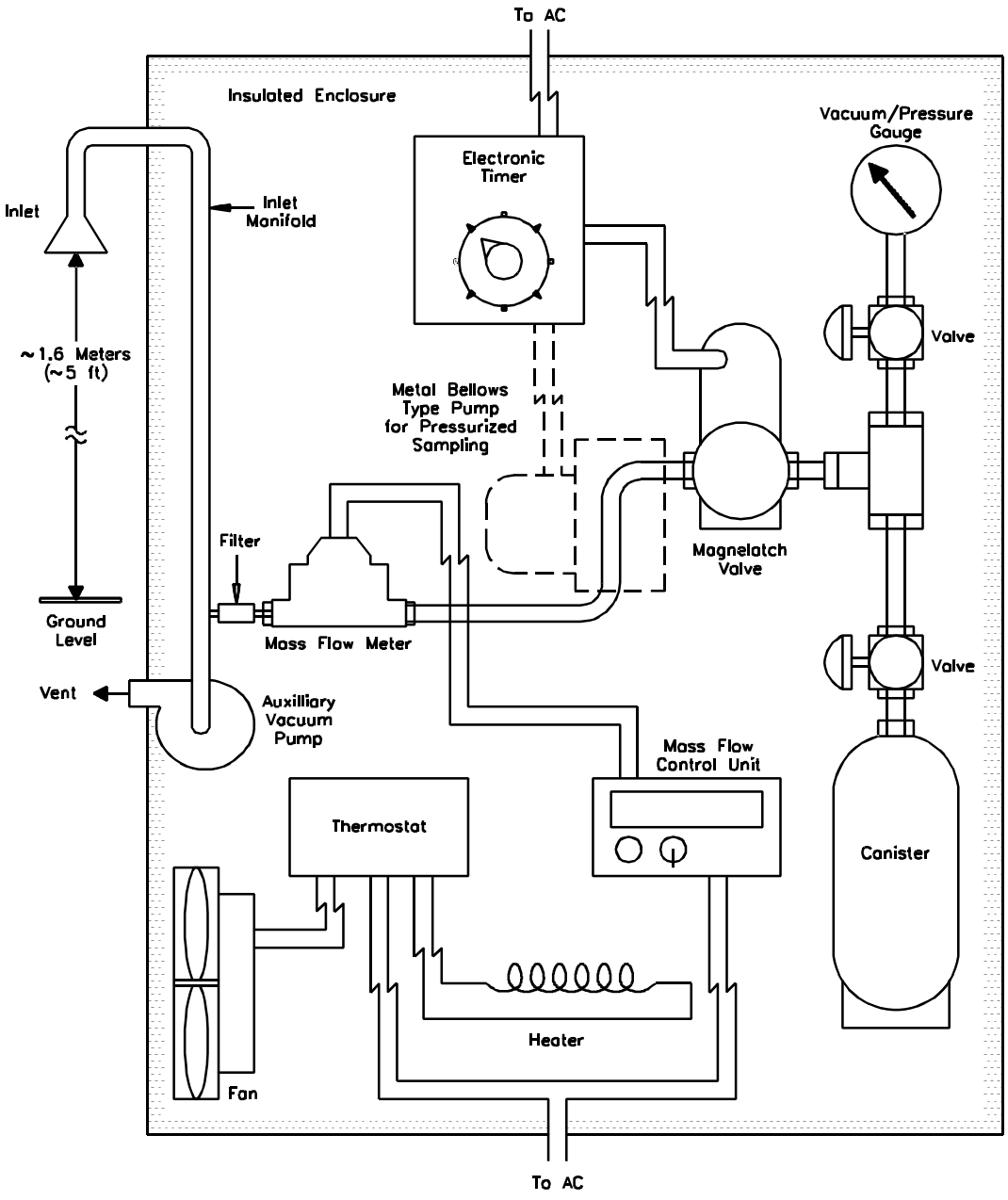
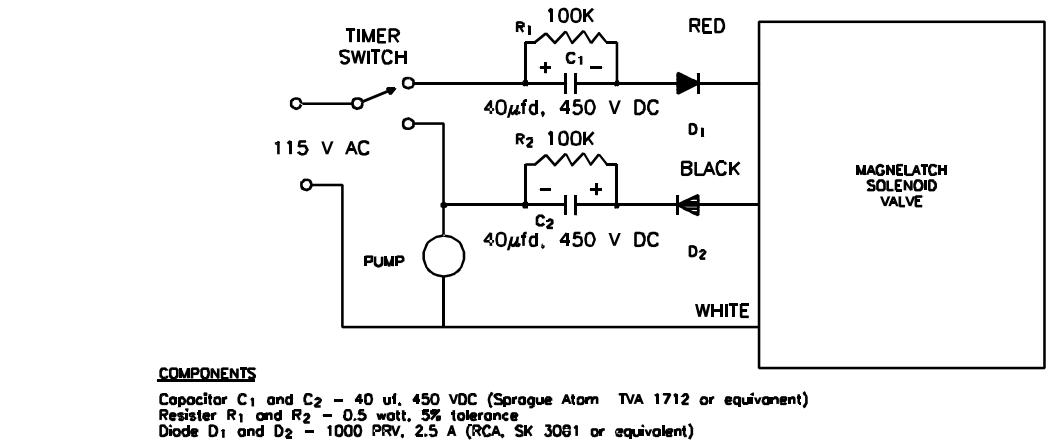
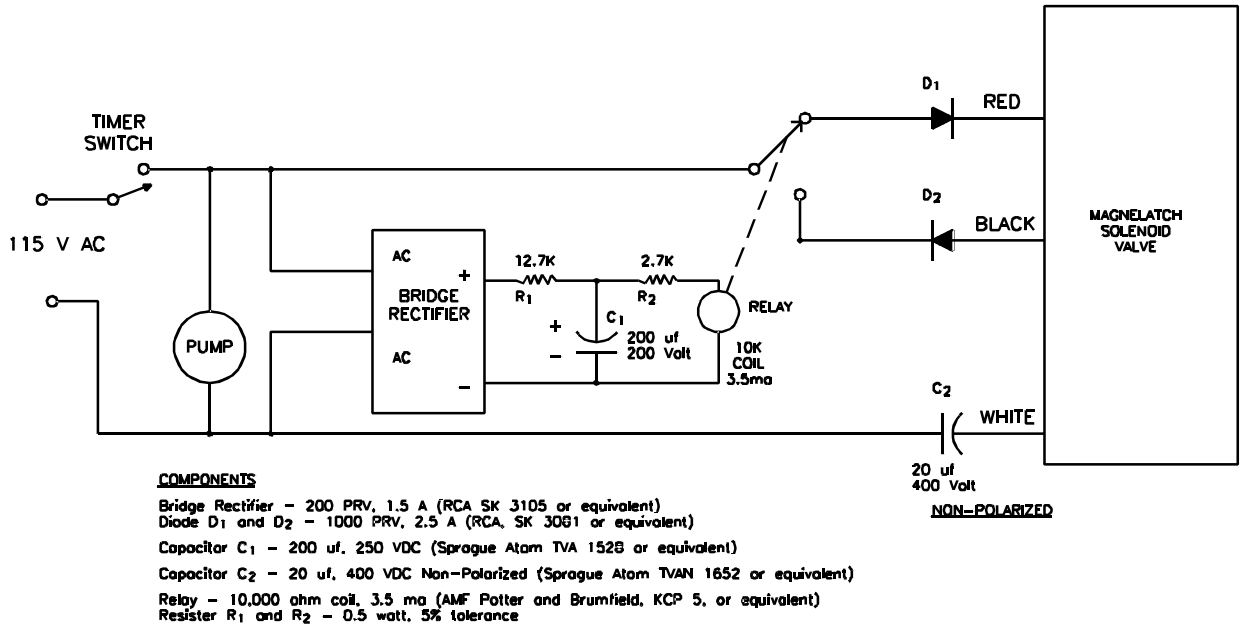


Figure 1. Sampler configuration for subatmospheric pressure or pressurized canister sampling.



(a). Simple Circuit for Operating Magnelatch Valve



(b). Improved Circuit Designed to Handle Power Interruptions

Figure 2. Electrical pulse circuits for driving Skinner magnelatch solenoid valve with mechanical timer.

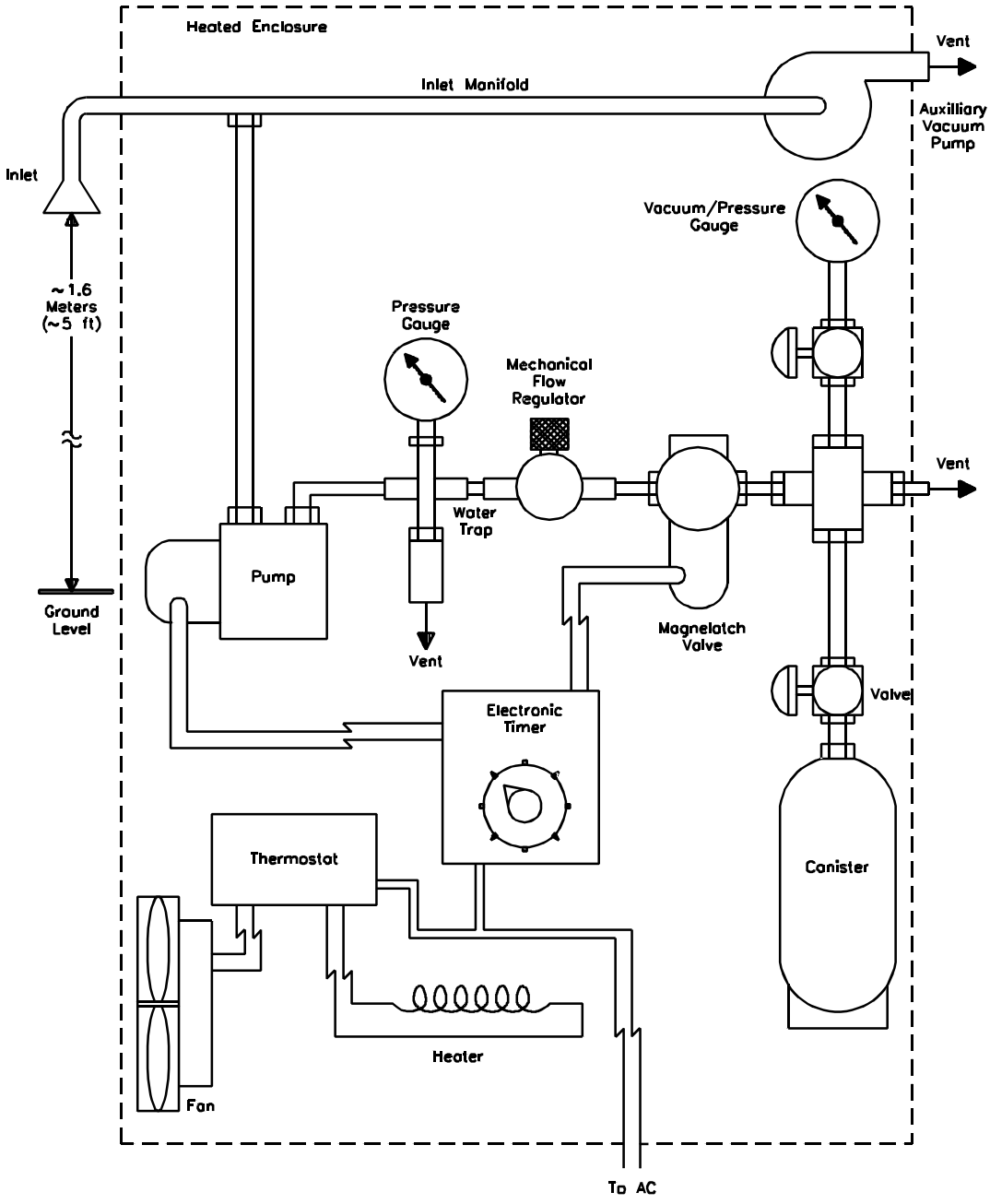


Figure 3. Alternative sampler configuration for pressurized canister sampling.

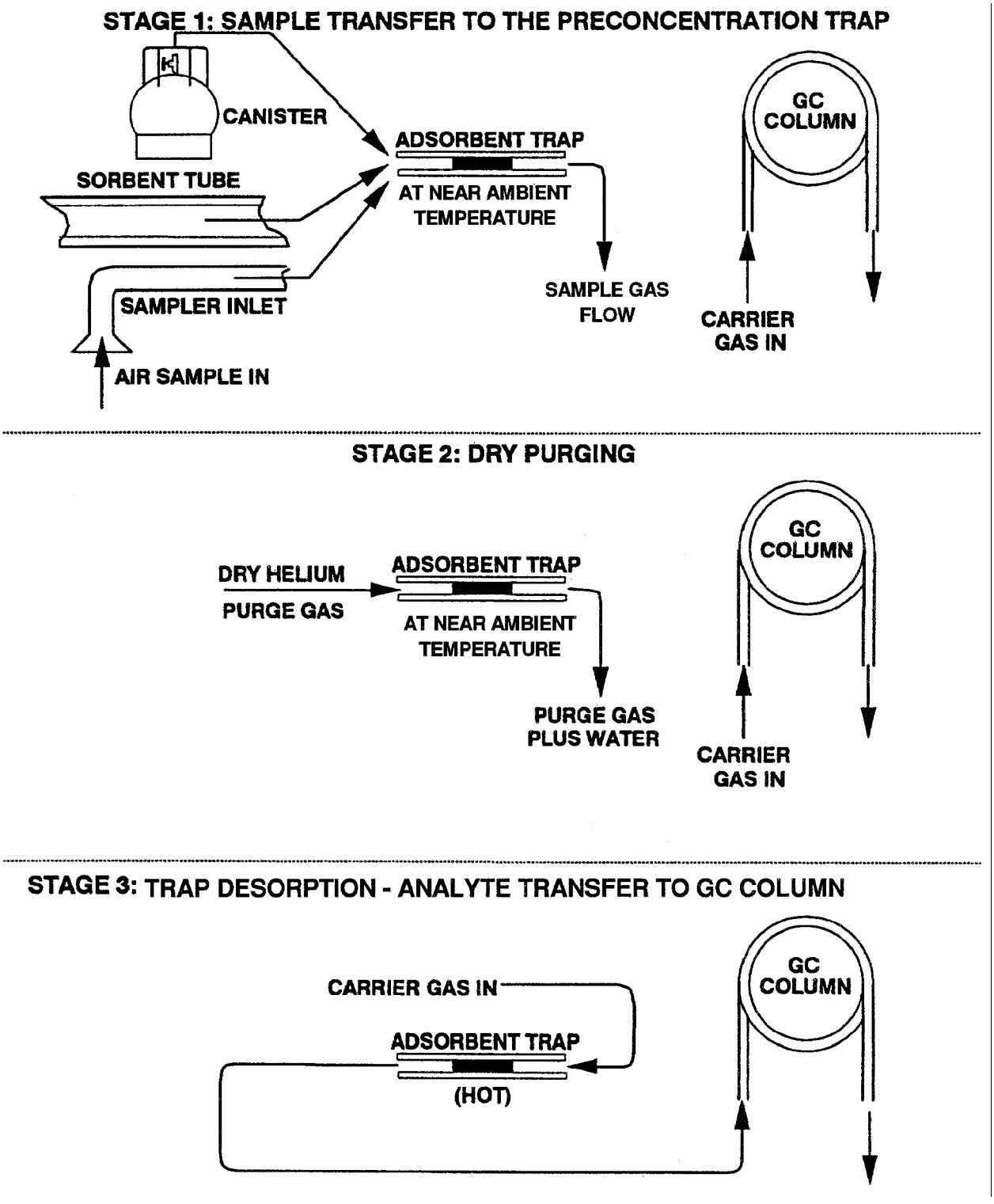


Figure 4. Illustration of three stages of dry purging of adsorbent trap.

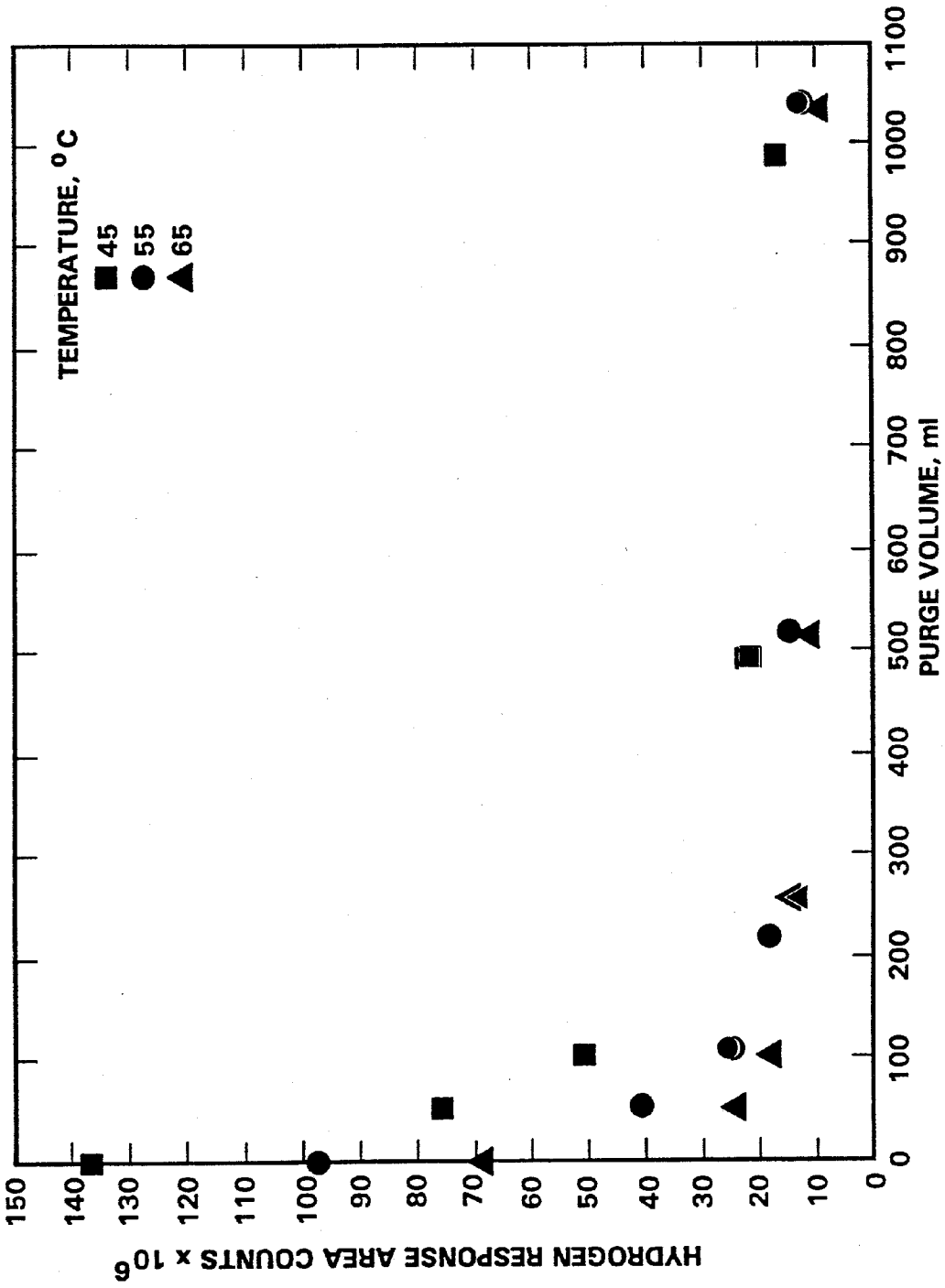


Figure 5. Residual water vapor on VOC concentrator vs. dry He purge volume.

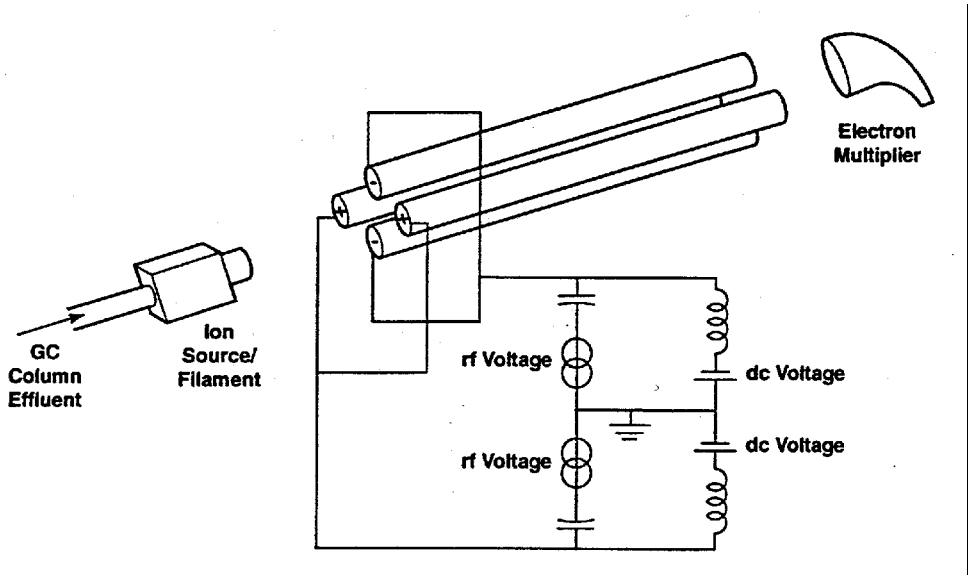


Figure 6. Simplified diagram of a quadrupole mass spectrometer.

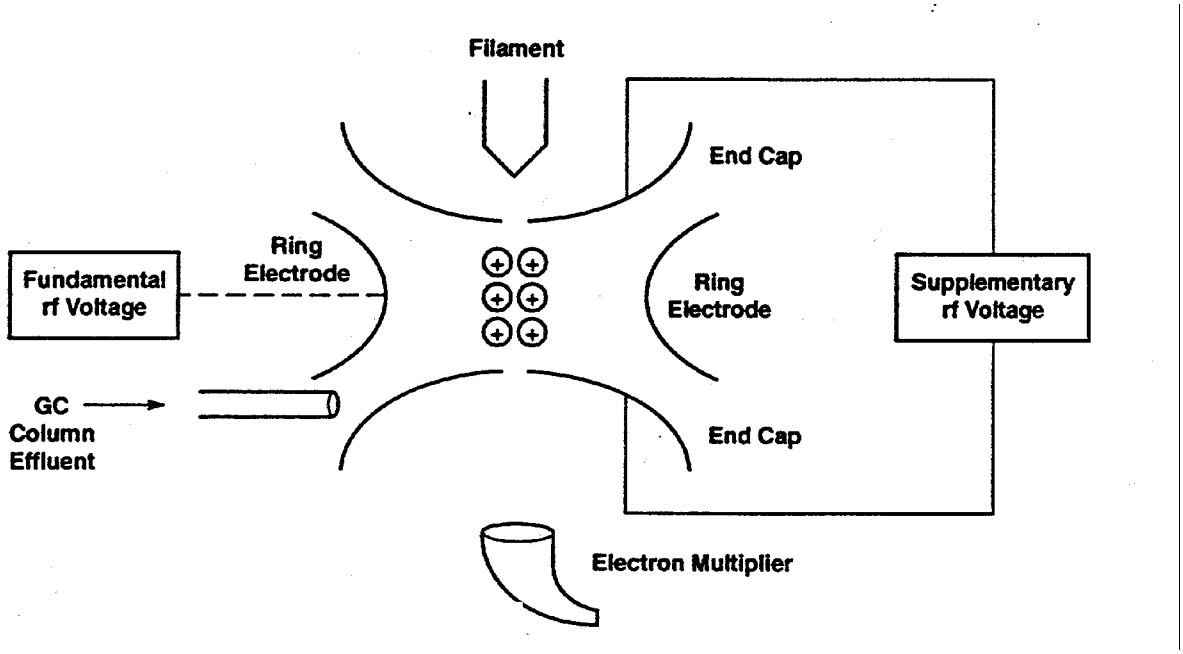


Figure 7. Simplified diagram of an ion trap mass spectrometer.

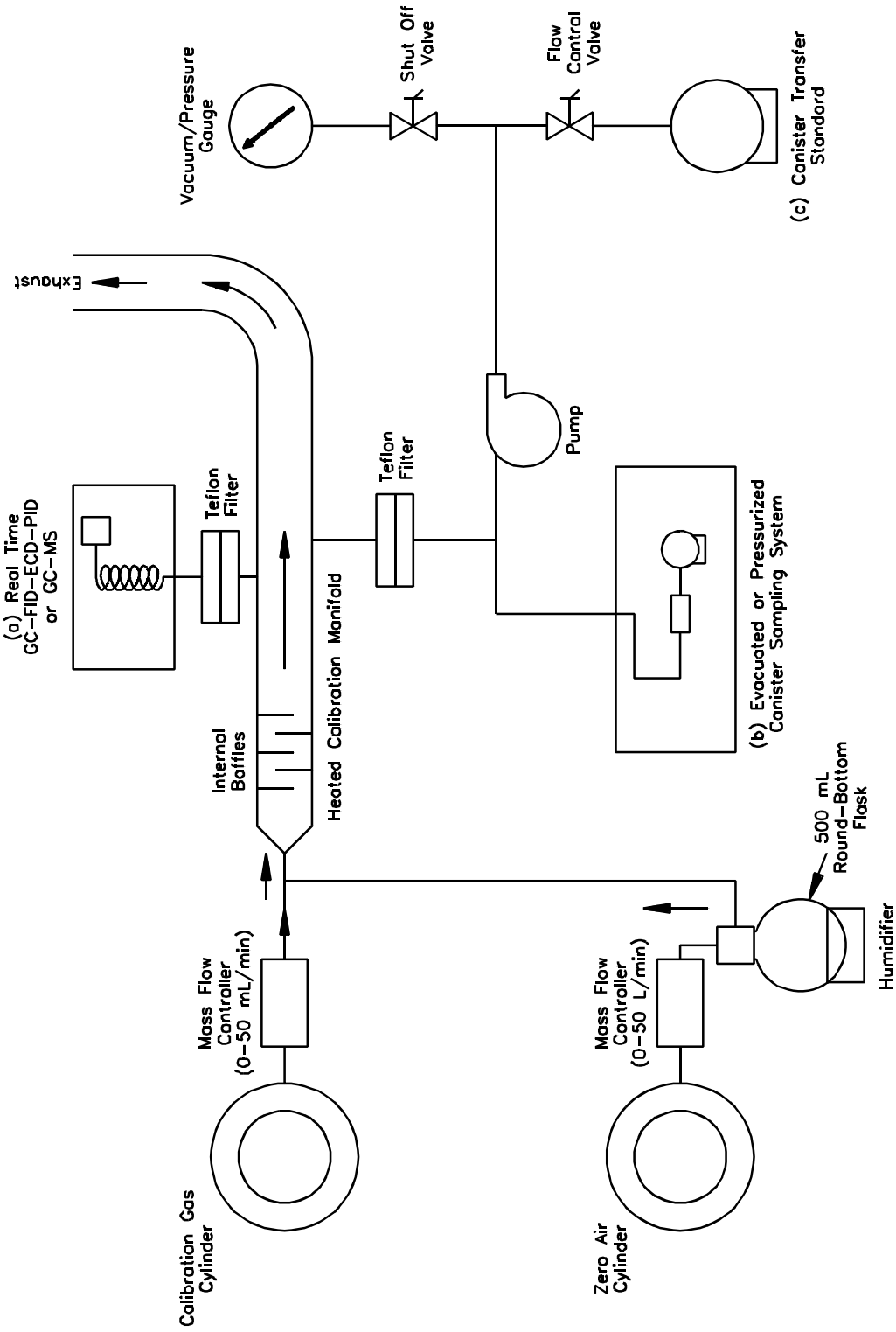


Figure 8. Schematic diagram of calibration system and manifold for (a) analytical system calibration, (b) testing canister sampling system and (c) preparing canister transfer standards.

COMPENDIUM METHOD TO-15
CANISTER SAMPLING FIELD TEST DATA SHEET

A.GENERAL INFORMATION

SITE LOCATION: _____
SITE ADDRESS: _____
_____
SAMPLING DATE: _____

SHIPPING DATE: _____
CANISTER SERIAL NO.: _____
SAMPLER ID: _____
OPERATOR: _____
CANISTER LEAK
CHECK DATE: _____

B. SAMPLING INFORMATION

	TEMPERATURE				PRESSURE	
	INTERIOR	AMBIENT	MAXIMUM	MINIMUM	CANISTER PRESSURE	
START						
STOP						

	SAMPLING TIMES		FLOW RATES		
	LOCAL TIME	ELAPSED TIME METER READING	MANIFOLD FLOW RATE	CANISTER FLOW RATE	FLOW CONTROLLER READOUT
START					
STOP					

SAMPLING SYSTEM CERTIFICATION DATE: _____
QUARTERLY RECERTIFICATION DATE: _____

C. LABORATORY INFORMATION

DATA RECEIVED: _____
RECEIVED BY: _____
INITIAL PRESSURE: _____
FINAL PRESSURE: _____
DILUTION FACTOR: _____
ANALYSIS
GC-FID-ECD DATE: _____
GC-MSD-SCAN DATE: _____
GC-MSD-SIM DATE: _____
RESULTS*: _____
_____
GC-FID-ECD: _____
GC-MSD-SCAN: _____
GC-MSD-SIM: _____

_____
SIGNATURE/TITLE

Figure 9. Canister sampling field test data sheet (FTDS).

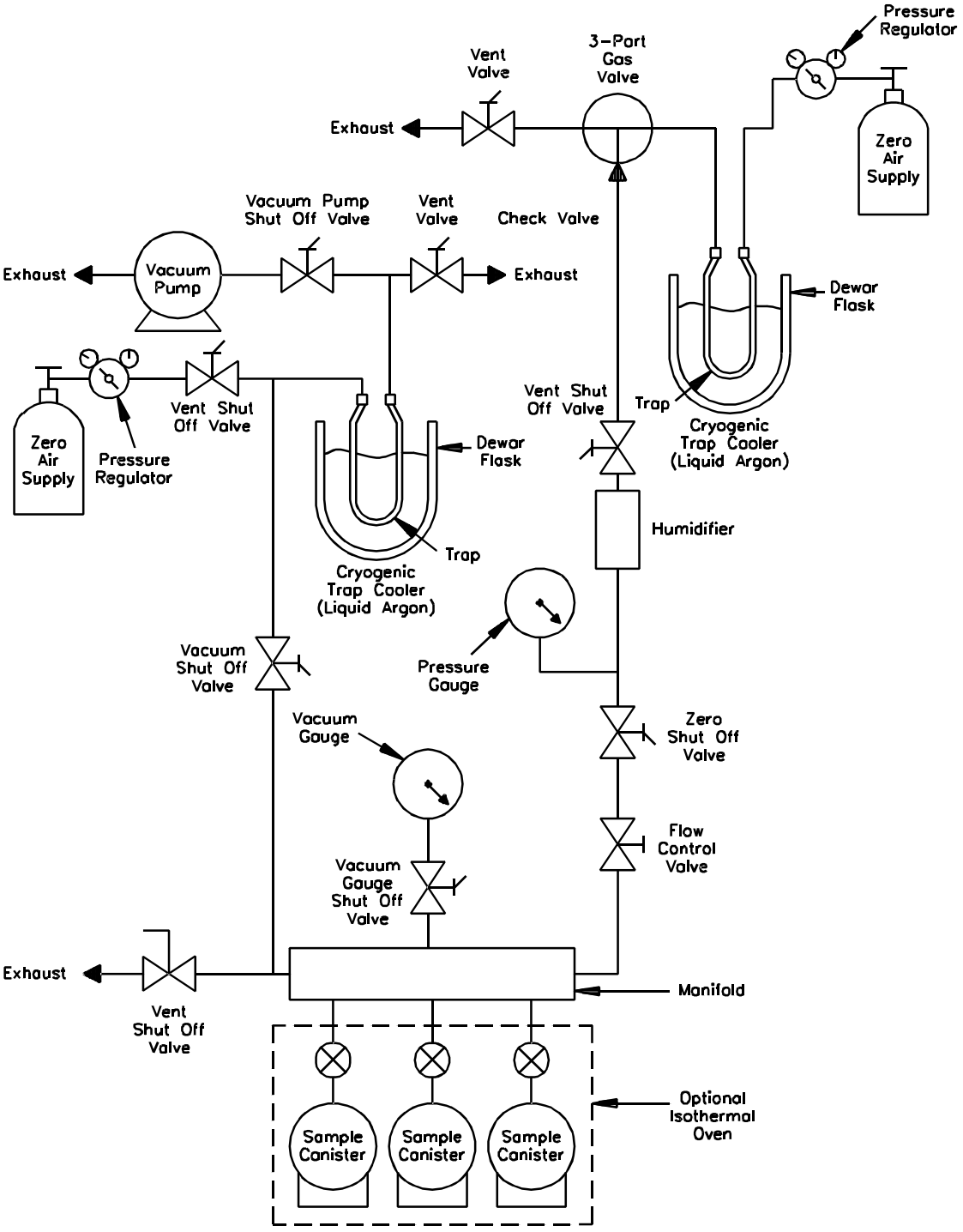


Figure 10. Canister cleaning system.

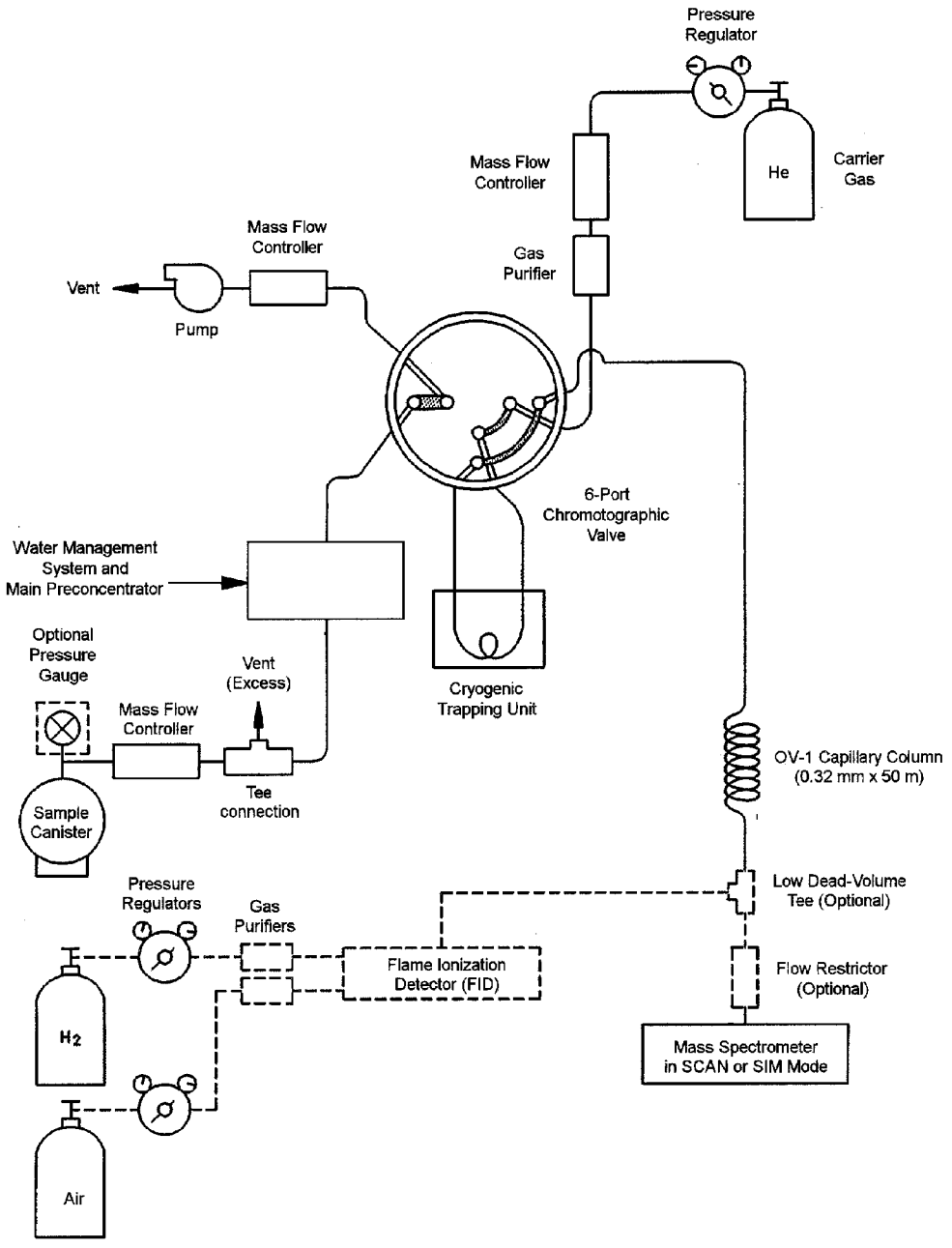
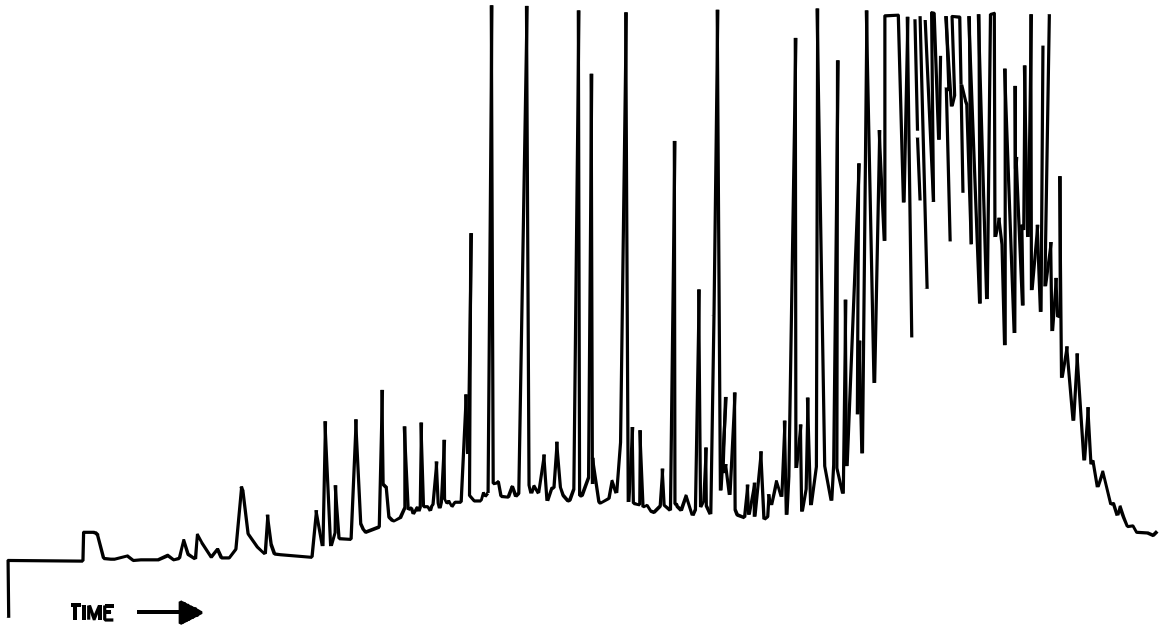


Figure 11. Canister analysis utilizing GC/MS/SCAN/SIM analytical system with optional flame ionization detector with 6-port chromatographic valve in the sample desorption mode.
[Alternative analytical system illustrated in Figure 16.]

(a). Certified Sampler



(b). Contaminated Sampler

Figure 12. Example of humid zero air test results for a clean sample canister (a) and a contaminated sample canister (b).

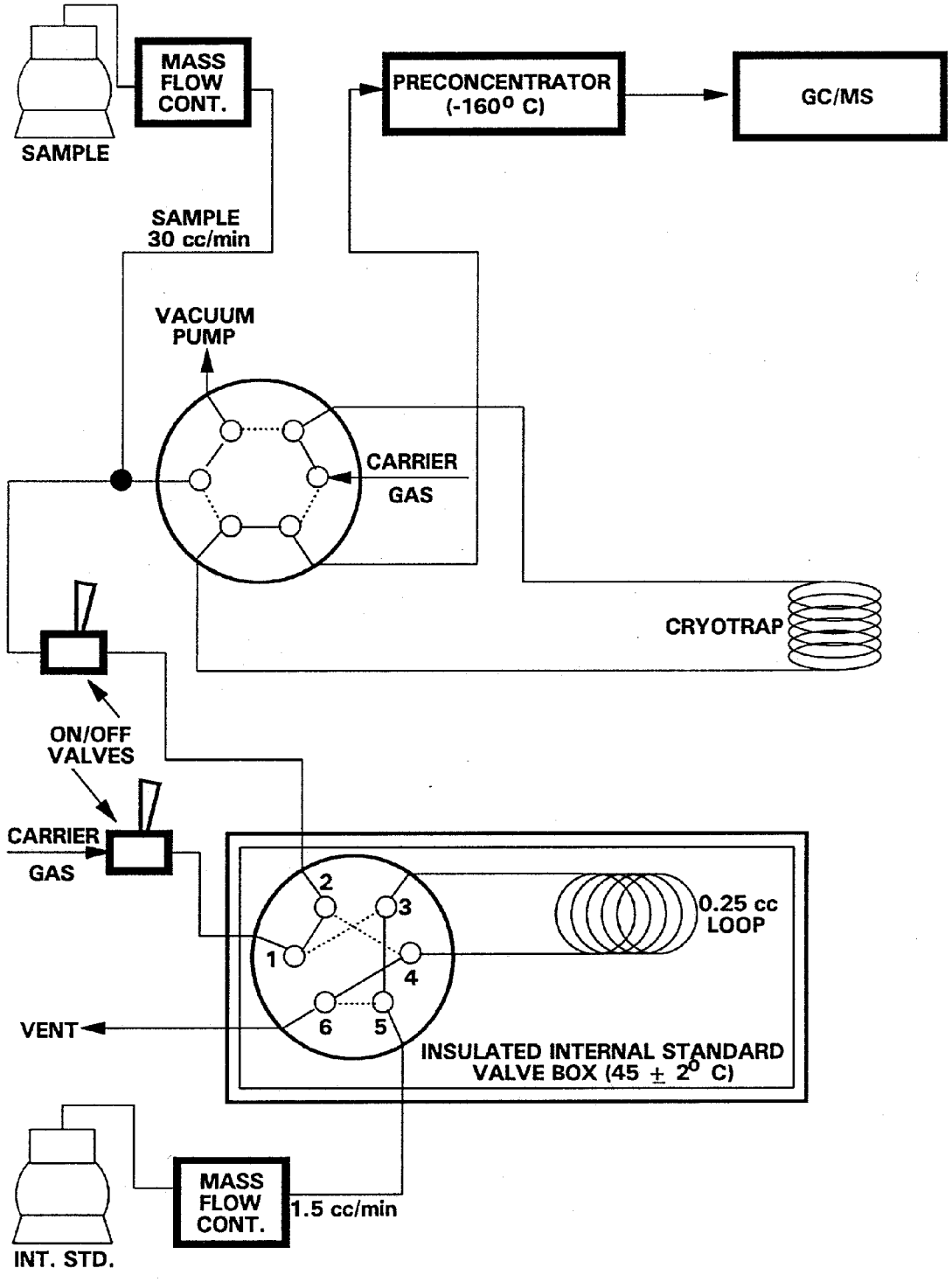


Figure 13. Diagram of design for internal standard addition.

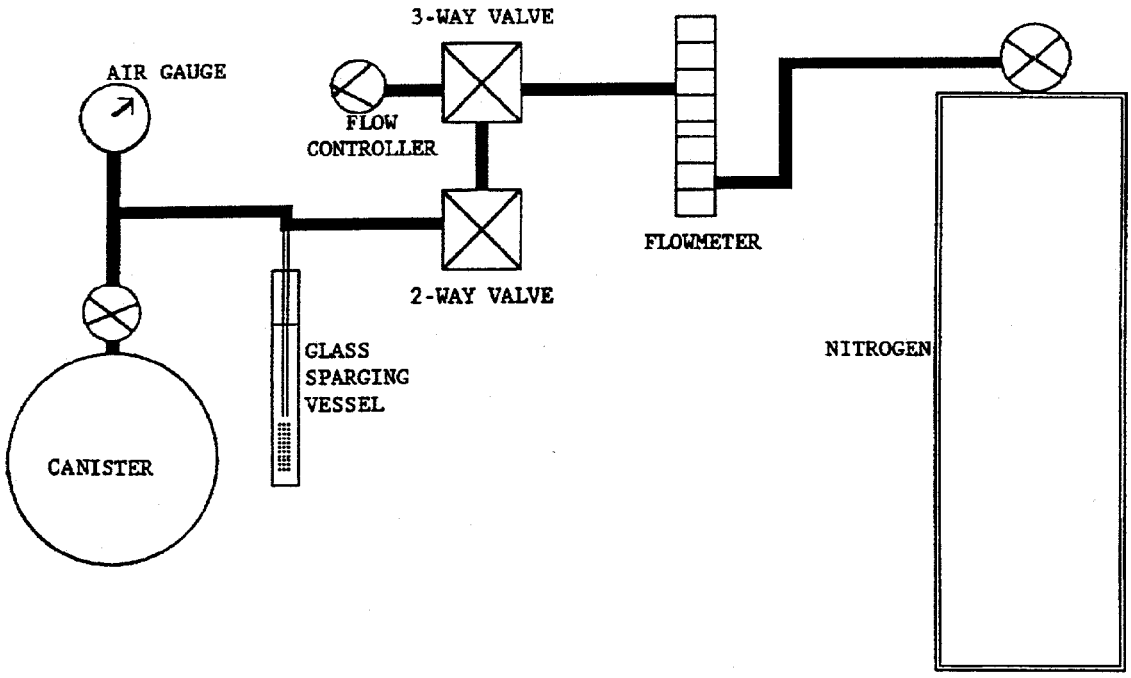


Figure 14. Water method of standard preparation in canisters.

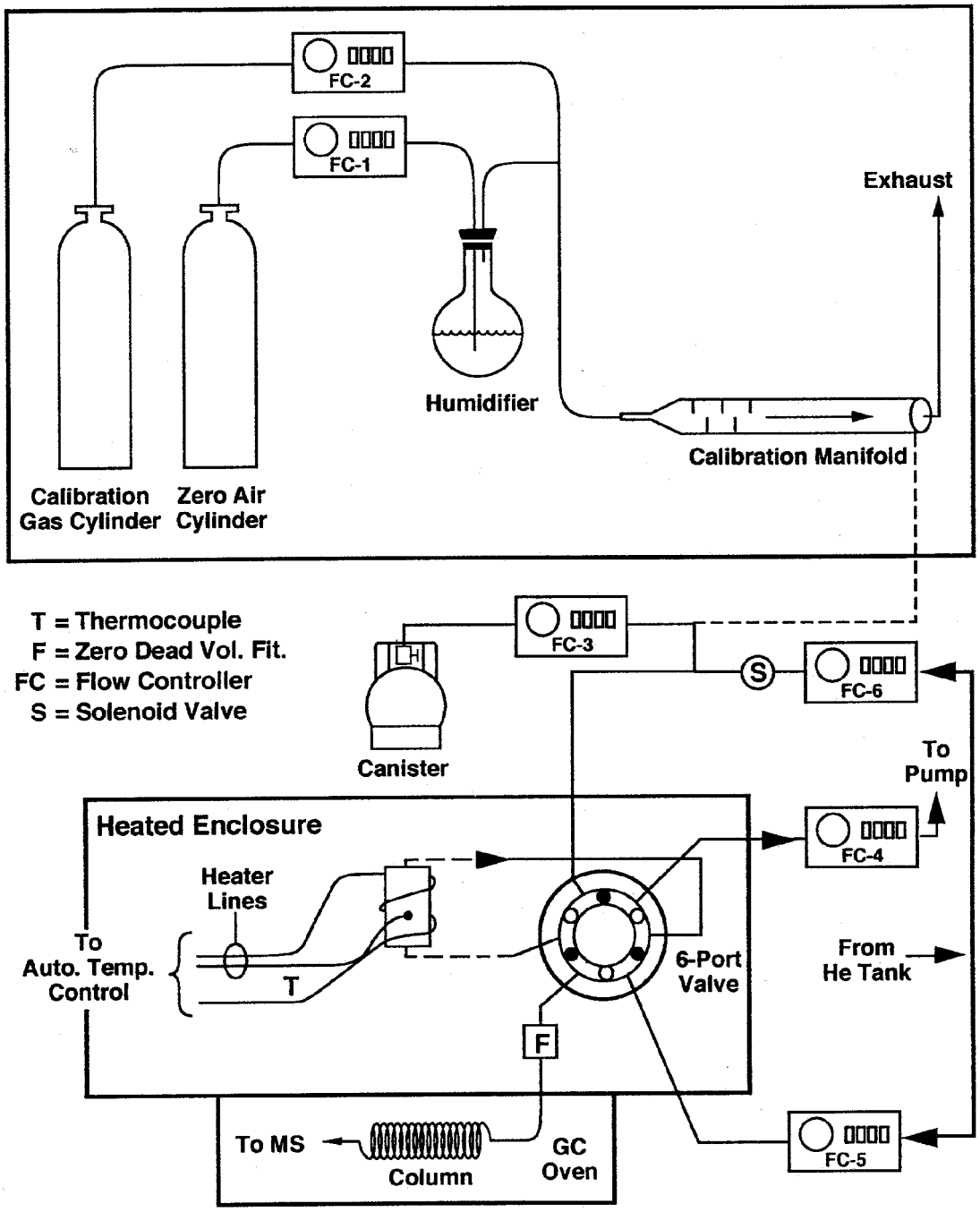


Figure 15. Diagram of the GC/MS analytical system.

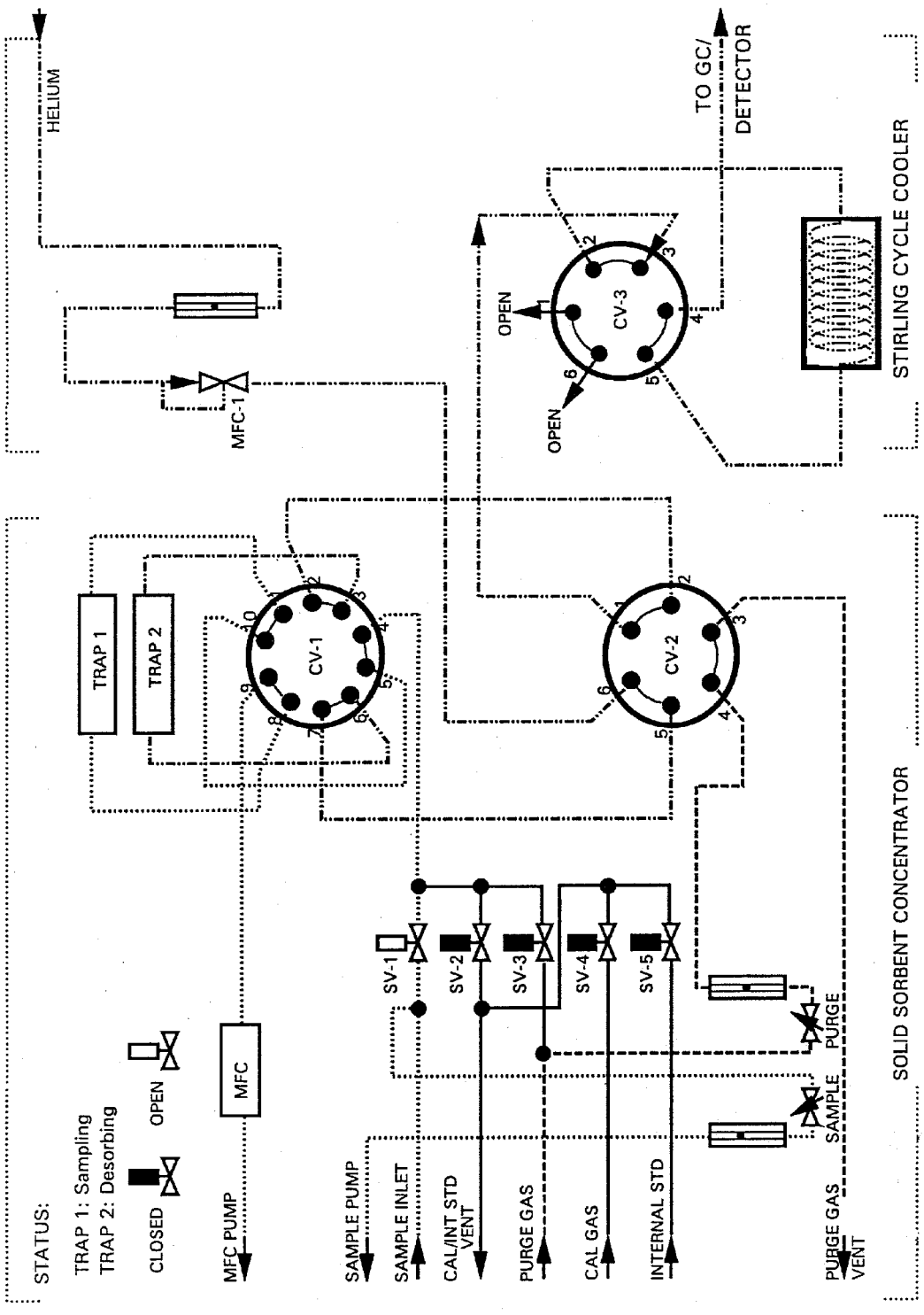
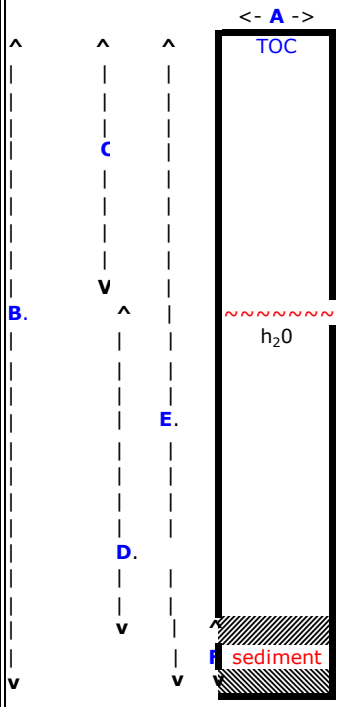


Figure 16. Sample flow diagram of a commercially available concentrator showing the combination of multisorbent tube and cooler (Trap 1 sampling; Trap 2 desorbing).

Appendix F
Water Sampling Log - Example

ECOR Solutions

Groundwater Sampling Log
Former Monarchs Chemical Facility
Geneva, NY
Monitoring Well MW-114



Condition of Well	Good
Method of Well Purging	Bladder pump
Method of Water Sampling	flow thru cell

A. Diameter	2	inches
B. Well Depth	16	feet below TOC
C. Depth to Water	9.95	feet below TOC
Potentiometic Elevation	452.08	ft AMSL
D. Length of Water Column	4.05	
	0.16	gal/ft
well volume	0.648	gallons
pore volumes evacuated	3	
calculated evacuation volum	1.944	gallons
actual evacuation volume	2	gallons
E. Installed Depth	448.03	feet below TOC
F. Occlusion Depth		feet

Field Measurements

		initial	1st Volume	2nd Volume	Final	Stabilization RPD
date		9/26/2007				
time						
EH	mV	-114	-114		-118	3.5%
temp.	°C or **°F**	12.1	12.1		11.9	1.7%
pH	SU	6.9	6.9		7.1	2.9%
spec. con	umhos/cm	765	765		811	6.0%
DO	mg/l	1.3	1.3		1.4	7.7%
appearance		clear				

Recharge

initial depth to water	9.95
purge depth to water	9.99
Recharge	0.4%

Weather sunny, cool (40°F)

Observations

Sampler

JLG — name

9/26/2007 — date

Appendix G
Chain-of-Custody - Example

CHAIN OF CUSTODY RECORD

TestAmerica
ANALYTICAL TESTING CORPORATION

☐ Nashville, TN ☐ Orlando, FL ☐ Cedar Falls, IA
☐ Dayton, OH ☐ Watertown, WI ☐ Pontiac, MI
☐ Indianapolis, IN

To assist us in using the proper analytical methods, is this work being conducted for regulatory purposes?

Client Name/Account #: _____ Compliance Monitoring? Yes No

Address: _____ Enforcement Action? Yes No

City/State/Zip: _____ **Report To:** _____

Project Manager: _____ **Invoice To:** _____

Telephone Number: _____ **Fax No.:** _____ **TA Quote #:** _____

Sampler Name: (Print) _____ **Project ID:** _____

Sampler Signature: _____ **Project #:** _____

| | | | | | | | Preservative | | | | | | | Matrix | | | | | | Analyze For: | | | | | | |

Appendix H
TestAmerica Quality Management Plan

(Document on CD-ROM)

QUALITY MANAGEMENT PLAN

Revision: 7

July 2005

Approved by:

President and CEO: _____ Date: 7/28/2005
Rachel Brydon Janetta

Senior Vice President,
Chief Operating Officer: _____ Date: 7/28/2005
Dr. Keith C. Wheatstone

Vice President,
Client and Operations
Services: _____ Date: 7/28/2005
Dr. Charles W. Carter

Corporate
Quality Director: _____ Date: 10/28/2005
Raymond J. Frederici

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SEVERN TRENT STL

Vision

STL will be the recognized industry leader for environmental analysis.

Mission

Through the innovation and dedication of our people, together with the quality of our systems, we will deliver levels of performance that delight our clients, retain the confidence of our stakeholders and enable the profitable growth of our business.

Severn Trent Laboratories

1. Introduction, Purpose, and Scope

1.1. Severn Trent Laboratories (STL) Overview

Severn Trent Laboratories (STL) is the largest environmental laboratory company in the world with revenues in excess of $300 million and 31 laboratory locations in 19 states nationwide. These facilities have the combined experience of over 500 years in the environmental testing business.

Through continued investment in facilities, equipment, methods and people, STL has developed an unprecedented team of resources, experience and capabilities. With over 2000 chemists, microbiologists and environmental scientists, STL is well positioned to support a variety of clients including commercial, governmental and chemical industries.

STL offers a broad range of environmental testing services. STL's testing capabilities include chemical, physical, and biological analyses of a variety of matrices, including aqueous, solid, drinking water, waste, tissue, air, mold and fungus (Mycology) and saline/estuarine samples. Specialty capabilities include air toxics testing, radiological, mixed waste testing, geotechnical testing, tissue preparation and analysis, aquatic toxicology, dioxin/furan testing, indoor air quality and microscopy services, aquatic toxicology, asbestos analysis, High Resolution Mass Spectrometry (HRMS), Inductively Coupled Plasma/MS (ICP/MS), Liquid Chromatography/MS (LC/MS), PCR microbiology and on-site technologies including mobile laboratories. STL facility locations and contact information are outlined in Table 1.

1.2. Quality Assurance Policy

It is STL's policy to:

- Provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- Generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- Ensure employee adherence to quality documentation and implementation of Corporate Policies and Procedures.
- Provide STL clients with the highest level of professionalism and the best service practices in the industry.
- Build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- Maintain a working environment that fosters open communication with both clients and staff and ensures data integrity.

Table 1 STL Facility Locations

Aerotech (AEL)
4645 East Cotton Center
Blvd., Suite 189
Phoenix, AZ 85040
Phone: 602-437-3340
Fax: 623-445-6192

STL Austin
14050 Summit Drive
Suite A100
Austin, TX 78728
Phone: 512-244-0855
Fax: 512-244-0160

STL Billerica
148 Rangeway Road
N. Billerica, MA 01862
Phone: 978-667-1400
Fax: 978-667-7871

STL Buffalo
10 Hazelwood Drive
Suite 106
Amherst, NY 14228
Phone: 716-691-2600
Fax: 716-691-7991

STL Burlington
208 South Park Drive
Suite 1
Colchester, VT 05446
Phone: 802-655-1203
Fax: 802-655-1248

STL Connecticut
128 Long Hill Cross Road
Shelton, CT 06484
Phone: 203-929-8140
Fax: 203-929-8142

STL Chicago
2417 Bond Street
University Park, IL 60466
Phone: 708-534-5200
Fax: 708-534-5211

STL Corpus Christi
1733 N. Padre Island Drive
Corpus Christi, TX 78408
Phone: 361-289-2673
Fax: 361-289-2471

STL Denver
4955 Yarrow Street
Arvada, CO 80002
Phone: 303-736-0100
Fax: 303-431-7171

STL Edison
777 New Durham Road
Edison, NJ 08817
Phone: 732-549-3900
Fax: 732-549-3679

STL Houston
6310 Rothway Drive
Houston, TX 77040
Phone: 713-690-4444
Fax: 713-690-5646

STL Knoxville
5815 Middlebrook Pike
Knoxville, TN 37921
Phone: 865-291-3000
Fax: 865-584-4315

STL Los Angeles
1721 South Grand Avenue
Santa Ana, CA 92705
Phone: 714-258-8610
Fax: 714-258-0921

STL Miami
10200 USA Today Way
Miramar, FL 33025
Phone: 954-431-4550
Fax: 954-431-1959

STL Mobile
900 Lakeside Drive
Mobile, AL 36693
Phone: 251-666-6633
Fax: 251-666-6696

STL Newburgh
315 Fullerton Avenue
Newburgh, NY 12550
Phone: 845-562-0890
Fax: 845-562-0841

STL North Canton
4101 Shuffel Drive NW
North Canton, OH 44720
Phone: 330-497-9396
Fax: 330-497-0772

STL On-Site Technology
Westfield Executive Park
53 Southampton Road
Westfield, MA 01085
Phone: 413-572-4000
Fax: 413-572-3707

STL Pensacola
3355 McLemore Drive
Pensacola, FL 32514
Phone: 850-474-1001
Fax: 850-478-2671

STL P&K
1936 Olney Ave.
Cherry Hill, NJ 08003
Phone: 856-489-4455
Fax: 856-489-4085

STL Pittsburgh
301 Alpha Drive
RIDC Park
Pittsburgh, PA 15238
Phone: 412-963-7058
Fax: 412-963-2468

STL Richland
2800 George Washington
Way
Richland, WA 99354
Phone: 509-375-3131
Fax: 509-375-5590

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605
Phone: 916-373-5600
Fax: 916-372-1059

STL San Francisco
1220 Quarry Lane
Pleasanton, CA 94566-4756
Phone: 925-484-1919
Fax: 925-484-1096

STL Seattle
5755 8th Street East
Tacoma, WA 98424
Phone: 253-922-2310
Fax: 253-922-5047

STL St. Louis
13715 Rider Trail North
Earth City, MO 63045
Phone: 314-298-8566
Fax: 314-298-8757

STL Savannah
5102 LaRoche Avenue
Savannah, GA 31404
Phone: 912-354-7858
Fax: 912-351-3673

STL Tallahassee
2846 Industrial Plaza Dr.
Tallahassee, FL 32301
Phone: 850-878-3994
Fax: 850-878-9504

STL Tampa
6712 Benjamin Road
Suite 100
Tampa, FL 33634
Phone: 813-885-7427
Fax: 813-885-7049

STL Valparaiso
2400 Cumberland Drive
Valparaiso, IN 46383
Phone: 219-464-2389
Fax: 219-462-2953

STL Westfield
Westfield Executive Park
53 Southampton Road
Westfield, MA 01085
Phone: 413-572-4000
Fax: 413-572-3707

1.3. Management Commitment to Quality Assurance and Data Integrity

STL management is committed to providing data of known and documented quality and the best service in the environmental testing industry. To ensure that the data produced and reported by STL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL maintains quality and data integrity systems that are clear, effective, well communicated, and supported at all levels in the company.

1.4. Purpose

The purpose of the Quality Management Plan (QMP) is to describe the STL quality system and to outline how that system enables all employees of STL to meet the Quality Assurance (QA) policy. The QMP also describes specific QA activities and requirements and prescribes their frequencies. Roles and responsibilities of management and laboratory staff in support of the quality system are also defined in the QMP.

1.5. Scope

The requirements set forth in this document are applicable to all STL facilities. Where the document uses the terms “must” and “shall”, this denotes required activities. Practices described in this QMP denote how those activities are performed in general; and each laboratory may have a more detailed description of that activity.

Each STL facility has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where this QMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The facility’s Laboratory Quality Manual (LQM) shall take precedence over the QMP in those cases. Secondarily, each STL facility has the responsibility and authority to operate in compliance with documented client requirements, where they do not conflict with regulatory requirements. STL shall not enter any client agreements that conflict with regulatory requirements in the jurisdiction in which the work is performed. Where documented client agreements conflict with this QMP, but meet the regulatory requirements of the jurisdiction in which the work is performed, the client agreements shall supersede requirements in this QMP.

STL operates under the regulations and guidelines of the following federal programs:

- Air Force Center for Environmental Excellence (AFCEE)
- US Army Corp of Engineers, Hazardous, Toxic and Radioactive Waste (USACE HTRW)
- Clean Air Act (CAA)
- Clean Water Act (CWA)

- Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
- Department of Energy (DOE)
- Marine Protection, Research, and Sanctuaries Act (MPRSA)
- Navy Facilities Engineering Service Center (NFESC)
- National Pollutant, Discharge, and Elimination System (NPDES)
- Nuclear Regulatory Commission (NRC)
- Occupational Safety and Health Administration (OSHA)
- Resource Conservation and Recovery Act (RCRA)
- Safe Drinking Water Act (SDWA)
- Toxic Substances Control Act (TSCA)

STL also provides services under various state and local municipal guidelines. A listing of each laboratory's service offerings and certifications is presented on STL's website under the MySTL webpage or available from the laboratory.

This QMP was written to comply with the National Environmental Laboratory Accreditation Conference (NELAC) standards. Refer to Table 2 for a cross-section comparison of this QMP to the NELAC standards.

Table 2 Correlation of QMP Sections with NELAC Quality Manual Requirements

NELAC Chapter 5.4.2.3 Quality Manual	Quality Management Plan Section
a. Quality policy statement, including objectives and commitments, by top management	1.2 Quality Assurance Policy 4.2.1 Objectives of the Quality System
b. Organization and management structure	4.1 Organization and Management
c. Relationship between management, technical operations, support services and the quality systems	4.1.2 Roles and Responsibilities 4.2 Quality System
d. Records retention procedures; document control procedures	4.3 Document Control 4.12.2 Record Retention
e. Job descriptions of key staff and references to job descriptions of other staff	4.1.2 Roles and Responsibilities
f. Identification of laboratory approved signatories	4.1 Organization and Management
g. Procedures for achieving traceability of measurements	5.5 Measurement Traceability
h. List of all test methods under which the laboratory performs its accredited testing	5.3.1 Method Selection
i. Mechanisms for assuring the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	4.4.2 Project-Specific Quality Planning
j. Reference to the calibration and/or verification test procedures used	5.4.3 Equipment Verification and Calibration
k. Procedures for handling submitted samples	4.7.1 Sample Acceptance Policy 5.7 Sample Handling, Transport and Storage
l. Reference to the major equipment and reference measurement standards used as well as the facilities and services used in conducting tests	4.1.1 Laboratory Facilities 5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
m. Reference to procedures for calibration, verification and maintenance of equipment	5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
n. Reference to verification practices including inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal QC schemes	5.8.1 Proficiency Testing 5.8.2 Control Samples

Table 2 Correlation of QMP Sections with NELAC Quality Manual Requirements

NELAC Chapter 5.4.2.3 Quality Manual	Quality Management Plan Section
o. Procedures for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur	4.9 Control of Non-Conformances 4.10 Corrective Action 4.11 Preventive Action 5.8.5 Permitting Departures from Documented Procedures
p. Laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications	4.4.2 Project-Specific Quality Planning 5.8.5 Permitting Departures from Documented Procedures
q. Procedures for dealing with complaints	4.8 Complaints
r. Procedures for protecting confidentiality (including national security concerns) and proprietary rights	4.7.2 Client Confidentiality and Proprietary Rights
s. Procedures for audits and data review	4.13 Internal Audits 4.14 External Audits 5.3.6 Data Reduction and Review
t. Process/procedures for establishing that personnel are adequately experienced in duties they are expected to carry out and are receiving any needed training	5.1.2 Training
u. Reference to procedures for reporting analytical results	5.3.6 Data Review 5.9 Project Reports
v. Table of contents, listing reference, glossaries and appendices	TOC Table of Contents Appendix I: List of Cited SOPs and Work Instructions

2. References

The following references were used in preparation of this document and as the basis of the STL Quality System:

EPA Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA/G-6, US EPA, Office of Environmental Information, March 2001.

EPA Requirements for Quality Management Plans, EPA QA/R-2, US EPA, Office of Environmental Information, March 2001.

EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, US EPA, Office of Environmental Information, March 2001.

EPA Quality Manual for Environmental Programs, 5360 A1, US EPA Office of Environmental Information, Quality Staff, May 2000.

General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025, December 1999.

Good Automated Laboratory Practices, EPA 2185, US EPA, Office of Environmental Information, Resource Management, August 1995.

Quality Assurance Project Plan, HQ Air Force Center for Environmental Excellence, Version 3.1, August 2001.

National Environmental Laboratory Accreditation Conference Standards, EPA/600/R-04/003, US EPA Office of Research and Development, July 2003.

Navy Installation Restoration Laboratory Quality Assurance Guide, Interim Guidance Document, Naval Facilities Engineering Service Center, February 1996.

Navy Installation Restoration Chemical Data Quality Manual, Navy IR CDQM, September 1999.

Quality Systems Manual for Environmental Laboratories, Department of Defense, Final Version 3, March 2005.

Shell for Analytical Chemistry Requirements, US Army Corps of Engineers, December 1998.

3. Terms and Definitions

Accuracy: the degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Audit: a systematic evaluation to determine the conformance to specifications of an operational function or activity.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of 1 to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Chain of Custody (COC): A system of documentation demonstrating the physical possession and traceability of samples.

Clean Air Act: legislation in 42 U.S.C. 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): legislation (42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and reauthorization Act of 1986 (SARA), 42 U.S.C. 9601et seq.

Compromised Sample: a sample received in a condition that jeopardizes the integrity of the results. See Section 4.7.1 for a description of these conditions.

Confidential Business Information (CBI): information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products.

Confirmation: verification of the presence of a component using an additional analytical technique. These may include second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Equipment Blank: a portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Rinsate.

Document Control: the act of ensuring that documents (electronic or hardcopy and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): legislation under 7 U.S.C. 135 et seq., as amended.

Federal Water Pollution Control Act (Clean Water Act, CWA): legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat. 816.

Field Blank: a blank matrix brought to the field and exposed to field environmental conditions.

Field of Proficiency Testing: NELAC's approach to offering proficiency testing by matrix, technology, and analyte/analyte group.

Good Laboratory Practices (GLP): formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729 and required for activities performed under FIFRA and TSCA.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Instrument Blank: a blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis.

Internal Chain of Custody: an unbroken trail of accountability that ensures the physical security of samples, data and records. Internal Chain of Custody refers to additional documentation procedures implemented within the laboratory that includes special sample storage requirements, and documentation of all signatures and/or initials, dates, and times of personnel handling specific samples or sample aliquots.

Internal Standard: A standard added to samples in known amount and carried through the procedure as a reference for calibration and controlling instrumental and analytical precision and bias.

Instrument Detection Limit (IDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Laboratory Quality Manual (LQM): a document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system.

Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent.

Matrix: the substrate of a test sample. Common matrix descriptions are defined in Table 3.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Table 3 Matrix Descriptions

Matrix	Description
Air	Air samples as analyzed directly or as adsorbed into a solution or absorption matrix and desorbed.
Aqueous	Aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine source. Includes surface water, groundwater and effluents.
Chemical Waste	A product or by-product of an industrial process that results in a matrix not previously defined.
Drinking Water	Aqueous sample that has been designated a potable water source.
Saline	Aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.
Liquid	Liquid with <15% settleable solids.
Solid	Soil, sediment, sludge or other matrices with ≥15% settleable solids.
Waste	A product or by-product of an industrial process that results in a matrix not previously defined.
Tissue	Sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Method Blank (MB): a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): one way to establish a Limit of Detection, defined as the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Precision: an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

Proficiency Testing: determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.

Proficiency Test (PT) Sample: a sample, the composition of which is unknown to the analyst, that is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. Also referred to as Performance Evaluation (PE) Sample.

Proprietary: belonging to a private person or company.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Assurance Project Plan (QAPP): a formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

Quality Control (QC): the overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control Sample: a sample used to assess the performance of all or a portion of the measurement system. QC samples may be Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking.

Quality Management Plan (QMP): a formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory to ensure the quality of its product and the utility of the product to its users.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA/QC.

Quantitation Limit (QL): the minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Raw Data: any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports specifying inclusion of "raw data" do not need all of the above included, but sufficient information to create the reported data.

Record Retention: the systematic collection, indexing and storing of documented information under secure conditions.

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Resource Conservation and Recovery Act (RCRA): legislation under 42 USC 321 et seq. (1976).

Safe Drinking Water Act (SDWA): legislation under 42 USC 300f et seq. (1974), (Public Law 93-523).

Sampling and Analysis Plan (SAP): a formal document describing the detailed sampling and analysis procedures for a specific project.

Selectivity: the capability of a measurement system to respond to a target substance or constituent.

Sensitivity: the difference in the amount or concentration of a substance that corresponds to the smallest difference in a response in a measurement system using a certain probability level.

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Storage Blank: a blank matrix stored with field samples of a similar matrix.

Systems Audit: a thorough, systematic, on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

Test Method: an adoption of a scientific technique for performing a specific measurement, as documented in a laboratory SOP or as published by a recognized authority.

Toxic Substances Control Act (TSCA): legislation under 15 USC 2601 et seq., (1976).

Traceability: the property of a result of a measurement that can be related to appropriate international or national standards through an unbroken chain of comparisons.

Trip Blank: a blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Verification: confirmation by examination and provision of evidence against specified requirements.

4. Management Requirements

4.1. Organization and Management

4.1.1. Organization

STL's organizational structure is presented in Figure 1. Corporate employees are located at various STL facilities as outlined in the organizational structure. A QA Manager shall be designated at each STL facility.

4.1.2. Roles and Responsibilities

President and CEO
The President of STL, Inc. has overall management responsibility and authority for Severn Trent's laboratory division, including responsibility for budgeting, resource allocation, long term planning, sales, marketing, and final approval on all management and administrative policies and management plans. The President authorizes the QMP and as such, sets the standards for the quality system.

Senior Vice President and Chief Operating Officer (COO)
The COO is responsible for daily management of all STL facilities. The COO's responsibilities include allocation of personnel and resources, long term planning, and development of technical policies and management plans. The COO authorizes the QMP and is responsible for ensuring that business and technical operations are conducted in accordance with its requirements.

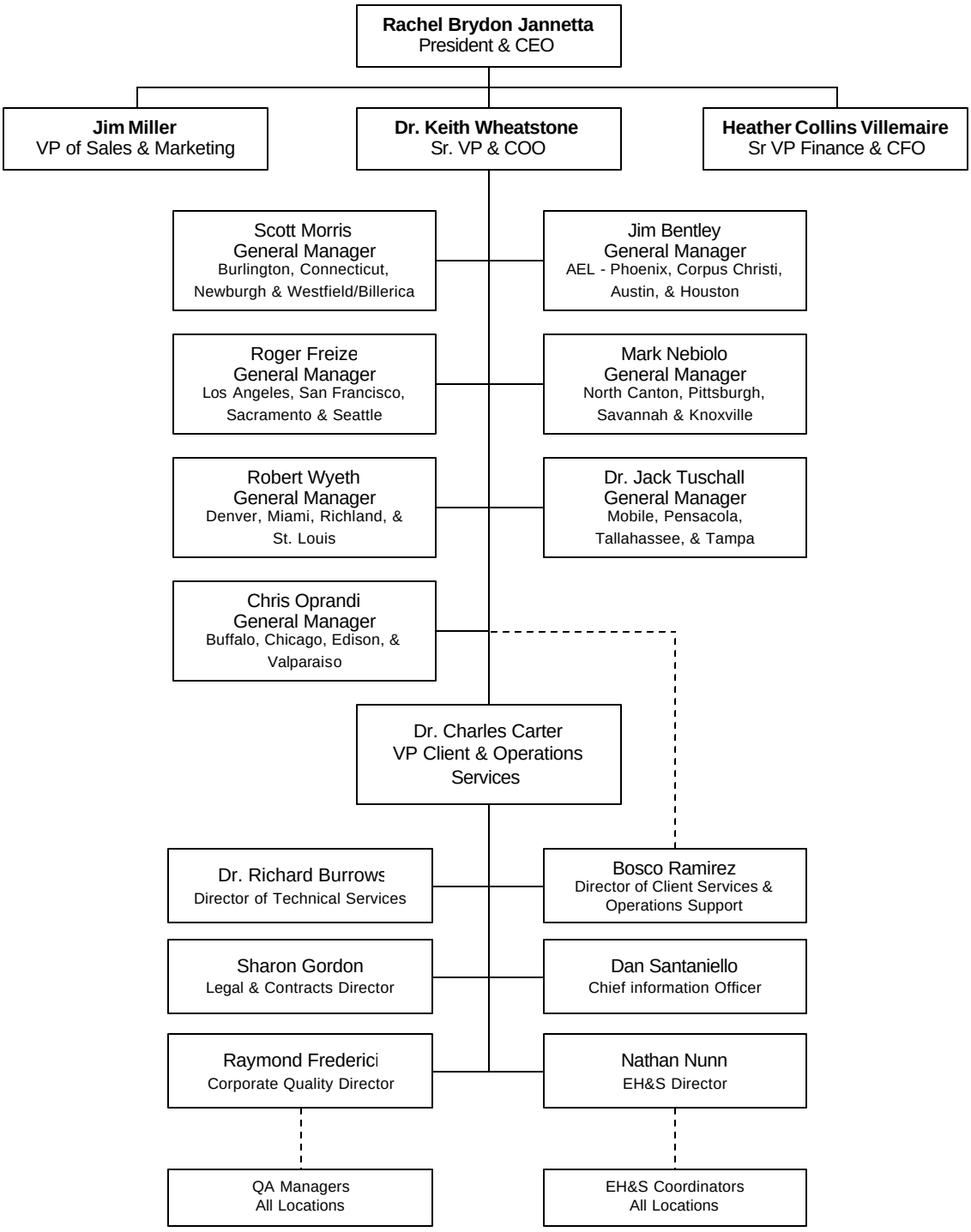
Vice President Client and Operations Services (VP COS)
The VP COS is responsible for all essential elements of offerings to clients, including risk management, legal compliance and contract administration, quality assurance, information technology, and environmental health and safety. The VP COS authorizes the QMP and responsibilities include authorization of Manuals, Policies and Procedures, providing support and direction to the Managers of these

areas, and supporting the COO in decisions regarding long term planning, resource allocation, and capital expenditures.

Corporate Quality Director

The Quality Director is responsible for establishing, implementing and communicating STL's quality system. The Quality Director monitors compliance with the QMP, provides regulatory and technical updates to the STL facilities, assists in development of management plans and technical policies to be approved by the COO, and coordinates training within STL. The Quality Director is available to any employee in STL to resolve data quality or ethical issues. The Quality Director is independent of operational functions.

Figure 1. STL Organizational Chart



Director of Technical Services
The Director of Technical Services is responsible for establishing, implementing and communicating STL's Technical Policies, Standard Operating Procedures, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices throughout STL, advising STL staff on technology advances, innovations, and applications, and organizing and running STL's technical committee.

Director of Client Services and Operations Support
The Director of Client Services and Operations Support is responsible for establishing, implementing and communicating STL's Client Services policies, guidelines and applicable Standard Operating Procedures. The Client Services Directors monitors overall client services indicators and compliance with the appropriate company policies and procedures. The Client Services Director is available to any laboratory to assist in addressing client services issues or to perform reviews and assessments of local client services systems. Other duties include identifying and establishing best client services practices, assisting in Project Management and Client Services training and coordinating STL's Client Service Managers' group.

Chief Information Officer (CIO)
The CIO is responsible for establishing, implementing and communicating STL's Information Technology (IT) Policies, Standard Operating Procedures, and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as STL's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with Good Automated Laboratory Practices (GALP), and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various STL facilities.

Environmental Health and Safety (EH&S) Director
The EH&S Director is responsible for establishing, implementing and communicating STL's Environmental Health and Safety Policies, Standard Operating Procedures, and Manuals. Other responsibilities include conducting EH&S assessments as required, acting as a resource for all STL facilities to ensure EH&S compliance, coordinating safety committees, providing guidance to the EH&S Coordinator at various STL facilities, and advising STL facilities on new EH&S regulations.

General Manager (GM)
The GM is directly responsible for the daily operations of one or more operating facilities within STL. The GM's responsibilities include allocation of personnel and resources, long term planning, setting goals, and achieving the financial, business, and quality objectives of STL. The GM ensures timely compliance with corporate management directives, policies, and management systems reviews.

Laboratory Director
The Laboratory Director oversees the daily operations of the laboratory. The Laboratory Director's responsibilities include supervision of staff, setting goals and objectives for both the business and the employees, and achieving the financial, business, technical and quality objectives of the facility. The Laboratory Director ensures timely compliance with audits and corrective actions, and is responsible for maintaining a working environment which encourages open, constructive problem solving and continuous improvement.

QA Manager
The QA Manager is responsible for ensuring that the laboratory's quality system and LQM meet the requirements set forth in the QMP, providing quality systems training to all new personnel, maintaining an LQM, and performing or overseeing systems, data, special, and external audits. The QA Manager performs, or supervises, the maintenance of QA records, the maintenance of certifications and accreditations, the submission of monthly QA Reports, and assists in reviewing new work as needed. The QA Manager shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QA Manager is available to any employee at the facility to resolve data quality or ethical issues. The QA Manager shall be independent of laboratory operations. The facility QA Manager has an indirect reporting relationship to the Quality Director. Each LQM has further descriptions of roles and responsibilities at the facility level.

Technical Director
The Technical Director(s) of a laboratory has overall responsibility for a defined portion of the technical operations of the laboratory, and may or may not be the Laboratory Director. The Technical Director solves day to day technical issues, provides technical training and guidance to staff, project managers, and clients, investigates technical issues identified by QA, and directs evaluation of new methods.

4.2. Quality System

4.2.1. Objectives of STL Quality System

The goal of the STL quality system is to ensure that business and technical operations are conducted with the highest standards of professionalism and ethics in the industry.

To achieve this goal, it is necessary to provide STL clients with not only scientifically sound, well documented, and regulatory compliant data, but also to ensure that STL provides the highest quality service available in the industry with uncompromising data integrity. A well-structured and well-communicated quality system is essential in meeting this goal. STL's quality system is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

The QMP is the basis for STL's quality and data integrity system. It contains requirements and general guidelines under which all STL facilities shall conduct their operations. A table listing the minimum quality system policies and procedures is appended to this QMP. The table includes a citation to the applicable QMP section where a procedure or policy is discussed. It also includes a column indicating the document "Reference".

4.2.2. Laboratory Quality Manual (LQM)

Each STL facility shall have an LQM that further describes the specific QA program at the laboratory.

Each STL facility's LQM shall address:

1. Table of Contents, lists of references and glossaries, and appendices.
2. Quality policy statement, including objectives and commitments, by facility management.
3. Organization and management structure of the laboratory, its place in the STL organization and relevant organizational charts.
4. Relationship between management, technical operations, support services and the quality system.
5. Required Training Elements (e.g., Ethics Training and Technical Training).
6. Record retention procedure.
7. Document control procedure.
8. Job descriptions of essential staff and reference to job descriptions of other staff.
9. Identification of the laboratory's approved signatories.
10. Procedure for achieving traceability of measurements.
11. List of test methods under which the laboratory performs its testing.
12. Procedure for reviewing new work.
13. Reference to the calibration and/or verification test procedures used.
14. Sample handling procedure.
15. Reference to the major equipment, reference standards, facilities and services used by the laboratory in conducting tests.

16. Reference to procedures for calibration, verification and maintenance of equipment.
17. Reference to verification practices including inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal QC practices.
18. Procedures for feedback and corrective action when testing discrepancies are detected, or departures from policies and procedures occur.
19. Procedure for exceptionally permitting departures from documented policies and procedures or from standard specifications.
20. Procedure for handling client complaints.
21. Procedure for protecting client confidentiality and proprietary rights.
22. Procedure for audits and data review.
23. Procedure for establishing that personnel are adequately experienced and trained.
24. Reference to procedures for reporting analytical results.

4.3. Document Control

4.3.1. Document Type

The following documents, at a minimum, must be controlled at each STL Facility:

- Laboratory Quality Manual
- Standard Operating Procedures (SOP)
- Corporate Quality Management Plan
- Corporate Policies and Procedures

4.3.2. Document Control Procedure

Security and control of documents are necessary to ensure that confidential information is not distributed and that all current copies of a given document are from the latest applicable revision. Unambiguous identification of a controlled document is maintained by identification of the following items in the document header: Document Name, Document Number, Revision Number, Revision Date, Effective Date, Number of Pages. Controlled documents are authorized by Management and/or the QA Department. Controlled documents are marked as such and records of their distribution are kept by the QA Department. Document control maybe achieved by either electronic or hardcopy distribution.

Controlled documents shall be available at all locations where the operational activity described in the document is performed.

4.3.3. Document Revision

Quality system policies and procedures will be reviewed at a minimum of every two years and revised as appropriate. Changes to documents occur when a procedural change warrants a revision of the document. When an approved revision of a controlled document is ready for distribution, obsolete copies of the document shall be replaced with the current version of the document. The previous revision of the controlled document must be archived by the QA Department.

4.3.4. Official Documents

The STL Corporate Operations staff posts Corporate Manuals, Standard Operating Procedures, Policies, Work Instructions, White Papers and Training Materials on STL's intranet site. These are collectively termed "Official Documents" and encompass the Policies and Procedures that all STL facilities are required to employ. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving official documents is found in Corporate SOP S-Q-001, *Official Document Control and Archive*.

4.4. Request, Tender, and Contract Review

4.4.1. Contract Review

For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is STL's intent to provide both standard and customized environmental testing services to our clients. To ensure project success, technical staff shall perform a thorough review of technical and QC requirements contained in contracts. Contracts are reviewed for adequately defined requirements and STL's capability to meet those requirements.

Contract review shall include a review of the client's requirements in terms of compound lists, test methodology requested, sensitivity, accuracy, and precision requirements. The STL representative ensures that the laboratory's test methods are suitable to achieve these requirements and must ensure that the laboratory holds the appropriate certifications and approvals to perform the work. The review also includes the laboratory's capabilities in terms of turnaround time, capacity, and resources to provide the services requested, as well the laboratory's ability to provide the documentation, whether hardcopy or electronic. If the laboratory cannot provide all services but intends to subcontract such services, whether to another STL facility or to an outside firm, this must be documented and discussed with the client prior to contract approval.

All contracts entered into by STL shall be reviewed and approved by the appropriate personnel at the facility or facilities performing the work. Any contract requirement or amendment to a contract communicated to STL verbally must be documented and confirmed with the client in writing. Any discrepancy between the client's requirements and STL's capability to meet those requirements is resolved in writing before acceptance of the contract. Contract amendments, initiated by the client and/or STL, are documented in writing for the benefit of both the client and STL.

All contracts, Quality Assurance Project Plans (QAPPs), Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the permanent project record as defined in Section 4.12.1.

4.4.2. Project Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, STL assigns a Project Manager (PM) to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively communicated to the laboratory personnel before and during the project.

Each STL facility shall have established project planning procedures in order to ensure that communication is inclusive and effective. These include project memos, designation and meetings of project teams, and meetings between the laboratory staff and the client. STL has found it very effective to invite the client into this process. STL strongly encourages our clients to visit the laboratories and hold formal or informal sessions with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

4.4.3. Data Quality Objectives

Data Quality Objectives (DQO) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application. Typically, DQOs are identified before project initiation, during the development of QAPPs and SAPs. The analytical DQOs addressed in this section are precision, accuracy, representativeness, completeness, and comparability.

The components of analytical variability (uncertainty) can be estimated when QC samples of the right types and at the appropriate frequency are incorporated into measurement process at the analytical laboratory. STL incorporates numerous QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QC samples and their applications, described in Section 5.8.2, are selected based on regulatory, method- or client-specific requirements. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses may include calibration blanks, instrument blanks, method blanks, LCS, calibration standards, MS, MSD, surrogate spikes, and yield monitors.

The DQOs discussed below ensure that data are gathered and presented in accordance with procedures appropriate for its intended use, that the data is of known and documented quality, and are able to withstand scientific and legal scrutiny.

Precision is an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. Precision is expressed either as Relative Standard Deviation (RSD) for greater than two measurements or as Relative Percent

Difference (RPD) for two measurements. Precision is determined, in part, by analyzing data from aggregate LCS results, MS, MSD, and MD. For radiochemical determinations, counting statistics can also provide an estimate of uncertainty.

Precision also refers to the measurement of the variability associated with the entire process, from sampling to analysis. Total precision of the process can be determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations.

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. It reflects the total error associated with a measurement.

Both random and systematic errors can affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100). Accuracy is determined, in part, by analyzing data from LCS, MS, and MSD. For radiochemical determinations, counting statistics can also provide an estimate of uncertainty.

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result is representative of the constituent concentration in the sample matrix. STL makes every effort to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before sub-sampling.

Completeness is defined as the percentage of measurements that are judged valid or useable. Factors negatively affecting completeness include the following: sample leakage or breakage in transit or during handling, loss of sample during laboratory analysis through accident or improper handling, improper documentation such that traceability is compromised, or sample result is rejected due to failure to conform to QC specifications. A completeness objective of greater than 90% of the data specified by the statement of work is the goal established for most projects.

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (e.g., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

4.5. Subcontracting

Subcontracting must be arranged with the documented consent of the client, in a timely response which shall not be unreasonably refused. All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. The originating laboratory shall obtain proof of certification from the subcontract facility, and retain this information in STL records. Where applicable, specific QC guidelines, QAPPs, and/or SAPs are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC). It is not acceptable to subcontract work outside of STL without attempting to negotiate alternative requirements with the client and/or the proposed STL subcontract lab.

Non-STL subcontract laboratories may receive an on-site audit by a representative of STL's QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements. The originating laboratory may also perform a paper audit of the subcontractor, which would entail reviewing the LQM, the last two PT studies, and a copy of any recent regulatory audits with the laboratory's responses.

Intra-company subcontracting within STL must be arranged with the documented consent of the client, in a timely response which shall not be unreasonably refused. The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

Project reports from both STL and external subcontractors are discussed in Section 5.9.4.

4.6. Purchasing Services and Supplies

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

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents meet with the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with Corporate SOP S-T-001, *Testing Solvents and Acids*.

4.7. Service to the Client

4.7.1. Sample Acceptance Policy

Each STL facility shall maintain a sample acceptance policy that describes compromised sample receipt. Samples shall be considered "compromised" if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservative.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it must be documented in the project records and the client must be contacted for instructions. If the client decides to proceed with the analysis, the project report shall clearly indicate any of the above conditions and the resolution. The PM reviews of daily sample logins must be completed within one business day or prior to sample processing by the laboratory. Specific review criteria are established by each facility to meet specific project needs (e.g., rush samples, hold times, etc..).

4.7.2. Client Confidentiality and Proprietary Rights

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL and client contract) subject to any disclosure required by law or legal process. STL's reports, and the data and information provided therein, are for the exclusive use and benefit of the client, and are not released to a third party without written consent from the client.

4.8. Complaints

STL believes that an effective client complaint handling process has important business and strategic value. Listening to and documenting client's concerns captures "client knowledge" that helps to continually improve the process and outpace the competition. Implementing a client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

Client complaints shall be documented, communicated to management, and addressed promptly and thoroughly. Client complaints are documented by the employee receiving the complaint. The documentation can take the form of a corrective action report (as described in Section 4.10) or in a format specifically designed for that purpose. The Laboratory Director, PM, Customer Service Manager, and QA Manager are informed of all client complaints, and assist in resolving the complaint.

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

The number and nature of client complaints shall be reported to the Quality Director in the QA Monthly report submitted by each facility. The overall number of complaints received per facility is tracked and the appropriateness of the response to client complaints is assessed. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Management Systems Review. Most client feedback comes either verbally or in writing to STL employees. However, STL also uses a number of additional mechanisms to obtain client feedback including the ACIL Seal of Excellence survey, a biannual customer satisfaction survey, and a response card system. Each of these is monitored for trends and opportunities for improvement.

4.9. Control of Non-conformances

Each STL facility shall have a procedure to control and document non-conformances. Non-conformances include any out of control occurrence. Non-conformances may relate to client specific requirements, procedural requirements, or equipment issues. All non-conformances in the laboratory are documented at the time of their occurrence.

All non-conformances that affect a sample and/or sample data become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If

the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, Manager, PM, Laboratory Director, or QA Manager for direction may be required. All records of reanalysis are kept with the project files.

Where non-conformances specifically affect a client's sample and/or data, the client shall be informed and action must be taken. Action can take the form of reporting and flagging the data, and including a description of the non-conformance in the project narrative or cover letter.

4.10. Corrective Action

4.10.1. General

Each STL facility shall maintain an established, documented corrective action process. Each corrective action is thoroughly investigated, and the investigation, outcome of the investigation, action taken, and follow-up is documented. Corrective action reports are reviewed, approved, and maintained by the QA department.

4.10.2. Initiation

Any employee in STL shall be authorized to initiate a corrective action. The initial source of corrective action can also be external to STL (e.g., corrective action due to client complaint, regulatory audit, or proficiency test). When a problem that requires corrective action is identified, the following items are identified by the initiator on the corrective action report: the nature of the problem, the name of the initiator, and the date. If the problem effects a specific client project, the name of the client and laboratory project number is recorded, and the PM is informed immediately.

4.10.3. Cause Analysis

The corrective action process must be embarked upon as a joint, problem solving, constructive effort. Identification of systematic errors, or errors that are likely to occur repetitively due to a defect or weakness in a system, is particularly valuable in maintaining an environment of continuous improvement in laboratory operations.

When a corrective action report is initiated, the initiator works with the affected employee(s) and/or department(s) to identify the root cause of the problem. An essential part of the corrective action process is to identify whether the problem occurred due to a systematic or isolated error.

If the initiator of the corrective action report is uncertain as to what would constitute appropriate corrective action or is unable to resolve the situation, the problem is identified to the Supervisor, Manager, Laboratory Director or the QA Manager who

provides assistance in the corrective action process. The root cause of the problem and associated cause analysis is documented.

4.10.4. Corrective Action

Once the root cause of a problem is identified, the initiator and affected employee(s) and/or department(s) examine potential actions that will rectify the present problem to the extent possible, and prevent recurrence of future, similar occurrences. An appropriate corrective action is then recommended. The corrective action must be appropriate for the size and nature of the issue.

If the corrective action concerns a specific project related issue, the PM or Customer Service Manager approves the corrective action before its implementation.

Implementation of the corrective action and the date of implementation are documented on the corrective action report.

If a corrective action is related to a specific project report, it is included in the project file. An essential part of the corrective action process is communication and awareness of the problem, the cause, and the action taken to prevent future occurrences and/or rectify the immediate problem.

4.10.5. Monitoring Corrective Action

The QA department reviews corrective action reports and selects one or more of the more significant corrective actions for inclusion in the annual systems audit. The QA Department also may implement a special audit. The purpose of inclusion of the corrective action process in both routine and special audits is to monitor the implementation of the corrective action and to determine whether the action taken has been effective in overcoming the issue identified.

4.11. Preventative Action

Each STL facility shall maintain an established, documented preventative action process. Preventative action is identifying process weaknesses which have the potential to lead to failure(s). Preventative action includes analysis of the quality system to detect, analyze, and eliminate potential causes of non-conformances. When potential problems are identified, preventative action is initiated to effectively address the problem to eliminate or reduce the risk identified.

4.12. Records

4.12.1. Record Types

Table 4 STL Record Types[1]

Raw Data	Controlled Documents	QA Records	Project Records	Administrative Records
See Section 3. Terms and Definitions	LQM	Audits/ Responses	COC Documentation	Accounting
	QMP	Certifications	Contracts and Amendments	EH&S Manual, Permits, Disposal Records
	SOPs	Corrective Action	Correspondence	Employee Handbook
		Logbooks[2]	QAPP	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)
		Method & Software Validation, Verification data	SAP	
		Standards Certificates	Telephone Logbooks	Technical and Administrative Policies
		Technical Training Records		

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

4.12.2. Record Retention

Table 5 outlines STL's standard record retention time. For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 6 have lengthier retention requirements and are subject to the requirements in Section 4.12.3.

Table 5 STL Record Retention

Record Type[1]		Archival Requirement
Raw Data	All*	5 Years from analytical report issue
Controlled Documents	All*	5 Years from document retirement date
QA	All*	5 Years from archival
Project	All*	5 Years from analytical report issue
Administrative	Personnel/Training	7 years
	Accounting	See Accounting and Control Procedures Manual

[1] Record Types encompass hardcopy and electronic records.
* Exceptions listed in Table 6.

Table 6 Special Record Retention Requirements

Program	[1]Retention Requirement
Colorado – Drinking Water	10 years
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Minnesota – Drinking Water	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
OSHA - 29 CFR Part 1910	30 years
Pennsylvania – Drinking Water	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

4.12.3. Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the STL standard record retention time. These are detailed in Table 6 with their retention requirements. In these cases, the longer retention requirement must be implemented and noted in the archive or addressed in a facility specific records retention procedure. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

4.12.4. Archives and Record Transfer

Archives must be indexed such that records are accessible on either a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented. On-site and/or off-site facilities may be used.

STL ensures that all records are maintained as required by the regulatory guidelines and per the QMP upon facility location change or ownership transfer. Upon STL facility location change, all archives are retained by STL in accordance with the QMP. Upon ownership transfer, record retention requirements shall be

addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established.

4.13. Internal Audits

4.13.1. Audit Types and Frequency

A number of types of audits shall be performed at STL. Audit type and frequency are categorized in Table 7.

Table 7 Audit Types and Frequency

Audit Type	Performed by	Frequency
Systems	QA Department or Designee	Annual
Data	QA Department or designee	**Data Report Review:** As necessary to ensure an effective secondary review process
		Analyst Data Audits: 100% of all analysts annually
		Electronic Data Audits: 100% of all organic instruments
Special	QA Department or Designee	As Needed

4.13.2. Systems Audits

Facility systems audits are technical in nature and are conducted on an ongoing basis by the QA Manager or his/her designee at each facility. Systems audits cover all departments of the facility, both operational and support.

The audit report is issued by internal auditor within 30 calendar days of the audit. The audit report is addressed to the Laboratory Director, and copied to the Corporate Quality Director and General Manager. If the internal audit is performed by someone other than the facility's QA Manager, the report must also be addressed to the QA Manager.

Written audit responses are required within 30 calendar days of the audit report issue. The audit response follows the format of the audit report, and corrective actions and time frames for their implementation are included for each deficiency. The audit response is directed to all individuals copied on the audit report. Where a corrective action requires longer than 30 days to complete, the target date for the corrective action implementation is stated and evidence of the corrective action is submitted to the QA Department in the agreed upon time frame.

4.13.3. Data Audits

Data audits assess the level of customer service, SOP compliance, regulatory compliance, accuracy and completeness of test results and reports, documentation, and adherence to established QC criteria, laboratory SOPs, technical policy, and project specific QC criteria. Data Audits may be accomplished through electronic instrument data audits, analyst data authenticity audits or through final project report reviews.

Records of the data audits shall be kept, and the frequency of data audits shall be included in the monthly QA report. In performing data audits, it is essential that data be assessed in terms of differentiating between systematic and isolated errors. Upon noting anomalous data or occurrences in the data audits, the QA Department is responsible for seeking clarification from the appropriate personnel, ascertaining whether the error is systematic or an isolated error, and overseeing correction and/or revision of the project report if necessary. Errors found in client project reports are revised and the revision sent to the client. The QA Department is also responsible for assisting in the corrective action process where a data audit leads to identification of the need for process evaluation and change.

Where specific clients and regulatory programs require more frequent data auditing, the individual facility must meet the data auditing frequency for that program.

4.13.3.1. Data Authenticity Audits

Data authenticity audits shall be performed on 100% of all analysts by the QA department or a designee independent from the operations. Performing data authenticity checks will typically include verifying raw data, evaluating calculation tools and independently reproducing the final results and comparing it to the hardcopy on randomly selected batches of data. Analyst data audits must include spot-checking of manual integrations by QA personnel in order to determine that the manual integration is appropriate and documented according to Section 5.3.6. The laboratory will report the percentage of analysts reviewed (for the year) in their monthly QA report and should average about 8% per month.

4.13.3.2. Electronic Data Audits

Electronic data audits shall be performed on 100% of all organic instruments by the QA department or a designate independent from the operations. This may include Mint Miner® scanning of randomly selected batches of electronic data followed by a chromatography system review. The laboratory will report the percentage of instruments reviewed (for the year) in their monthly QA report and should average about 8% of instruments per month. Electronic data audits must include spot-checking of manual integrations by QA personnel in

order to determine that the manual integration is appropriate and documented according to Section 5.3.6.

4.13.3.3. Final Report Reviews

The frequency of auditing final reports depends on the effectiveness of the laboratory's secondary review process. If the laboratory infrequently finds report errors or there is a low percentage of revised reports due to analytical error, audits may be less frequent.

4.13.4. Special Audits

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

4.14. External Audits

STL facilities are routinely audited by clients and external regulatory authorities. STL is available for these audits and makes every effort to provide the auditors with the personnel, documentation, and assistance required by the auditors. STL recommends that the audits be scheduled with the QA Department so that all necessary personnel are available on the day of the audit.

4.15. Management Reviews

4.15.1. QA Reports to Management

A monthly QA report shall be prepared by the QA Manager or their designee and forwarded to the Laboratory Director and the Quality Director. The reports include statistical results that are used to assess the effectiveness of the quality system. At a minimum, the content of the monthly report is shown in Figure 2.

A Corporate QA Monthly Report containing a compilation of the Facility QA reports statistics, information on progress of the Corporate QA program, and a narrative outlining significant occurrences and/or concerns shall be prepared by the Quality Director and forwarded to the General Manager of Operational and Technical Services and the COO.

4.15.2. Management Systems Review

Each STL facility shall perform a management quality system review annually in accordance with the corporate standard operating procedure. This will synchronize quality planning with fiscal year planning. The management quality system will assess the adequacy of the laboratory's quality system and plan any changes in

laboratory organization, policies, practices, certifications, accreditations in order to achieve operational efficiencies, meet regulatory requirements and client expectations.

4.15.3. Monthly QA Report and Metrics

Each laboratory's QA Manager will prepare a monthly QA report which is due by the third working day of the month. The report will be sent to the Laboratory Director, General Manager and Quality Director. The report will contain a narrative summary and metrics spreadsheet. At a minimum, the report content will contain the items listed below. During the course of the year, the Laboratory Director, General Manager or Quality Director may request that additional information be added to the report.

Figure 2 Monthly QA Report Format

1	**Audits**
	Internal System Audits External System Audits
2	**Revised Reports**
	Revised Reports Corrective/Preventive action measures
3	**Client Complaints**
	Describe situations and resolutions in progress
4	**Certifications/Approvals**
	Issues/changes Lapses/potential revocations
5	**Proficiency Testing**
	Study participation and scores Combined PT scores Repeat failures
6	**SOP Status**
	Report the percentage of SOPs that have been revised or reviewed within the last 24 months
7	**Miscellaneous QA and Operational Issues**
	Narrative outlining improvements, regulatory compliance issues and general concerns
Appended	**Metrics Spreadsheet**
	Summarize metrics in the template provided by the Corporate Quality Director

5. Technical Requirements

5.1. Personnel

5.1.1. General

STL management believes that its highly qualified, ethical and professional staff is the single most important aspect in assuring the highest level of data quality and service in the industry.

STL staff consists of over two thousand professionals and support personnel that include the following positions:

- General Manager
- Customer Service Manager
- Quality Assurance (QA) Manager
- Laboratory Director
- Technical Director
- Laboratory Manager
- Department Supervisor
- Information Technology Manager
- Human Resources Manager
- Project Manager
- Department Manager
- Analyst
- Sample Custodian
- Technician
- Quality Assurance Specialist
- Data Review Specialist
- Information Technology Specialist

5.1.2. Training

STL is committed to furthering the professional and technical development of employees at all levels. Minimum training requirements for STL employees are outlined in Table 8.

Table 8 STL Employee Minimum Training Requirements

Required Training	Time Frame*	Employee Type
Environmental Health & Safety	Month 1	All
Ethics – New Hires	1-2 days of hire	All
Ethics - Comprehensive Data Integrity Quality Assurance	30 days of hire (All training)	All Technical and PMs All
Ethics Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

**From date of initial employment unless otherwise indicated.*

Technical training is accomplished within each laboratory by management to ensure method comprehension. All new personnel shall be required to demonstrate competency in performing a particular method by successfully completing an Initial Demonstration of Capability (DOC) before conducting analysis independently on client samples.

DOCs are performed by analysis of four replicate QC samples. Results of successive LCS analyses can be used to fulfill the DOC requirement. The accuracy and precision, measured as average recovery and standard deviation (using n-1 as the population), of the 4 replicate results are calculated and compared to those in the test method (where available). If the test method does not include accuracy and precision requirements, the results are compared to target criteria set by the laboratory. The laboratory sets the target criteria such that they reflect the DQOs of the specific test method or project. A DOC Certification Statement is recorded and maintained in the employee's training or personnel file. Figure 3 shows an example of a DOC Certification Statement.

The following evidence must be on file at the laboratory for each technical employee:

- DOC.
- The employee has read and understood the latest version of the laboratory's quality documentation.
- The employee has read and understood the latest, approved version of all test methods and/or SOPs for which the employee is responsible.
- Annual evidence of continued DOC that may include successful analysis of a blind sample on the specific test method, or a similar test method, or an annual DOC, or four successive, successful LCSs.
- An Ethics Agreement signed by each staff member (renewed each year).
- A Confidentiality Agreement signed by each staff member (renewed each year).

Figure 3 Example Demonstration of Capability Certification Statement

Demonstration of Capability
Certification Statement

Date: Matrix:
Laboratory Name: Method:
Laboratory Address:
Analyst Name:

We the undersigned certify that:

1. The analyst identified above, using the cited test method, which is in use at this facility for the analysis of samples under the National Environmental Laboratory Accreditation Program, has met the Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification.
3. Copies of the test method and SOP are available for all personnel on site.
4. The data associated with the DOC are true, complete and representative.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is available for review by authorized inspectors.

Laboratory Manager/Supervisor _____ Signature _____ Date _____

5.1.3. Ethics Policy

Establishing and maintaining a high ethical standard is an important element of a quality system. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times, STL has established an *Ethics Policy*, P-L-006, and an Ethics Agreement (Figure 4). Each employee shall sign the Ethics Agreement, signifying agreed compliance with its stated purpose. The ethics agreement is required to be re-signed on an annual basis.

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

Ethics is also a major component of STL's quality and data integrity systems. Each employee must be introduced to STL's Ethics Policy within 1-2 days of hire; and receive the Comprehensive Ethics, Data Integrity Training and Quality Training within 30 days of hire. Annually, Ethics Refresher Training will be provided. Employees must be trained as to the legal and environmental repercussions that result from data misrepresentation. A data integrity hotline is maintained by STL and administered by the Quality Director.

Figure 4 STL Ethics Agreement

I understand that STL is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:

- I will not intentionally report data values that are not the actual values obtained;
- I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;
- I will not intentionally misrepresent another individual's work;
- I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers;
- I agree to inform my Supervisor of any accidental reporting of non-authentic data by me in a timely manner; and I agree to inform my Supervisor of any accidental or intentional reporting of non-authentic data by other employees;
- If a supervisor or a member of STL management requests me to engage in or perform an activity that I feel is compromising data validity or quality, I will not comply with the request and will report this action immediately to a member of senior management, up to and including the President of STL; and
- I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the Severn Trent family of companies.

As a STL employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE _____ Date _____

Supervisor/Trainer: _____ Date _____

5.2. Facilities

Each STL facility must be secure and access must be controlled and documented. Access is controlled by various measures including locked doors, passwords, electronic access cards, security codes, and staffed reception areas. All visitors sign in and are escorted by STL personnel while at an STL facility.

STL's facilities are designed for efficient, automated high-quality operations. All laboratories are equipped with Heating, Ventilation, and Air Conditioning (HVAC) systems appropriate to the needs of environmental testing laboratories. Environmental conditions in the facilities, such as hood flow, are routinely monitored and documented. Table 9 summarizes the square footage at each STL facility.

All STL facilities are equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. STL also provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, respirators, etc.

Table 9 STL Laboratory Square Footage

Facility	Square Footage
STL Austin	27,000
STL Billerica	3,5,000
STL Buffalo	32,000
STL Burlington	36,000
STL Chicago	48,500
STL Connecticut	17,000
STL Corpus Christi	14,000
STL Denver	54,000
STL Edison	42,000
STL Houston	28,000
STL Knoxville	29,000
STL Los Angeles	27,000
STL Miami	17,000
STL Mobile	14,000
STL Newburgh	8,000

Facility	Square Footage
STL North Canton	53,000
STL Pensacola	25,000
STL Pittsburgh	30,000
STL Richland	33,000
STL Sacramento	66,000
STL Savannah	55,000
STL San Francisco	21,000
STL Seattle	20,000
STL St. Louis	31,000
STL Tallahassee	22,000
STL Tampa	14,000
STL Valparaiso	14,500
STL Westfield	10,000
AEL, Phoenix	24,000

5.3. Test Methods

5.3.1. Method Selection

Most of the test methods performed at STL originate from test methods published by a regulatory agency such as the U.S. EPA and other state and federal regulatory agencies. These include, but are not limited to, the following published compendiums of test methods:

Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999

Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, August 1980.

Eastern Environmental Radiation Facility Radiochemistry Procedures Manual, EPA, PB84-215581, June 1984.

HASL-300 28th Edition, Environmental Measurements Laboratory (EML), 1997.

Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fourth Edition, EPA/600/4-90/027F, August 1993.

Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, EPA-821-R-02-012, October 2002.

Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, Fourth Edition, EPA-821-R-02-013, October 2002.

Analytical Method for Determination of Asbestos Fibers in Water, EPA-600/4-83, September 1983.

Determination of Asbestos Structures Over 10-mm in Length in Drinking Water, EPA-600/R-94-134, June 1994.

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water.

Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.

Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.

Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.

Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992.

NIOSH Manual of Analytical Methods, 4th ed., August 1994.

Statement of Work for Inorganics Analysis, ILM04.1, USEPA Contract Laboratory Program Multi-media, Multi-concentration.

Statement of Work for Organics Analysis, OLM04.2, USEPA Contract Laboratory Program, Multi-media, Multi-concentration.

Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLMO4.1, USEPA Contract Laboratory Program, September 1998.

Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.

National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.

5.3.2. SOPs

Each STL facility shall maintain an SOP Index for both Method and Process SOPs. Method SOPs are maintained to describe a specific test method. Process SOPs are maintained to describe functions and processes not related to a specific test method.

Method SOPs contain the following information:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 5).

Identification of Test Method
Applicable Matrix
Reporting Limit
Scope and Application, including test analytes
Summary of the Test Method
Definitions
Interferences
Safety
Equipment and Supplies
Reagents and Standards
Sample Collection, Preservation, Shipment and Storage
Quality Control

Calibration and Standardization
Procedure
Calculations
Method Performance
Pollution Prevention
Data Assessment and Acceptance Criteria for Quality Control Measures
Corrective Actions for Out-of-Control Data
Contingencies for Handling Out-of-Control or Unacceptable Data
Waste Management
References
Tables, Diagrams, Flowcharts and Validation Data

Process SOPs may contain the following information:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 5).

Scope
Summary
Definitions
Responsibilities

Safety
Procedure
References
Tables, Diagrams and Flowcharts

The QA Department is responsible for maintenance of SOPs, archival of SOP historical revisions, maintenance of an SOP index, and records of controlled distribution. SOPs, at a minimum, must undergo periodic review as described the each facility's LQM or SOP. Where an SOP is based on a published method, the laboratory must maintain a copy of the reference method.

Figure 5 Proprietary Information Statement

This documentation has been prepared by Severn Trent Laboratories (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to STL upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

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SOP Appendix

In some cases, a standard laboratory procedure is modified slightly for a specific client or project at the client or regulatory agency's request. In these cases, an Appendix to the SOP may be attached that indicates the modifications to the SOP which are specific to that project. SOP appendices shall not be used to alter test methods required by regulation such that the modifications would result in non-compliance with the regulation.

5.3.3. Method Validation

Laboratory developed methods are validated and documented according to the procedure described in Section 5.3.5.

5.3.4. Method Verification

Method verification is required when a validated standard test method or a method modification is implemented. The level of activity required for method verification is dependent on the type of method being implemented, or on the level of method modification and its affect on a method's robustness. Method modification often takes advantage of a method's robustness, or the ability to make minor changes in a method without affecting the method's outcome. Method verification may require some, but not all, of the activities described in Section 5.3.5.

5.3.5. Method Validation and Verification Activities

Before analyzing samples by a particular method, method validation and/or method verification must occur. A complete validation of the method is required for laboratory developed methods. While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

Determination of Method Selectivity
Method selectivity is demonstrated for the analyte(s) in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

Determination of Method Sensitivity
Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. Each STL facility must document their approach to estimation and/or demonstration of sensitivity. Refer to the Corporate SOP S-Q-003, *Method Detection Limit Studies*, additional information.

Relationship of the Limit of Detection (LOD) to the Quantitation Limit (QL)

Each laboratory shall have a procedure to relate the QL to the LOD (or MDL if appropriate). An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be reported with a qualification that denotes the semi-quantitative nature of the result.

Determination of Interferences
A determination that the method is free from interferences in a blank matrix is performed.

Determination of Range
Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

Initial Demonstration of Capability
DOCs are performed prior to method performance.

Determination of Accuracy and Precision
Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

Documentation of Method
The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Appendix describing the specific differences in the new method is acceptable in place of a separate SOP.

Continued Demonstration of Method Performance
Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCSs and Method Blanks.

5.3.6. Data Review

All data, regardless of regulatory program or level of reporting, shall be subject to a thorough review which involves a primary, secondary, and completeness review process. All levels of the review must be documented.

Primary Review

The primary review is often referred to as a "bench-level" review. In most cases, the analyst who generates the data (e.g., logs in, prepares and/or runs the samples) is the primary reviewer. In some cases, an analyst may be reducing data for samples run by an auto-sampler set up by a different analyst. In this case, the identity of both the analyst and the primary reviewer is identified in the raw data.

One of the most important aspects of primary review is to make sure that the test instructions are clear, and that all project specific requirements have been understood and followed.

Once an analysis is complete, the primary reviewer must ensure that:

- Sample preparation information is complete, accurate, and documented.
- Calculations have been performed correctly.
- Quantitation has been performed accurately.
- Qualitative identifications are accurate.
- Manual integrations are appropriate.
- Data flags to indicate manual integrations are recorded.
- Manual integrations are authorized by a date and signature or initials (hardcopy or electronic) of primary analyst.
- Client specific requirements have been followed.
- Method and process SOPs have been followed.
- Method QC criteria have been met.
- QC samples are within established limits.
- Dilution factors are correctly recorded and applied.
- Non-conformances and/or anomalous data have been properly documented and appropriately communicated.
- COC procedures have been followed.
- All unused portions of hardbound logbooks are 'Z'ed out; corrections are made with a single line drawn through the error and are dated and initialed.
- Primary review is documented by date and initials/signature of primary analyst.

Any anomalous results and/or non-conformances noted during the Primary Review are documented on a data review checklist (defined by each facility) communicated to the Supervisor and the PM for resolution. Resolution can require sample reanalysis, or it may require that data be reported with a qualification. Non-conformances are documented per Section 4.9.

Secondary Review

The secondary review shall be a complete technical review of a data set. The secondary review must be documented and the secondary reviewer identified. The following items are reviewed:

- Qualitative Identification
- Quantitative Accuracy
- Calibration
- QC Samples
- Method QC Criteria
- Adherence to method and process SOPs
- Accuracy of Final Client Reporting Forms

- Manual Integrations – Minimal requirement is to spot-check raw data files for manual integration, as verified by date and initials or signature (hardcopy or electronic) of secondary data reviewer. Some regulatory programs require 100% secondary review of manual integrations.
- Completeness
- Special Requirements/Instructions

If problems are found during the secondary review, which are documented on the data review checklist (defined above), the reviewer must work with the appropriate personnel to resolve them. If changes are made to the data, such as alternate qualitative identifications, identifications of additional target analytes, re-quantitation, or re-integration, the secondary reviewer must contact the laboratory analyst and/or primary reviewer of the data so that the primary analyst and/or reviewer is aware of the appropriate reporting procedures.

Completeness Review

The completeness review shall include the generation of a project narrative and/or cover letter which outlines anomalous data and non-compliances using project narrative notes and non-compliance reports generated during the primary and secondary review. The completeness review addresses the following items:

- Is the project report complete?
- Does the data meet with the client's expectations?
- Were the data quality objectives of the project met?
- Are QC outages and/or non-conformances approved and appropriately explained in the narrative notes?

5.3.7. Data Integrity and Security

This section details those procedures that are relevant to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data.

Security and Traceability

Access to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data must be both controlled and recorded. There are various systems at STL to which this applies, which include the Laboratory Information Management System (LIMS), as well as specific systems such as chromatography data systems.

Control of the system is accomplished through limitation of access to the system by users with the education, training and experience to perform the task knowledgeably and accurately. System users are granted privileges that are commensurate with their experience and responsibilities.

Computer access is tracked by using unique login names and passwords for all employees that have access to the computer system. "General" or "multi-user" account access to computer systems that collect, analyze and process raw instrumental data, and those that manage and report data shall not be permitted. Entries and changes are documented with the identity of the individual making the entry, and the time and date. Where a computer system is processing raw instrumental data, the instrument identification number as described in Section 5.4.1 is recorded. Many of these systems have the capability of maintaining audit trails to track entries and changes to the data. This function is activated on any computer system that has that capability.

STL requires that all sensitive computer systems, defined as LIMS servers and other servers of critical importance, be locked in a secured room. Access must be limited only to employees who need physical access to those systems. This room must also provide climate control within the parameters provided by the vendor of the secured equipment.

Verification

All commercially obtained software shall be verified prior to use and after version upgrade. Verification involves assessing whether the computer system accurately performs its intended function. Verification generally is accomplished by comparing the output of the program with the output of the raw data manually processed, or processed by the software being replaced. The records of the verification are required to contain the following information: software vendor, name of product, version, comparison of program output and manual output, raw data used to verify the program, date, and name of the individual performing the verification. Records of verification are retained as QC records.

Validation

Software validation involves documentation of specifications and coding as well as verification of results. Software validation is performed on all in-house programs. Records of validation include original specifications, identity of code, printout of code, software name, software version, name of individual writing the code, comparison of program output with specifications, and verification records as specified above. Records of validation are retained as QC records.

Auditing

The QA Departments system audits includes review of the control, security, and tracking of IT systems and software.

Version Control

The laboratory shall maintain copies of outdated versions of software and associated manuals for all software in use at the laboratory for a period of five years from its retirement date. The associated hardware, required to operate the software, must also be retained for the same time period.

5.4. Equipment

5.4.1. Equipment Operation

STL is committed to routinely updating and automating instrumentation. STL facilities maintain state of the art instrumentation to perform the analyses within the QC specifications of the test methods. Each STL facility shall maintain an equipment list that must include the following information:

- Date Installed or year placed in service
- Manufacturer's Name, Model Number, Serial Number
- Current Location
- Preventative Maintenance Schedule

All equipment is subject to rigorous checks upon its receipt, upgrade, or modification to establish that the equipment meets with the selectivity, accuracy, and precision required by the test method for which it is to be used. All manufacturer's operations and maintenance manuals are kept up to date and accessible for the use of the equipment operator. Documentation of equipment usage is maintained using analytical run and maintenance logbooks or the electronic versions of said documents.

5.4.2. Equipment Maintenance

Each STL facility must employ a system of preventative maintenance in order to ensure system up time, minimize corrective maintenance costs and ensure data validity. All routine maintenance is performed as recommended by the manufacturer and may be performed by an analyst, instrument specialist or outside technician. Maintenance logbooks or electronic records are kept on all major pieces of equipment in which both routine and non-routine maintenance is recorded. Notation of the date and maintenance activity is recorded each time service procedures are performed. The return to analytical control following instrument repair is documented. Maintenance logbooks or electronic records are retained as QA records.

Maintenance contracts are held on specific pieces of equipment where outside service is efficient, cost-effective, and necessary for effective operation of the laboratory.

5.4.3. Equipment Verification and Calibration

All equipment shall be tested upon receipt to establish its ability to meet the QC guidelines contained in the test method for which the instrumentation is to be used. This testing shall be documented. Once an instrument is placed in routine service, ongoing instrument calibration is demonstrated at the appropriate frequency as defined in the test method. Refer to the Corporate Policy P-T-001, *Selection of Calibration Points*, for guidance on using calibration data. Any instrument that is

deemed to be malfunctioning is clearly marked and taken out of service. When the instrument is brought back into control, acceptable performance is documented.

5.5. Measurement Traceability

5.5.1. General

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

Laboratory DI and RO water systems have documented preventative maintenance schedules and the conductivity of the water is recorded on each day of use.

5.5.2. Reference Standards Traceability

The receipt of all reference standards must be documented. ~~References~~ standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All standards should be purchased with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The documentation of standard purity is archived, and references the Standard Identification Number.

All efforts are made to purchase standards that are $\geq$ 97.0% purity or as prescribed by the methods. If this is not possible, the purity is used in performing standards calculations.

The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a different lot is acceptable for use as a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS is used as the second source confirmation.

5.5.3. Reagents

Reagents are, in general, required to be analytical reagent grade unless otherwise specified in method SOPs. Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented.

5.6. Sampling Plans

Sample representativeness and integrity are the foundations upon which meaningful analytical results rely. Where documented and approved SAPs and/or QAPPs are in place, they must be made available to the laboratory before sample receipt, and approved by laboratory management before sample receipt.

5.7. Sample Handling, Transport, and Storage

5.7.1. General

Chain of Custody (COC) can be established either when bottles are sent to the field, or at the time of sampling. STL can provide all of the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory.

Samples are received at the laboratory by a designated sample custodian and a unique Laboratory Project Identification Number is assigned. The following information is recorded for each sample shipment: Client/Project Name, Date and Time of Laboratory Receipt, Laboratory Project Number, and Signature or initials of the personnel receiving the cooler and making the entries.

Upon inspection of the cooler and custody seals, the sample custodian opens and inspects the contents of the cooler, and records the cooler temperature. If the cooler arrival temperature exceeds the required or method specified temperature range by $\pm 2^{o}C$ (for samples with a temperature requirement of $4^{o}C$, a cooler temperature of just above the water freezing temperature to $6^{o}C$ is acceptable); sample receipt is considered “compromised” and the procedure described in Section 4.7.1 is followed. All documents are immediately inspected to assure agreement between the test samples received and the COC.

Any non-conformance, irregularity, or compromised sample receipt as described in Section 4.7.1 must be documented and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the permanent project record.

Samples that are being tested at another STL facility or by an external subcontractor shall be appropriately packaged, and sent out under COC.

Following sample labeling as described in Section 5.7.2, the sample is placed in storage. Sample storage is required to be access-controlled. All samples are stored according to the requirements outlined in the test method and in a manner such that they are not subject to cross contamination or contamination from their environment. Unless specified by method or state regulation, a tolerance range of $4 \pm 2^{o}C$ is used. Sample storage temperatures are monitored daily.

5.7.2. Sample Identification and Traceability

Each sample container shall be assigned a unique Sample Identification Number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a sample identification label.

All unused portions of samples, including empty sample containers, are returned to the secure sample control area.

5.7.3. Sub-sampling

Sample preparation procedures must be referenced in each STL facility's LQM and documented in the laboratory SOPs.

5.7.4. Sample Preparation

Sample preparation procedures must be referenced in each STL facility's LQM and documented in the laboratory SOPs.

5.7.5. Sample Disposal

Each facility shall have an SOP describing sample retention and disposal procedures. Samples should be retained in STL storage facilities for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (example, 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Samples may be returned to the client per written request. Unused portions of samples found or suspected to be hazardous according to state or

federal guidelines may be returned to the client upon completion of the analytical work.

Samples shall be disposed of in accordance with federal, state and local regulations. Each facility must have an SOP detailing the disposal of samples, digestates, and extracts. All laboratories shall remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated).

5.8. Assuring the Quality of Test Results

5.8.1. Proficiency Testing

Each STL facility must analyze Proficiency Test (PT) samples as required for accreditation. As required by NELAC, each STL facility participates in the PT program semi-annually for each PT Field of Testing (FoT) for which it is accredited, according to the NELAC PT FoT published guidelines. Under SDWA, the laboratory also analyzes a PT sample by each method once per year, if the laboratory uses more than one method for the analyte.

In addition to the PT program required for NELAC accreditation, STL participates in a number of additional PT programs, as appropriate for the specific facility.

PT samples must be handled and tested in the same manner (procedural, equipment, staff) as environmental samples. PT test sample data is archived using the requirements for project and raw data record retention.

Each STL facility performing chemical analyses also participates in a double blind proficiency test evaluation annually. An external vendor is contracted to submit double blind samples to the STL facility. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor, who provides a detailed report to the Quality Director and to each of the STL facilities. This is administered as a double blind program in order to assess all facets of STL operations.

5.8.2. Control Samples

Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity, selectivity, and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch. Control samples must be uniquely identified and correlated to unique batches. There are also a number of QC sample types that monitor field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Control sample types and typical frequency of their application are outlined in Table 10. Note that frequency and use of control samples vary with specific regulatory, methodology and project specific criteria.

Table 10 does not define STL's approach to the application of QC samples for each regulatory program or test method.

5.8.3. Calibration

Each STL Facility must define calibration protocols in STL facility SOPs.

5.8.4. Glassware Cleaning

5.8.5. Permitting Departures from Documented Procedure

Each STL facility must have a procedure that defines the process, documentation, and level of authorization required to permit departures from documented procedures.

Table 10 Control Samples

Laboratory QC Sample Type	Use	Required Frequency
Laboratory Control Sample (LCS) (Laboratory Fortified Blank)	Measures accuracy of the method in a blank matrix	1 per batch of 20 or less samples per matrix type per sample extraction or preparation method[1]
Method Blank (MB)	Measures method contribution to any source of contamination	1 per batch of 20 or less samples per matrix type per sample extraction or preparation method[1]
Instrument Blank	Measures instrumental contribution to any source of contamination	As specified in test method
Cleanup Blank	Measures clean up step contribution to any source of contamination	As specified in test method
Storage Blank	Measures storage contribution to any source of contamination (Volatiles only)	As specified in test method or SOP
Control, Brine Control, or Dilution Water	Measures the effect of blank water on test organisms (Aquatic toxicology)	As specified in test method and permit
Reference Toxicant	Measure sensitivity of test organisms (Aquatic toxicology)	Annually
Field QC Sample Type	**Use**	**Typical Frequency**
Matrix Duplicate	Measures the effect of the site matrix on the precision of the method	Per 20 samples per matrix or per SAP/QAPP[1,2]
Matrix Spike	Measures the effect of the site matrix on the accuracy of the method	Per 20 samples per matrix or per SAP/QAPP[1]
Matrix Spike Duplicate	Measures the effect of the site matrix on the precision of method	Per 20 samples per matrix or per SAP/QAPP[1,2]
Equipment Blank (Equipment Rinsate)	Measures field equipment contribution to any source of contamination	Per SAP/QAPP
Trip Blank	Measures shipping contribution to any source of contamination (Volatiles only)	Per Cooler
Field Blank	Measures the field environment contribution to any source of contamination	Per SAP/QAPP
Field Duplicate	Measures representativeness of the sampling and the effect of the site matrix on precision	Per SAP/QAPP

[1] Denotes an STL required frequency
[2] Either an MSD or an MD is required per 20 samples per matrix or per SAP/QAPP.

Where a departure from a documented SOP, test method, or policy is determined to be necessary, or unavoidable, the departure shall be documented and be authorized by the appropriate level of management, which is defined in the policy. In some instances, it is appropriate to inform the client before permitting a departure. Any such occurrence is documented in the cover letter and/or project narrative.

5.8.6. Development of QC Criteria, Non-Specified in Method/Regulation

Where a method or regulation does not specify acceptance and/or rejection criteria, the laboratory must develop a policy for doing so. The policy must address how the laboratory examines the data user's needs and the demonstrated sensitivity, accuracy and precision of the available test methods in determining appropriate QC criteria.

Data users often need the laboratory's best possible sensitivity, accuracy, and precision using a routinely offered test method, or are unsure of their objectives for the data. For routine test methods that are offered as part of STL's standard services, the laboratory bases the QC criteria on statistical information such as determination of sensitivity, historical accuracy and precision data, and method verification data. The method SOP includes QC criteria for ongoing demonstration that the established criteria are met (e.g., acceptable LCS accuracy ranges, precision requirements, method blank requirements, initial and continuing calibration criteria, etc.).

In some cases, a routine test method may be far more stringent than a specific data user's needs for a project. The laboratory may either use the routinely offered test method, or may opt to develop an alternate test method based on the data user's objectives for sensitivity, accuracy, and precision. In this case, it can be appropriate to base the QC criteria on the data user's objectives, and demonstrate through method verification and ongoing QC samples that these objectives are met.

For example, a client may require that the laboratory test for a single analyte with specific DQOs for sensitivity, accuracy, and precision as follows: Reporting Limit of 10 ppm, accuracy $\pm$25%, and RSD of less than 30%. The laboratory may opt to develop a method that meets these criteria and document the results of the Method Blanks, MDL study, and LCSs that the method satisfies those objectives. In this case, both the method and the embedded QC criteria have been based on the client's DQOs.

In some cases, the data user needs more stringent sensitivity, accuracy, and/or precision than the laboratory can provide using a routine test method. In this case, it is appropriate that the laboratory provide documentation of the sensitivity, accuracy, and precision obtainable to the data user and let the data user determine whether to use the best available method offered by the laboratory, or determine whether method development or further research is required.

5.9. Project Reports

5.9.1. General

All STL Project Reports that are generated under NELAC requirements must contain the content as described in Section 5.9.2. The criteria described in Section 5.9.3 and 5.9.4 applies to all Project Reports.

5.9.2. Project Report Content

- Title
- Laboratory Name, Address, Telephone Number, Contact Person
- Unique Laboratory Project Number
- Total Number of Pages (report must be paginated)
- Name and Address of Client
- Client Project Name (if applicable)
- Laboratory Sample Identification
- Client Sample Identification
- Matrix and/or Description of Sample
- Dates: Sample Receipt, Collection, Preparation and/or Analysis Date
- Definition of Data Qualifiers
- Reporting Units
- Test Method

The following are required where applicable to the specific test method or matrix:

- Solid Samples: Indicate Dry or Wet Weight
- Whole Effluent Toxicity: Statistical package used
- If holding time < 72 hours, Sample Collection, Preparation and/or Analysis Time
- Indication by flagging where results are reported below the quantitation limit.

5.9.3. Project Narrative

A Project Narrative and/or Cover Letter shall be included with each project report and at a minimum includes an explanation of any and all of the following occurrences:

- Non-conformances
- “Compromised” sample receipt (see Section 4.7.1)
- Method Deviations
- QC criteria failures

Project Release

The Laboratory Director or his/her designee must authorize the release of the project report with a signature.

Where amendments to project reports are required after issue, these shall be in the form of a separate document and/or electronic data deliverable. The revised report is clearly identified as revised with the date of revision and the initials of the person making the revision. Specific pages of a project report may be revised using the above procedure with an accompanying cover letter indicating the page numbers of the project revised. The original version of the project report must be kept intact and the revisions and cover letter included in the project files. The authorized SOP deviations, non-conformances and QC failures must be covered in the case narrative, cover letter or within the report.

5.9.4. Subcontractor Test Results

Project reports from a subcontract laboratory shall not be altered, and shall be included in original form in the final project report provided by STL. Data from subcontractors' reports may be added to an STL electronic deliverable.

Subcontracted data shall be clearly identified as such, and the name and address of the laboratory performing the test shall be included in the project report. If the report is being generated under NELAC requirements, all information outlined in Section 5.9.2 are required for both the originating laboratory and the subcontracting laboratory.

Data subcontracted within STL may be reported on the originating laboratory's report forms provided the following mandatory requirements are met:

- The name, address, and telephone number of the facility are provided.
- Analytical results produced by the STL intra-company subcontractor are clearly identified as being produced by the subcontractor facility.
- The intra-company subcontractor's original report, including the COC, is retained by the originating laboratory.
- Proof of certification is retained by the originating laboratory.
- All information as outlined in Section 5.9.2 is included in the final report where the report is required to be compliant with NELAC, for both the originating and subcontracting laboratory.

5.9.5. Electronic Data Deliverables

Electronic Data Deliverables (EDD) are routinely offered as part of STL's services. STL offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process outlined in Section 4.4.1. Once the facility has committed to providing diskettes in a specific format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained as a QC record.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

5.9.6. Project Report Format

STL offers a wide range of project reporting formats, including EDDs, short report formats, and complete data deliverable packages modeled on the Contract Laboratory Protocol (CLP) guidelines. More information on the range of project reports available can be obtained by contacting any STL facility. Regardless of the level of reporting, all projects must undergo the levels of review as described in Section 5.3.6.

Appendix: List of Quality System Policies and Procedures

QMP Citation	Description	Reference
1.2	Quality Policy	QMP
4.4	Contract Review	QMP
4.4.2	Project Planning Process	LAB Procedure
4.7.1	Sample Acceptance Policy	LAB Procedure
4.5	Subcontracting	QMP
5.3.2	Approved SOP Listing	LAB Procedure
4.3.2	Document Control	S-Q-001 & Lab Procedure
4.12.2	Record Retention & Purging	QMP
4.6	Purchasing Services and Supplies	QMP
4.7.2	Client Confidentiality	QMP
4.8	Complaints	QMP
4.9	Document and Control of Non-conformances	LAB Procedure
4.10	Corrective Action process	LAB Procedure
4.15.2	Quality Systems Management Review	QMP
4.11	Preventive Action Process	LAB Procedure
4.12.4	Archives and Record Transfer	QMP
4.13	Internal Audits	QMP
4.15	Management Reviews	QMP
5.1.2	Training	QMP
5.1.3	Ethics Policy	P-L-006
5.3.2	SOP Index	LAB Procedure
5.3.5	Method Detection Limit Studies	S-Q-003
5.3.5	Relationship of Limit of Detection to Quantitation Limit	LAB Procedure
5.3.7	Data Integrity and Security	QMP
5.3.6	Data Review	QMP
5.4.1	Equipment Operation	QMP
5.4.1	Equipment Tracking List	LAB Procedure
5.4.2	Equipment Maintenance	QMP
5.4.3	Equipment Verification and Calibration	QMP
5.4.3	Selection of Calibration Points	P-T-001
5.5	Measurement Traceability	QMP
5.5.1	Procedures for Checking Specifications for Ancillary Equipment	LAB Procedure
5.5.2	Reference Standards Traceability	QMP
5.7	Sample Handling, Transport and Storage	QMP
5.7.2	Sample Identification and Traceability	QMP
5.7.3	Subsampling	QMP
5.7.4	Sample Preparation	QMP
5.7.5	Sample Disposal	LAB Procedure
5.8.3	Calibration	LAB Procedure
5.8.4	Glassware Cleaning Procedures	LAB Procedure
5.8.5	Permitting Departures From Documented Procedures	LAB Procedure
5.8.6	Development of QC Criteria, Non-specified in Methods/Regulations	QMP
5.9	Reporting Analytical Results	QMP

Note: Where "QMP" is referenced it indicates the policy or procedure is covered by the QMP and not covered by a corporate procedure, and it does not require a laboratory specific procedure. However, when QMP is listed, the laboratories' may still address it in more detail in their LQM or laboratory quality system procedures. When "LAB Procedure" is indicated, it requires the laboratory to address the item in its LQM or have a have a specific laboratory quality system policy or procedure for that item. Where a procedure number is listed, it refers to a corporate policy or procedure.

Appendix I
TestAmerica Buffalo - Laboratory Quality Manual

(Document on CD-ROM)

LABORATORY QUALITY MANUAL

STL BUFFALO
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Approved by: (Signature / Date)

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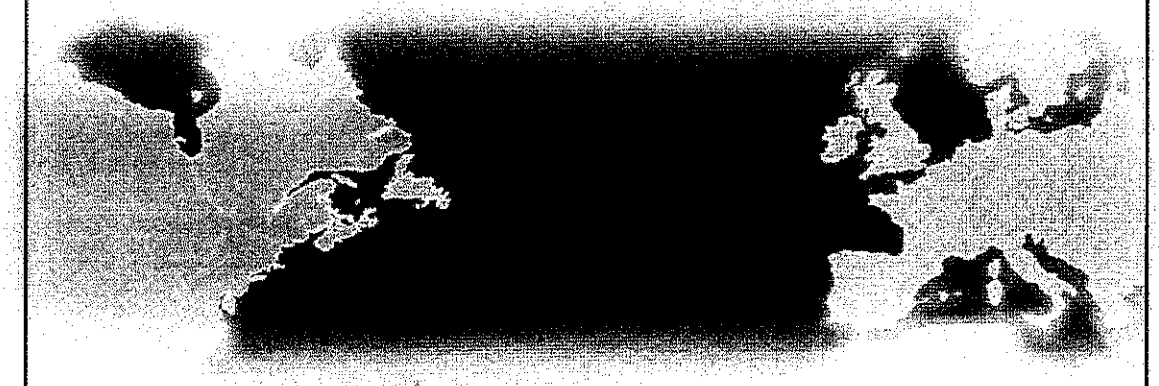
SEVERN TRENT **STL**

Vision

STL will be the recognized industry leader for environmental analysis.



Mission

Through the innovation and dedication of our people, together with the quality of our systems, we will deliver levels of performance that delight our clients, retain the confidence of our stakeholders and enable the profitable growth of our business.

Severn Trent Laboratories

1.0 Introduction, Purpose, and Scope

1.1 STL Overview

STL Buffalo (STL) is a part of Severn Trent Laboratories, a major group of U.S. based companies. STL is a full-service environmental laboratory that provides quality comprehensive and integrated professional analytical services effectively and efficiently. A broad range of environmental testing services are offered that span a variety of matrices including aqueous, saline, solid, tissue and drinking water.

Associated with this activity are services to assure client requirements are known, communicated and satisfactorily addressed, and a deliverables package presenting the analytical results. The laboratory provides expert personnel for supervision, technical consultation, and project review for effective planning and implementation of analytical assignments.

STL operates under the regulations and guidelines of the following federal programs:

- Clean Water Act (CWA)
- National Pollution, Discharge, and Elimination System (NPDES)
- Occupational Safety and Health Administration (OSHA)
- Resource Conservation and Recovery Act (RCRA)
- Safe Drinking Water Act (SDWA)
- Toxic Substances Control Act (TSCA)

STL also provides services under various state and local municipal guidelines. A current table of analytical services and general service listing is presented on STL's website under the MySTL webpage or available from the laboratory. A current listing of STL Buffalo certifications (STLBuffCertList) is maintained by the laboratory on the company network directory. Copies of the actual certificates are available on the STL Buffalo intra-net site (BufNet).

1.2 Quality Assurance Policy

It is STL's policy to:

- Provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- Generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- Promote employee adherence to quality documentation and implementation of Corporate Policies and Procedures
- Provide STL clients with the highest level of professionalism and the best service practices in the industry.
- Build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- Maintain a working environment that fosters open communication with both clients and staff and ensures data integrity.

1.3 Management Commitment to Quality Assurance

STL management is committed to providing the highest quality data and the best service in the environmental testing industry. To ensure that the data produced and reported by STL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL maintains a quality system that is clear, effective, well communicated, and supported at all levels in the company.

Line organizations verify that specifications are achieved; QA organizations assist and provide oversight and verification of processes through planning, reviews, audits, and surveillances. The quality objectives are derived from this Laboratory Quality Manual (LQM), Standard Operating Procedures (SOPs) and Work Instructions.

1.4 Purpose

The purpose of the LQM is to describe STL's Quality System and to outline how that system enables all employees to meet the Quality Assurance (QA) policy. This LQM also describes specific QA activities and requirements and prescribes their frequencies. Roles and responsibilities of management and laboratory staff in support of the Quality System are also defined in this LQM.

1.5 Scope

This LQM is specific to STL Buffalo's quality systems and laboratory operations. All other STL locations have LQMs under the Corporate Quality Management Plan (QMP) or the Corporate QMP itself.

The laboratory is committed to ensuring that resources are available and deployed to meet client expectations. This includes gathering project information prior to sample receipt to ensure client expectations will be met with respect to:

- Sampling containers;
- Analytical methods employed;
- Accuracy and precision;
- Reporting limits;
- Personnel qualifications, training, and experience;
- Calibration and quality control measures employed;
- Regulatory requirements;
- Report contents;
- Supporting documentation, records and evidence; and
- Review of data

1.6 Servicing

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

The laboratory has established procedures for performing and verifying that client servicing meets requirements. Typical services provided are:

- Sample Containers/Supplies – *Container Management: Process Operation/Bottle Order Set-Up (APM-BottleOrder-03)*
- Project QAP preparation – Project Planning Process: *Project Information Requirements (APM-ProjInfo-20)*
- Regulatory advisory functions – Project Planning Process: *Project Information Requirements (APM-ProjInfo-20)*
- Consulting – Project Planning Process: *Project Information Requirements (APM-ProjInfo-20)*

Regulatory and advisory functions are addressed under the same procedures used for project planning.

2.0 References

The following references were used in preparation of this document and as the basis of the STL Quality System:

EPA Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA/G-6, US EPA, Office of Environmental Information, EPA/240/B-01/004, March 2001.

EPA Requirements for Quality Management Plans, EPA QA/R-2, US EPA, Office of Environmental Information, EPA/240,B-01/002 March 2001.

EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, US EPA, Office of Environmental Information, EPA/240/B-01/003, March 2001.

EPA Quality Manual for Environmental Programs, 5360 A1, US EPA Office of Environmental Information – Quality Staff, May 2000.

General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025, December 1999.

Good Automated Laboratory Practices, Principles and Guidance to Regulations for Ensuring Data Integrity in Automated Laboratory Operations with Implementation Guidance, EPA 2185, US EPA Office of Information Resources Management, August 1995.

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Shell for Analytical Chemistry Requirements, US Army Corps of Engineers, December 1998.

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This LQM was written to comply with the National Environmental Laboratory Accreditation Conference (NELAC) standards. Refer to Table 1 for a cross-section comparison of this LQM to the NELAC standards.

Table 1.

Correlation of QAPP Sections with NELAC 5.4.2.3 Quality Manual Requirements

NELAC Chapter 5.4.2.3 Quality Manual	Laboratory Quality Manual Section
a. Quality policy statement, including objectives and commitments	1.2 Quality Assurance Policy 4.2.1 Objectives of the Quality System
b. Organization and management structure	4.1 Organization and Management
c. Relationship between management, technical operations, support services and the quality systems	4.1.2 Roles and Requirements 4.2 Quality System
d. Records retention procedures; document control procedures	4.3 Document Control 4.12.2 Record Retention
e. Job descriptions of key staff and references to job descriptions of other staff	4.1.2 Roles and Requirements
f. Identification of laboratory approved signatories	4.1 Organization and Management
g. Procedures for achieving traceability of measurements	5.5 Measurement Traceability
h. List of all test methods under which the laboratory performs its accredited testing	5.3.1 Method Selection
i. Mechanisms for assuring the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	4.4.2 Project-Specific Quality Planning
j. Reference to the calibration and/or verification test procedures used	5.4.3 Equipment Verification and Calibration 5.3.6.2 Data Review
k. Procedures for handling submitted samples	4.7.1 Sample Acceptance Policy 5.7 Sample Handling, Transport and Storage
l. Reference to the major equipment and reference measurement standards used as well as the facilities and services used in conducting tests	1.6 Servicing 4.1.1 Laboratory Facilities 5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
m. Reference to procedures for calibration, verification and maintenance of equipment	5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
n. Reference to verification practices including inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal QC schemes	5.8.1 Proficiency Testing 5.8.2 Control Samples
o. Procedures for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur	4.9 Control of Non-Conformances 4.10 Corrective Action 4.11 Preventive Action 5.8.6 Permitting Departures from Documented Procedures
p. Laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications	4.4.2 Project-Specific Quality Planning 5.8.6 Permitting Departures from Documented Procedures
q. Procedures for dealing with complaints	4.8 Complaints

Table 1.

Correlation of QAPP Sections with NELAC 5.4.2.3 Quality Manual Requirements

NELAC Chapter 5.4.2.3 Quality Manual	Laboratory Quality Manual Section
r. Procedures for protecting confidentiality and proprietary rights (including national security concerns)	4.7.2 Client Confidentiality and Proprietary Rights
s. Procedures for audits and data review	4.13 Internal Audits 4.14 External Audits 5.3.6 Data Reduction and Review
t. Process/procedures for establishing that personnel are adequately experienced in duties they are expected to carry out and are receiving any needed training	5.1.2 Training
u. Ethics policy statement developed by the laboratory and training personnel in their ethical & legal responsibilities	5.1.3 Ethics Policy
v. Reference to procedures for reporting analytical results	5.3.6 Data Reduction and Review 5.9 Project Reports
w. Table of contents, listing reference, glossaries and appendices	TOC Table of Contents Appendix List of Cited SOPs and Work Instructions

3.0 Terms and Definitions

Accuracy: The degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Audit: A systematic evaluation to determine the conformance to specifications of an operational function or activity.

Batch: Environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of 1 to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (e.g., volatile organics, water), the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Chain of Custody (COC): A system of documentation demonstrating the physical possession and traceability of samples.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): Legislation (42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and reauthorization Act of 1986 (SARA), 42 U.S.C. 9601et seq.

Compromised Sample: A sample received in a condition that jeopardizes the integrity of the results. See Section 4.7.1 of this LQM for a description of these conditions.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products.

Confirmation: Verification of the presence of a component using an additional analytical technique. These may include second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures.

Corrective Action: Action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Demonstration of Capability (DOC): Procedure to establish the ability to generate acceptable accuracy and precision.

Document Control: The act of ensuring that documents (electronic or hardcopy and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed

ERA Sample: A control sample obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Equipment Blank (EB): A portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Rinsate.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): Legislation under 7 U.S.C. 135 et seq., as amended.

Federal Water Pollution Control Act (Clean Water Act, CWA): Legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat. 816.

Field Blank (FB): A blank matrix brought to the field and exposed to field environmental conditions.

Field Duplicate (FD): Duplicate field-collected sample most commonly used to assess the accuracy of the field collection process.

Field of Testing (FOT): A field of proficiency testing is based on NELAC's categorization of accreditation based on program, matrix and analyte.

Good Laboratory Practices (GLP): Formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729 and required for activities performed under FIFRA and TSCA.

Holding Time: The maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Instrument Blank: A blank matrix that is the same as the processed sample matrix (e.g. extract, digestate, condensate) and introduced onto the instrument for analysis.

Internal Chain of Custody (COC): An unbroken trail of accountability that ensures the physical security of samples, data and records. Internal COC refers to additional documentation procedures implemented within the laboratory that includes special sample storage requirements, and documentation of all signatures and/or initials, dates, and times of personnel handling specific samples or sample aliquots.

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (LCS): A blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure; also referred to as Matrix Spike Blank (MSB); Environmental Resource Associate Sample (ERA).

Laboratory Quality Manual (LQM): A document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system.

Limit of Detection (LOD): The minimum amount of a substance that an analytical process can reliably detect. Also referred to as the Method Detection Limit (MDL)

Matrix: The substrate of a test sample. Common matrix descriptions are defined in Table 2.

Table 2. Matrix Descriptions

Matrix	Description
Aqueous	Aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine source. Includes surface water, groundwater, effluents, leachates and wastewaters.
Drinking Water	Aqueous sample that has been designated a potable water source.
Saline	Aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.
Liquid	Liquid with <15% settleable solids.
Solid	Soil, sediment, sludge, ash, paint chips, filters, wipes or other matrices with ≥15% settleable solids.
Waste	A product or by-product of an industrial process that results in a matrix not previously defined (i.e., drum liquid or oils).
Tissue	Sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Matrix Duplicate (MD): Duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): Field sample to which a known amount of target analyte(s) is added.

Matrix Spike Blank (MSB): A blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure; also referred to as Laboratory Control Sample (LCS).

Matrix Spike Duplicate (MSD): A replicate matrix spike.

Method Blank (MB): A blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Precision: An estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Preservation: Refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

Proficiency Testing: Determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.

Proficiency Test (PT) Sample: A sample, the composition of which is unknown to the analyst, that is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. Also referred to as Performance Evaluation (PE) Sample.

Proprietary: Belonging to a private person or company.

Quality Assurance (QA): An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Assurance (Project) Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

Quality Control (QC): The overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control (QC) Sample: A control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Quality Management Plan (QMP): A formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory to ensure the quality of its product and the utility of the product to its users.

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA/QC.

Quantitation Limit (QL): The minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Raw Data: Any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports specifying inclusion of "raw data" do not need all of the above included, but sufficient information to create the reported data.

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Resource Conservation and Recovery Act (RCRA): Legislation under 42 U.S.C. 321 et seq. (1976).

Safe Drinking Water Act (SDWA): Legislation under 42 U.S.C. 300f et seq. (1974), Public Law 93-523.

Sampling and Analysis Plan (SAP): A formal document describing the detailed sampling and analysis procedures for a specific project.
Selectivity: The capability of a measurement system to respond to a target substance or constituent.

Sensitivity: The difference in the amount or concentration of a substance that corresponds to the smallest difference in a response in a measurement system using a certain probability level.

Spike: A known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Storage Blank: A blank matrix stored for 1 to 2 weeks with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Systems Audit: A thorough, systematic, on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

Test Method: Defined technical procedure for performing a test.

Toxic Substances Control Act (TSCA): Legislation under 15 U.S.C. 2601 et seq., (1976).

Traceability: The property of a result of a measurement that can be related to appropriate international or national standards through an unbroken chain of comparisons.

Trip Blank (TB): A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Verification: Confirmation by examination and provision of evidence against specified requirements.

4.0 Management Requirements

The organizational chart of STL is presented in Figure 1. Corporate employees are located at various STL facilities as outlined in the organizational structure. The organizational chart of STL Buffalo is presented in Figure 2.

4.1 Organization and Management

The Laboratory Director and Quality Assurance Manager are responsible and have the signature authority for approving and implementing this plan. The Laboratory Director and/or his designee also have signatory authority for approval of work and release of reports. The following listing defines those employees that may act as report signatory designees for the Laboratory Director.

Technical Director
Operations Manager
Quality Manager
Customer Service Manager
Project Manager
Project Manager Assistant

Figure 1. STL Organization Chart

Figure 1. STL Organizational Chart

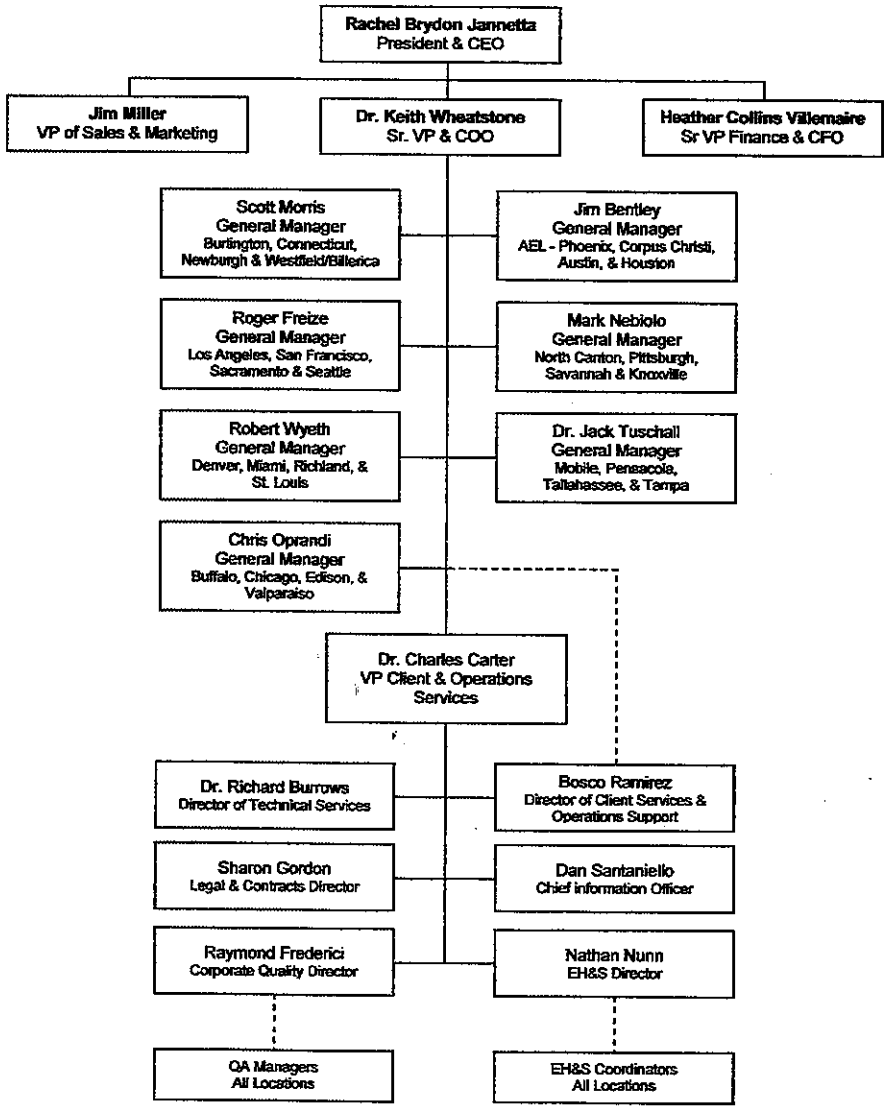
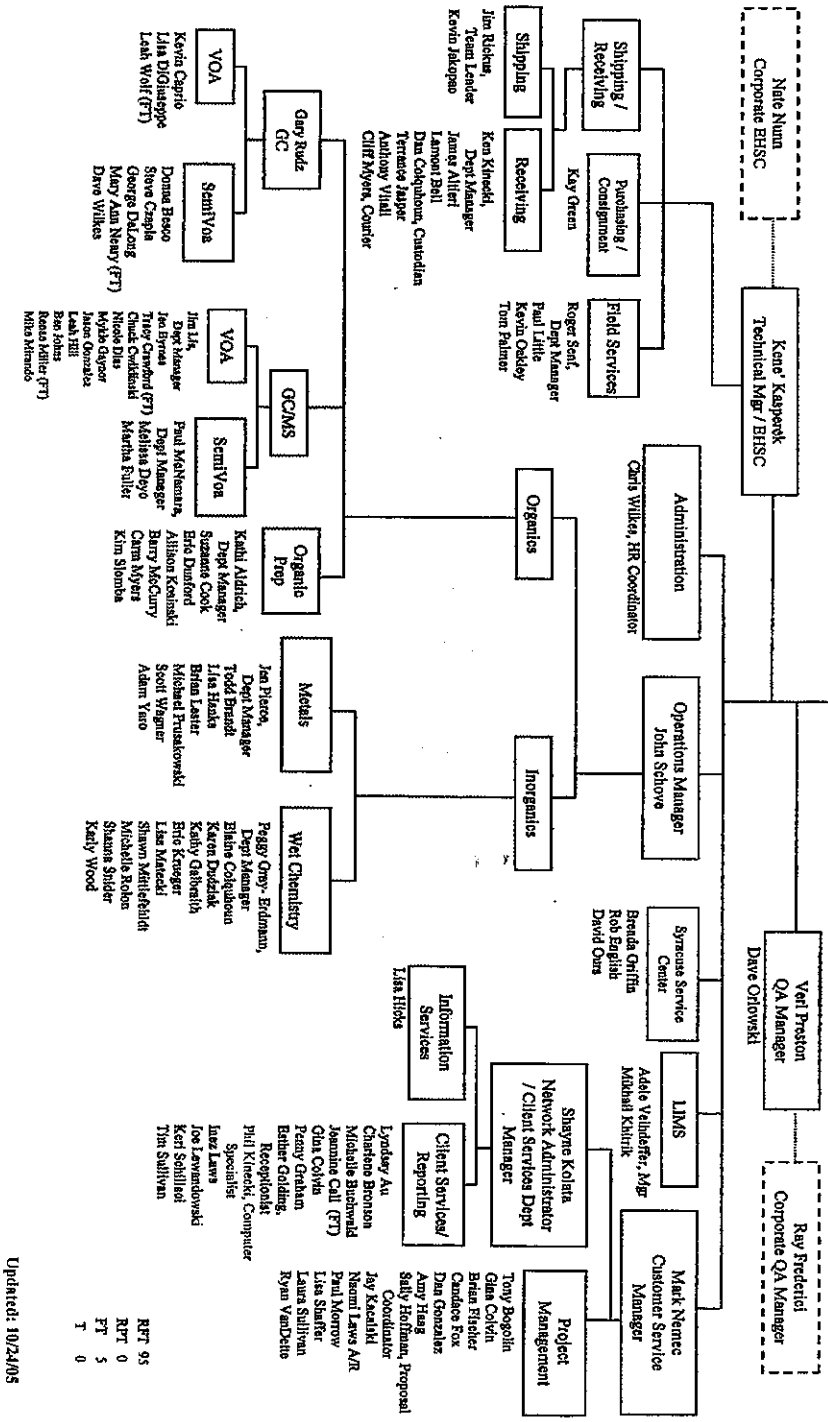


Figure 2. STL Buffalo Organizational Chart, November 2005



4.1.1 Laboratory Facilities

The laboratory is located in Amherst, New York, which is approximately 10 miles from Buffalo, New York, and is staffed by 100 professionals. The laboratory is comprised of 28,000 square feet of state-of-the-art commercial laboratory and office space and houses both inorganic and organic operations. The facility is divided into separate work areas to facilitate sample throughput. These areas include the following:

- Sample receipt and refrigerated storage
- Bottle kit preparation
- Organic and TCLP sample preparation
- Metals digestion
- Wet chemistry laboratory
- Organic instrumentation laboratories
- Metals instrumentation laboratory

The main instrumentation laboratories are equipped with state-of-the-art instrumentation and sufficient duplicate equipment to provide back-up service for most major systems. A listing of laboratory equipment and instrumentation is referenced as STL Buffalo Capital Equipment List, (STLBuffEquipList). Table 3 is a summary of the major laboratory instruments.

Table 3. Major Equipment List

GC	GC/MS	ICP	ICP/MS	CVAA	HPLC	Auto Analyzer	IC	TOC	TOX
20	12	2	1	2	1	4	3	2	1

Each of the laboratory areas has separate heating, ventilation, and air conditioning systems. Non-destructive gas chromatographic detectors, and GC/MS rotary pumps are vented out of the instrumentation through charcoal filters.

4.1.2 Roles and Responsibilities

The specific duties and responsibilities of the Laboratory Director, Technical Director/Environmental Health & Safety Officer, Quality Assurance Manager, Operations Manager, Customer Service Manager/Project Managers, Laboratory Supervisors, Sample Management Coordination, Information Technology Manager, and Chemists/Technicians and Data Packaging Specialists are as follows.

In the absence of any one individual, the staff or assistant within each department is professionally skilled in the ability to administer the function of the administrator or support personnel. This will allow for the continuance of the day-to-day operations of the laboratory.

4.1.2.1 Laboratory Director

The ultimate responsibility for the generation of reliable laboratory data rests with the Laboratory Director, who is accountable to his General Manager and oversees the daily operations of the laboratory. The Laboratory Director's responsibilities include allocation of personnel and resources, setting goals and objectives for both the business and employees, achieving the financial, business and quality objectives of STL. Furthermore, to see that all tasks performed in the laboratory are

conducted according to the requirements of this LQM, the Project Specifications and/or the appropriate QAPP; and to assure that the quality of service provided complies with the project's requirements.

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

4.1.2.2 Quality Assurance Manager

The Quality Assurance Manager (QAM) has the full-time responsibility to evaluate the adherence to policies and to assure that systems are in place to produce the level of quality defined in this LQM. The QAM is responsible for:

- Ensures that the laboratory's quality system and LQM meet the requirements of the Corporate QMP.
- Ensures IDL/MDL studies are completed and documented
- Ensures method validation studies are completed and documented
- Periodically performs data package inspections
- Performs data authenticity audits on 100% of analysts and instruments
- Assist in the preparation, compilation, and submittal of quality assurance project plans
- Reviews program plans for consistency with organizational and contractual requirements and advises appropriate personnel of deficiencies
- Maintains QA records
- Maintains certifications and accreditations
- Initiates and oversees both internal and external audits; documents root cause investigations for all noted deficiencies; and ensures timely audit closure
- Maintains a corrective action process for internally identified issues and ensures timely closure
- Manages the laboratory's PT Program and performs/documents root cause investigations for all failures
- Monitors to ensure the documentation of training and method demonstration is current
- Facilitates SOP development and document control
- Submits monthly QA reports to management

The QAM shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QAM is available to any employee at the facility to resolve data quality or ethical issues. The QAM shall be independent of laboratory operations and has an indirect reporting relationship to the STL Corporate QA Director.

4.1.2.3 Technical Director

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

In addition, the Technical Director solves day to day technical issues, provides technical training and guidance to staff, project managers and clients, investigates technical issues identified by operations personnel or QA, and directs evaluation of new methods.

4.1.2.4 Operations Manager

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

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

In addition, the Operations Manager works with the Technical Director in evaluating technical equipment and assessing capital budget needs.

4.1.2.5 Customer Service Manager/Project Managers

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

4.1.2.6 Laboratory Supervisors

The Laboratory Supervisors are as follows:

- Sample Management Supervisor
- Organic Preparation Supervisor
- GC Analysis Supervisor
- GCMS Volatiles Supervisor
- GCMS Semivolatiles Supervisor
- Metals Supervisor
- Wet Chemistry Supervisor

The Laboratory Supervisors serve as the technical experts on assigned projects, provide technical liaison, assist in resolving any technical issues within the area of their expertise; and implement established policies and procedures to assist the Operations Manager in achieving section goals. The Laboratory Supervisors are responsible for ensuring that their personnel are adequately trained to perform analyses; that equipment and instrumentation under their control is calibrated and functioning properly; that system and performance audits are performed on an as-needed basis; provide input and review in the development and implementation of project-specific QA/QC requirements; and for providing the critical review of proposal and project work for programs as

directed by the Operations Manager and Laboratory Director. The Laboratory Supervisors coordinate these activities with the project management and quality assurance sections.

4.1.2.7 Sample Management Coordination

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

4.1.2.8 Subcontract Sample Management Coordination

The Project Manager is designated as the Sample Management Coordinator for any work subcontracted under their management. The Project Manager verifies each subcontracting request to ensure that special client restrictions are not jeopardized (e.g., samples must be analyzed by the receiving affiliated or network laboratory and must maintain specific certification(s)). The Project Manager is also responsible for verifying the credentials; establishing the service agreement; ensuring data review; and invoicing of all laboratory subcontractors. The Project Manager discusses any deficiencies or anomalies with the subcontractor prior to reporting any data to the client.

4.1.2.9 Environmental Health and Safety Coordinator / Waste Management

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

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

4.1.2.10 Information Technology Manager

The overall role of the Information Technology (IT) Manager is to enhance laboratory productivity through improved information access, flow, and security. For information to be of greatest value, it must be readily accessible and reliable. It is the responsibility of the IT Manager to provide software tools that allow quick and user friendly access to that information, while at the same time controlling access to that information to those that have the need and proper authority.

Information flow can be enhanced through automation. Automation is the minimization of human intervention in a process. Reduction in human intervention can result in significant error reductions and time savings. The IT Manager assists the laboratory in automation by providing hardware and software solutions to help minimize human intervention in data collection, processing, and storage.

The IT Manager is responsible for providing data security by controlling access, as mentioned above, and for providing for disaster recovery. Data stored on the central Laboratory Information Management System (LIMS) is the direct responsibility of the IT Manager. No fewer than two copies of all data should exist at any time so that lost or destroyed data can always be retrieved from an alternate source. These copies may consist of data within the system and on electronic storage media. Data stored electronically in other departments is the direct responsibility of those departments. However, the IT Manager is responsible for providing procedures and training to all laboratory operations, as appropriate, to assist in making backup copies of local data.

STL has established procedures for IT management:

- *Computer Systems Account and Naming Policy* – P-I-003
- *Computer Systems Password Policy* – P-I-004
- *Software Licensing Policy* – P-I-005
- *Virus Protection Policy* – P-I-006

4.1.2.11 Chemists / Technicians

Any effective laboratory quality assurance/quality control program depends on the entire organization, including management and every individual on the laboratory staff. Analysts and technicians must read and be familiar with the LQM, method SOPs and other essential standard operating procedures. They must know where SOPs are located and agree to adhere to them explicitly unless an error in the SOP is evident and they brought this to the attention of their supervisor or QA manager. They must receive ethics and data integrity training and sign an ethics agreement annually.

Analysts and technicians must ensure that their training records are up to date prior to performing a method without direct supervision. This includes maintaining their training file, filing demonstration of capability evidence and receiving supervisor approval.

The initial review for acceptability of analytical results rests with the analysts conducting the various tests. Observations made during the performance of an analytical method may indicate that the analytical system is not in control. Analysts must use quality control indicators to assure that the method is within acceptance limits, corrective action is taken or a non-conformance (job exception) report is documented/approved before reporting results.

4.1.2.12 Data Packaging Specialist

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

4.2 Quality System

The quality system and quality objectives are driven by this LQM, SOPs and Work Instructions. Within these documents, the Laboratory Director and Quality Manager ensure that the quality policy is understood, implemented, and maintained at all levels of the organization. The development and implementation of appropriate accountabilities, duties, and authority by organizational positions are clearly delineated. Line organizations achieve and verify that specifications are achieved; the QAM provides oversight and verification of processes through planning, reviews, audits, and surveillances.

The Laboratory Director's leadership, support and direction ensure that the policies and procedures are implemented throughout the organization.

4.2.1 Objectives of the Quality System

The goal of the quality system is to ensure that business operations are conducted with the highest standards of professionalism and data integrity in the industry.

To achieve this goal, it is necessary to provide our clients with scientifically sound, well documented, regulatory compliant data, and to ensure that we provide the highest quality service available in the industry with uncompromising data integrity. A well-structured, organized and communicated quality system is essential in meeting this goal. The laboratory's quality system is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement.

This LQM, Work Instructions and the SOPs are the basis and outline for our quality and data integrity system and contain requirements and general guidelines under which the laboratory conducts operations. In addition, other documents may be used by the laboratory to clarify compliance with quality system or other client requirements. Within the LQM, SOP or Work Instruction numbers are noted in parenthetic text. These numbers refer to the laboratory procedure(s) associated with the subject item. A table listing these quality system policies and procedures is appended to this document.

The QA Manager is responsible for implementing and monitoring the Quality System. The QA Manager reports to the Laboratory Director on the performance of the quality system for review and continuous improvement. The QA Manager has sufficient authority, access to work areas, and organizational freedom (including sufficient independence from cost and schedule considerations) to:

- Initiate action to prevent the occurrence of any nonconformities related to product, process and quality system,
- Identify and record any problems affecting the product, process and quality system,
- Initiate, recommend, or provide solutions to problems through designated channels,
- Verify implementation of solutions, and
- Assure that further work is stopped or controlled until proper resolution of a non-conformance, deficiency, or unsatisfactory condition has occurred and the deficiency or unsatisfactory condition has been corrected.

The QA Manager identifies opportunities for continual improvement. When a situation arises where acceptable resolution of identified issues cannot be agreed upon at the laboratory, direct access to STL's Corporate QA Director is available. This provides laboratory QA personnel independence, where needed, to ensure that QA policies and procedures are enforced.

The QA Manager conducts annual training for all laboratory and administrative personnel to ensure their familiarity with the quality documentation and the implementation of the policies and procedures in their work.

4.3 Document Control

The laboratory maintains procedures to control documents and analytical data. Since an extensive quantity of data is generated and this is our primary product, document control is inherently segregated from data control, as described further in Sections 4.3.1 and 4.3.2.

4.3.1 Document Control Procedure

Organization, security and control of documents are necessary to ensure that confidential information is not distributed and that all current copies of a given document are from the latest applicable revision. Unambiguous identification of a controlled document is maintained by information in the document header: Document Number, Revision Number, Effective Date, and Number of Pages. Document control may be achieved by either electronic or hardcopy distribution. Documents may be controlled for a specific time period after issuance. In this case the document will be marked with the date issue and expiration date.

Controlled documents are authorized and records of their distribution and archiving are maintained by the QA Department. Controlled status is defined as the continuous distribution of document updates where document marked as either "Controlled" or "Uncontrolled". Uncontrolled status is defined as the single distribution of the current SOP. Document updates are not distributed to people holding documents marked "uncontrolled". For tracking purposes, a control copy number is assigned to documents distributed with a controlled status. All copy numbers are written or typed in red to easily identify the SOP as a controlled copy.

4.3.1.1 Document Revision

Changes to documents occur when a procedural change warrants a revision of the document. After document revisions are authorized, all outdated versions are removed from use and disposed or segregated from the active/current document versions. A single copy of the archived document is retained for historical purposes. This archived version is clearly identified as an "Archived Copy".

SOPs are reviewed and/or updated on a 12 month basis, which is tracked by an established review schedule *(SOP Master Index)*. These reviews are conducted by the analyst, QA Manager, Department Supervisor, Laboratory Director or the Health and Safety Coordinator, all of whom may provide the approval signature for each SOP.

4.3.2 Data Control

All raw data, such as bound logbooks, instrument printouts, magnetic tapes, electronic data, as well as final reports, are retained for a minimum period of 5 years, unless otherwise specified by client or regulatory requirements. Such data may be maintained longer, as defined by client and project requirements. Specifics on the procedure of archiving records and client or project specific requirements are contained in the SOP, Record Storage and Retention,*(AGP-RecordStorage-56).*

Raw data and reports are documented and stored in a manner which are easily retrievable. The procedure for maintaining raw data records is briefly described below:

- Instrument print-outs for conventional inorganic parameters are filed by parameter and month. Inorganic Metals are filed by Instrument and Filename. Generally, current year documents are kept on file in the laboratory sections.
- All raw data, for example, instrument print-outs and logbooks, are maintained in a secured storage area or records are scanned and retained on electronic media.

- The computer information is backed up on tape daily, and stored in a secured and temperature/humidity controlled environment to maintain the integrity of the electronic information in the event of system failure. Copies of all back-up tapes are maintained in secured off-site locations.
- All copies of client final reports are maintained in hard copy format or electronically (e.g., Adobe Acrobat).

4.4 Request, Tender, and Contract Review

4.4.1 Contract Review

For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is STL's intent to provide both standard and customized environmental laboratory services to our clients. To ensure project success, technical staff members perform a thorough review of technical and QC requirements contained in contracts. Contracts are reviewed for adequately defined requirements and STL's capability to meet those requirements.

All contracts entered into by the laboratory are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity, accuracy, and precision requirements. The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The review also includes the laboratory's capabilities in terms of turnaround time, capacity, and resources to provide the services requested, as well as the ability to provide the documentation, whether hardcopy or electronic. If the laboratory cannot provide all services but intends to subcontract such services, whether to another STL facility or to an outside firm, this will be documented and discussed with the client prior to contract approval.

Any contract requirement or amendment to a contract communicated to STL verbally is documented and confirmed with the client in writing (e.g., letter, contract, e-mail, etc.). Any discrepancy between the client's requirements and STL's capability to meet those requirements is resolved in writing before acceptance of the contract. Contract amendments, initiated by the client and/or STL, are documented in writing for the benefit of both the client and STL. All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the permanent project record.

4.4.2 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, STL assigns a Project Manager (PM) to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project (Project Information Requirements, APM-ProjInfo-20). QA department involvement may be needed to assist in the evaluation of custom QC requirements.

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

resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings *(APM-ProjInfo-20)* or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch (e.g., Job) as a reminder upon sample receipt and analytical processing.

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

Such changes are also communicated to the laboratory through the management Production Meetings which are conducted two times per week. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the Project Manager or the individual laboratory section manager.

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

4.4.3 Data Quality Objectives

Data quality objectives (DQO) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application. Typically, DQOs are identified before project initiation and during the development of QAPPs and SAPs. The analytical DQOs addressed in this section are precision, accuracy, representativeness, completeness, and comparability.

The components of analytical variability (uncertainty) can be estimated when QC samples of the right types and at the appropriate frequency are incorporated into the measurement process of the laboratory. STL incorporates numerous QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The control samples and their applications, described in Section 5.8.2, are selected based on analytical method or client-specific requirements. Analytical QC samples for inorganic and organic analyses may include calibration blanks, instrument blanks, method blanks, laboratory control standards, calibration standards, matrix spikes, matrix duplicates and surrogate spikes.

The DQOs discussed below ensure that data are gathered and presented in accordance with procedures appropriate for its intended use, that the data is of known and documented quality, and are able to withstand scientific and legal scrutiny.

4.4.3.1 Precision

Precision is an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. Precision is expressed either as Relative Standard Deviation (RSD) for greater than two measurements or as Relative Percent Difference (RPD) for two measurements. Precision is determined, in part, by analyzing data from LCSs, MS, MSD, and MD.

Precision also refers to the measurement of the variability associated with the entire process, from sampling to analysis. Total precision of the process can be determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations.

4.4.3.2 Accuracy

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. It reflects the total error associated with a measurement.

Both random and systematic errors can affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100). Accuracy is determined, in part, by analyzing data from LCSs, MS and MSD.

Accuracy and Precision objectives employed by the laboratory are as defined in the CERCLA's Inorganic and Organic Statements of Work (SOW); statistically-derived control limits; or default limits as listed in each respective method SOP.

4.4.3.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result is representative of the constituent concentration in the sample matrix. STL makes every effort to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before sub-sampling.

4.4.3.4 Completeness

Completeness is defined as the percentage of measurements that are judged valid or useable. Factors negatively affecting completeness include the following: sample leakage or breakage in transit or during handling, loss of sample during laboratory analysis through accident or improper handling, improper documentation such that traceability is compromised, or sample result is rejected due to failure to conform to QC specifications. A completeness objective of greater than 90% of the data specified by the statement of work is the goal established for most projects.

4.4.3.5 Comparability

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (e.g., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

A measure of inter-laboratory comparability is obtained through the laboratory's participation in proficiency testing (PT) programs established with Water Supply (WS), Water Pollution (WP), Solid Waste (SW), and Underground Storage Tank (UST) programs. In addition, the laboratory employs the use of NIST or EPA traceable standards, when available, to provide an additional measure of assurance of the comparability of data.

Project representativeness and comparability are dependent upon the sampling plan on a project specific basis, and are therefore not covered in this LQM. Assessment of site and collection representativeness and comparability is performed by client or field engineer.

4.4.3.6 Additional DQOs

Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually.

For the performance of non-routine methods, e.g., client/contract requirement, MDLs or Method Validation Studies will be completed on an as needed basis. The turnaround time for such studies will be as determined by the client and Project Manager. Such studies will be reviewed and approved by the client and/or regulatory agency prior to project implementation.

Instrument Detection Limits

There are a number of ways to determine Instrument Detection Limit (IDL) sensitivity (e.g., signal-to-noise ratio, precision of the low-level standard, lowest calibration curve point or the IDL study defined within CLP). The method and means in which IDLs are determined are documented and maintained in the QA department for each individual instrument.

IDLs are periodically generated for each element by the metals laboratory based on project or program requirements (i.e., CLP is quarterly for each instrument). These limits are used to gauge instrument sensitivity without the introduction of preparation method variance.

Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory can report with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory reporting limits are further related and verified by the lowest point on a calibration curve. Because of the high level of quantitative error associated with determinations at the level of the MDL, the laboratory endeavors to keep reporting limits higher than the MDL. Wherever possible, reporting is limited to values approximately 2-5x the respective MDL to ensure confidence in the value reported.

Client specific requests for reporting below the routine laboratory reporting limit or approaching the IDL or MDL are special circumstances not to be confused with the previous statement. Data evaluated down to the MDL/IDL is qualified as estimated with a 'J' for organic analyses, a 'B' for inorganic analyses or with a comment in the report case narrative.

MDL studies are performed annually, and reporting limits are assessed. If the MDL does not meet the routine laboratory reporting limit or the method specified limit, it is repeated or the laboratory reporting

limit is reassessed. If the laboratory continually demonstrates that the method reporting limits are not achieved, equipment, technique, and the method are reviewed to assure optimal performance or appropriate action is taken.

4.5 Subcontracting

Subcontracting is arranged with the consent of the client. Consent shall be requested in a timely manner and the client response shall be suitably prompt to ensure that it shall not be unreasonably refused. All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Proof of required certifications from the subcontract facility is maintained in the project records. Where applicable, specific QC guidelines, QAPPs, and/or SAPs are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC).

Subcontract laboratories may receive an on-site audit by a representative of STL's QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements (e.g., Technical Profile and LIMS Project Notes). STL may also perform a paper audit of the subcontractor, which would entail reviewing the LQM, the last two PT studies, and a copy of any recent regulatory audits with the laboratory's responses.

Intra-company subcontracting may also occur between STL facilities. Intra-company subcontracting within STL is arranged with the documented consent of the client or a QAPP specification. The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

Project reports from both STL and external subcontractors are not altered and are included in their original form in the final project report provided by STL. This clearly identifies the data as being produced by a subcontractor facility. If subcontract data are incorporated into the laboratory's report (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples , as required in Section 5.9.4.

4.6 Purchasing Services and Supplies

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

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents meet with the requirements of the specific method and testing procedures for which they are being purchased. The measurements for evaluation and selection of suppliers; the acceptance of supplies and services; and certificates of conformance are described in the procurement SOP *(Procurement of Laboratory Supplies and Services, APH-Supply-08).*

4.6.1 Solvent and Acid Lot Verification

Pre-purchase approval is performed for solvents and acids purchased in large quantities unless a certificate of conformance has been furnished. These may include acetone, ethyl ether, hexane, methylene chloride, nitric acid, hydrochloric acid, sulfuric acid, and hydrogen peroxide. Each lot of incoming supplies requiring pre-approval is checked against the previously approved lot number. If the lot number is not approved, the lot is refused. If the lot number is an approved lot number, it is accepted and documented. Solvents and acids are pre-tested in accordance with STLs Corporate *Testing Solvents and Acids* procedure (S-T-001) for all of the STL laboratories.

4.7 Service to the Client

4.7.1 Sample Acceptance Policy

Samples are considered "compromised" if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservation.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples >6mm.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it is documented on the hardcopy COC, the LIMS Sample Receipt Checklist or on an Analytical Receipt Resolution Form (ARRF); and the client is contacted for instructions. If the client decides to proceed with the analysis, the project report will clearly indicate any of the above conditions and the resolution.

4.7.2 Client Confidentiality and Proprietary Rights

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL and client contract) subject to any disclosure required by law or legal process. Technical, business and proprietary information provided by a client and data/information generated by the laboratory are restricted for the use within the laboratory for purposes of accomplishing the project. Client information is not to be used on other projects or revealed except in conjunction with project work to anyone outside the laboratory without permission of the client.

STL's reports, and the data and information provided therein, are for the exclusive use and benefit of client, and are not released to a third party without written consent from the client (*Client Confidentiality Section 6.9; APM-ProjInfo-20).*

4.8 Complaints

STL believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client's concerns captures "client knowledge" that helps to continually improve processes and outpace the competition. Implementing a client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly. The investigation of the cause, resolution and authorization of corrective action is documented *[Data Quality Request (DQR); SOP AQA-DQR-65 or Corrective Action Notice (CAN); SOP AQA-CA-65)].*

Client complaints are documented by the employee receiving the complaint. The documentation can take the form of a Data Quality Review request (DQR) or in a format specifically designed for that purpose (e.g., phone conversation record or e-mail). The Laboratory Director, CSM, Technical Director and/or QA Manager are informed of client complaints and assist in resolving the complaint.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. STL Buffalo uses an automated documentation and tracking mechanism for the DQR process which provides a system for trend analysis of repeat complaints. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA department is required to conduct a special audit to assist in resolving the issue. A written confirmation, or letter to the client, outlining the issue and response taken is strongly recommended as part of the overall action taken.

The number and nature of client complaints is reported by the QA Manager to the Corporate QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Quality Systems Management Review *(QMP, Section 4.15.2 and SOP AQA-Management Review-45).*

4.9 Control of Non-conformances

Non-conformances include any out of control occurrence. Non-conformances may relate to client specific requirements, procedural requirements, or equipment issues. All non-conformances in the laboratory are documented at the time of their occurrence on a Job Exception Report, also known as a non-conformance report *(AQA-CA-65)*

All non-conformances that affect a sample and/or sample data become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Section Manager, Project Manager or QA Manager for direction may be required. All records of reanalysis are kept with the project files.

Where non-conformances specifically affect a client's sample and/or data, the client is informed and action must be taken. Action can take the form of reporting and flagging the data, and including a description of the non-conformance in the project narrative.

4.10 Corrective Action

To consistently achieve technical and regulatory requirements, the laboratory data must be supported by an effective corrective action system. The system must be capable of isolating and rectifying both random and systematic errors. Identification of systematic errors, or errors that are likely to occur repetitively due to a defect or weakness in a system, is particularly valuable in maintaining an environment of continuous improvement in laboratory operations.

Mechanisms used to ensure problem definition include SOPs; internal and external audits and surveillances; and regular laboratory management meetings. When evaluation of performance against established criteria for good laboratory practices shows a condition that could adversely affect the quality of services provided, corrective action is initiated.

Any employee in STL can initiate a corrective action. The initial source of corrective action can also be external to STL (i.e., corrective action due to client complaint, regulatory audit, or PT(s)). When a problem that requires corrective action is identified, the following items are identified by the initiator on the corrective action report: the nature of the problem, the name of the initiator, and the date. If the problem affects a specific client project, the PM is informed immediately.

All corrective actions, whether immediate or long-term, will comprise the following steps to ensure a closed-loop corrective action process:

- Define the problem.
- Assign responsibility for investigating the problem.
- Determine a corrective action to eliminate the problem.
- Assign, and obtain commitment to, responsibility for implementing the corrective action.
- Implement the correction.
- Assess the effectiveness of the corrective action and verify that the corrective action has eliminated the problem.

4.10.1 Immediate Corrective Action

Immediate corrective actions to correct or repair non-conforming equipment and systems are generally initiated in response to adverse conditions identified through QC procedures. The analyst has relatively quick feedback that a problem exists, e.g., calibration does not meet or QC check samples exceed allowable criteria, and can take immediate action to repair the system.

The initial responsibility to monitor the quality of a function or analytical system lies with the individual performing the task or procedure. DQOs are evaluated against laboratory-established or against method or client specified QA/QC requirements. If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. When the appropriate corrective action measures have been defined and the analytical system is determined to be "in-control" or the measures required to put the system "in-control" have been identified and scheduled, the problem and resolution or planned action is documented in the

appropriate logbook or Job Exception Report. Data generated by an analytical system that is determined to be out-of-control must never be released without approval of the Section Manager, QA Manager, Laboratory Director and client notification.

When an acceptable resolution cannot be met or data quality is negatively affected, the analyst will notify their Section Manager and initiate a Job Exception. If a Job Exception is required, it is routed for proper authorizations and direction. Proper authorization and direction is given by the Project Manager and/or QA Manager. Based upon the circumstances and judgment of the Project Manager, in conjunction with the QA Manager, the client will be notified of the situation.

Data generated concurrently with an out-of-control system will be evaluated for usability in light of the nature of the deficiency. If the deficiency does not impair the usability of the results, data will be reported and the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written Job Exception Report and appropriate corrective action (e.g., reanalysis) is taken and documented.

A Job Exception documents analytical problems at the bench level. This form allows for the documentation of the out-of-control situation, actions undertaken to correct the problem and a return-to-control status. All Job Exceptions are signed/dated by the respective laboratory section manager.

The QA Manager has the authority to stop the analysis, e.g., failure to meet method or project requirements, and to hold all analyses of samples affected by an out-of-control situation. The method cannot be restarted without appropriate documentation leading to the QA Manager's approval and sign-off.

4.10.2 Long-term Corrective Action

Long-term corrective action is generally initiated due to QA issues, which are most often identified during internal and external audits (Sections 4.13 & 4.14). Typically, a deeper investigation into the root cause of the nonconformance is warranted, and the problem may take much longer to identify and resolve. Staff training, method revision, replacement of equipment, and LIMS reprogramming are examples of long-term corrective action.

4.10.3 Responsibility and Closure

The Section Manager is responsible for correcting out-of-control situations, placing highest priority on this endeavor. Associated corrective actions, once verified for effectiveness, are incorporated into standard practices. Ineffective actions will be documented and re-evaluated until acceptable resolution is achieved. Section Managers are accountable to the Operations Manager to ensure final acceptable resolution is achieved and documented appropriately.

The QA Manager also may implement a special audit (Section 4.13). The purpose of inclusion of the corrective action process in both routine and special audits is to monitor the implementation of the corrective action and to determine whether the action taken has been effective in overcoming the issue identified.

Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation. This provides laboratory QA personnel non-laboratory management support, if needed, to ensure QA policies and procedures are enforced.

4.11 Preventative Action

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

Preventive action opportunities may be identified from information obtained through activities related to but not limited to the corrective action process, performance evaluation program, internal audits, management review, and/or market trends, industry trends and competitive comparisons.

Established standard practices for preventive action are included in the Corrective and Preventive Action SOP *(AQA-CA-35)*; the Data Quality Request SOP *(AQA-DQR-65)* and the Quality System Management Review SOP *(AQA-Management Review-45)*. These procedures describe the information sources used to detect, analyze, and eliminate potential causes of nonconformities and to ensure effective implementation of solutions.

4.12 Records

4.12.1 Record Types

Record types are described in Table 4.

4.12.2 Record Retention

Data reports are filed electronically as .pdf files by job number. Hardcopy COC files are maintained and are filed with the original Job File in job number order.

Laboratory data, project management files, QA records (e.g., PT scores/corrective actions; MDLs/IDLs, statistical analysis, QAPPs, etc..), Human Resources information, etc.., are compiled by date order. The same procedure is followed both in current and archived hardcopy storage.

Upon archiving, a record is made in the Archive Logbook and a number is assigned for each storage box of records. This logbook documents the contents (description and dates) of each storage box. Records are maintained for the periods defined in Tables 5 and 6. On an annual basis, the storage boxes are reviewed and those records subject to disposal are purged.

Table 5 outlines the laboratory's standard record retention time. For raw data and project records, record retention is calculated from the date the project report is issued. For other records, such as Controlled Documents, QC, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 6 have lengthier retention requirements and are subject to the requirements in Section 4.12.3.

Table 4. STL Record Types

Raw Data	Controlled Documents	QC Records	Project Records	Administrative Records

Raw Data	Controlled Documents	QC Records	Project Records	Administrative Records
See Section 3. Terms and Definitions	LQMs/ QAPPs	Audits/ Responses	COC Documentation	Accounting
	QMP (Corporate)	Certifications	Contracts and Amendments	Corporate Safety Manual, Permits, Disposal Records
	SOPs	Job Exceptions / DQRs	Correspondence	Employee Handbook
		Logbooks*	QAPP	Personnel files, Employee Signature & Initials, Training Records
		Method & Software Validation, Verification	SAP	
		Standards Certificates	Telephone Logbooks	Technical and Administrative Policies
	Work Instructions	MDL/IDL/IDC Studies	E-mails	
		PTs	Electronic Data Report	
		Statistical Evaluations		

*Examples of Logbook types: Maintenance, Instrument, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, and Balance Calibration.

Table 5. STL Record Retention

Record Type		Archival Requirement *
Raw Data	All* (Electronic Data Reports (.pdf & EDD)	5 Years from completion
Controlled Documents	All*	5 Years from document retirement date
QC	All*	5 Years from archival
Project	All*	5 Years from project completion
Administrative	Personnel/Training	Indefinitely
	Accounting	10 years

* Exceptions listed in Table 6.

4.12.3 Programs with Longer Retention Requirements

Some regulatory programs and clients have longer record retention requirements than the laboratory's standard record retention time. These are detailed in Table 6 with their retention requirements and client-specific requirements are listed in the Record Retention and Storage SOP (*AQA-RecordStorage-56*). In these cases, the longer retention requirement is implemented and

noted in the archive. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 6. Special Record Retention Requirements

Program	Retention Requirement
NY Potable Water NYCRR Part 55-2	10 years
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Michigan Department of Environmental Quality – all environmental data	10 years
Minnesota – Drinking Water	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
OSHA - 40 CFR Part 1910	30 years
Pennsylvania – Drinking Water	10 years
TSCA – 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement
Louisiana – All environmental data	10 years

4.12.4 Archives and Record Transfer

Archives are indexed such that records are accessible on either a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented.

STL ensures that all records are maintained as required by the regulatory guidelines and per this LQM upon facility location change or ownership transfer. Upon facility location change, all archives are retained by STL in accordance with this LQM. Upon ownership transfer, all final test reports generated by the laboratory will be submitted to the clients if not previously provided. Any further record retention requirements will be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established.

In the event that the laboratory is closed, all final test reports generated by the laboratory will be submitted to the clients if not previously provided. All records will then be transferred to STL's corporate record storage location. All boxes and contents will be appropriately labeled with the dates of destruction (Refer to Tables 5 and 6) and managed in accordance their policies.

4.13 Internal Audits

Quality assurance audits and surveillances are conducted to assess the performance of laboratory systems in meeting technical, regulatory and client requirements; and to evaluate the operational details of the QA program *(Systems Audits; S-Q-002)*. They provide a means for management to be apprised of, and to respond to, a potential problem before it actually impacts the laboratory operations. They also are a mechanism for ensuring closure of corrective actions resulting from external audits.

4.13.1 Audit Types and Frequency

A number of types of audits are performed at STL. These audit types and frequency are categorized in Table 7.

Table 7. Audit Types and Frequency

Audit Type	Performed by	Frequency
Systems	QA Department or Designee	Annual
Data	QA Department or Designee	**Data Report Review:** As necessary to ensure an effective secondary review process
		Analyst Data Audits: 100% of all analysts annually
		Electronic Data Audits: 100% of all organic instruments
Special	QA Department or Designee	As Needed

4.13.2 Systems Audits

Systems audits are technical in nature and are conducted on an ongoing basis by the QA Manager. Systems audits cover all departments of the facility, both operational and support. The review consists of laboratory systems, procedures, documentation and issues noted in external audits.

The audit report is issued by the QA Manager within 21 calendar days of the audit. The audit report is addressed to the Operations Manager and Department Supervisors and copied to the Corporate Quality Director and the Laboratory Director.

Written audit responses are required within 30 calendar days of the audit report issue. A maximum of one calendar month is given to address any recommended corrective actions. The audit response is directed to all individuals copied on the audit report. Where a corrective action may require longer than a calendar month to complete, the target date for the corrective action implementation is stated and evidence of the corrective action is submitted to the QA Department in the agreed upon time frame.

4.13.3 Data Audits

Data audits are focused to assess the level of customer service, SOP compliance, regulatory compliance, accuracy and completeness of test results and reports, documentation, and adherence to established QC criteria, laboratory SOPs, technical policy, and project specific QC criteria.

The QA Department provides feedback and/or corrections and revisions to project reports where necessary. Records of the data audits are kept, and the frequency of data audits is included in the monthly QA report. In performing data audits, it is essential that data be assessed in terms of

differentiating between systematic and isolated errors. Upon noting anomalous data or occurrences in the data audits, the QA Department is responsible for seeking clarification from the appropriate personnel, ascertaining whether the error is systematic or an isolated error, and overseeing correction and/or revision of the project report if necessary. Errors found in client project reports are revised and the revision sent to the client (Section 4.8). The QA Department is also responsible for assisting in the corrective action process where a data audit leads to identification of the need for permanent corrective action.

The frequency of data auditing may also be dependent upon specific clients and regulatory programs. All active laboratory logbooks and QC files are subject to periodic audits/ surveillances by the QA personnel.

4.13.3.1 Data Authenticity Audits

Data authenticity audits shall be performed on 100% of all analysts by the QA department or a designee independent from the operations. Performing data authenticity checks will typically include verifying raw data, evaluating calculation tools and independently reproducing the final results and comparing it to the hardcopy on randomly selected batches of data. The QA Manager will report the percentage of analysts reviewed (for the year) in the monthly QA report and should average about 8% per month.

4.13.3.2 Electronic Data Audits

Electronic data audits are performed on 100% of all organic instruments by the QA department or a designee independent from the operations. This may include Mint Miner® scanning of randomly selected batches of electronic data followed by a chromatography system review. The QA manager will report the percentage of instruments reviewed (for the year) in the monthly QA report and should average about 8% of instruments per month. Electronic data audits include spot-checking of manual integrations by QA personnel in order to determine that the manual integration is appropriate and documented according to Section 5.3.6.1.

4.13.4 Special Audits

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

4.14 External Audits

STL is routinely audited by clients and external regulatory authorities – both government and non-government. Whether the audit is scheduled or unannounced, full cooperation with the audit team is provided by the laboratory and administrative staff. STL recommends that the audits be scheduled with the QA Department so that all necessary personnel are available on the day of the audit.

4.15 Management Reviews

4.15.1 QA Reports to Management

A monthly QA report is prepared by the QA Manager and forwarded to the Laboratory Director and Corporate Quality Director. The reports include statistical results that are used to assess the effectiveness of the quality system. The required information for the monthly report is shown in Figure 3.

4.15.2 Quality Systems Management Review

A quality systems management review is performed at least annually by the Laboratory Director and QA Manager *(SOP AQA-Management Review-45)*. This review ensures that the laboratory's quality system is adequate to satisfy the laboratory's policies and practices, government requirements, certification, accreditation, approval requirements, and client expectations. Quality systems management reviews are accomplished through the evaluation and revision of this LQM, monthly quality assurance reporting and goal setting.

Management reviews of specific quality system elements may be performed through continuous improvement activities, monthly QA reports, process changes, SOP revisions, and/or audit reports/responses. Documentation of these reviews are not required unless it is inherent in the review mechanism (e.g., approval signatures on SOP revisions).

4.15.3 Monthly QA Report and Metrics

By the 3rd day of the month, the QA manager prepares a monthly QA report. The report is sent to the Laboratory Director, General Manager and Corporate Quality Director. The report contains a narrative summary and metrics spreadsheet. At a minimum, the report content contains the items listed below (Figure 3). During the course of the year, the Laboratory Director, General Manager or Corporate Quality Director may request that additional information be added to the report.

Figure 3. Monthly QA Report Format

1	Audits
	Internal System Audits External System Audits
2	Revised Reports / Client Feedback
	Revised Reports Client Complaints Client Compliments
3	Certification Changes
	Changes Losses / Revocations
4	Proficiency Testing
	Study participation and scores Combined PT scores Repeat failures
5	SOP Status
	Report the percentage of SOPs that have been revised or reviewed within the last 24 months.
6	Miscellaneous QA and Operational Issues
	Narrative outlining improvements, regulatory

	compliance issues and general concerns.
Appended	Metrics Spreadsheet
	Summarize metrics in template provided by the Corporate Quality Director

5.0 Technical Requirements

5.1 Personnel

5.1.1 General

STL management believes that its highly qualified and professional staff is the single most important aspect in assuring the highest level of data quality and service in the industry. The staff consists of professionals and support personnel that include the following positions:

- General Manager
- Laboratory Director
- Technical Director
- QA Manager
- Human Resource Manager
- Customer Service Manager
- Operations Manager
- QA Specialist
- Health & Safety Coordinator / Waste Management
- Project Manager
- Information Technology Manager
- Network Administrator
- Department Supervisor
- Analyst
- Sample Custodian
- Technician
- Data Reporting Specialist

In order to ensure that employees have sufficient education and experience to perform a particular task, job descriptions are developed for all personnel. Job Descriptions are located on the STL Intranet Site's Human Resources web-page:

http://stlnet.stl-inc.com/Corporate/HR/JobDescriptions/JobDescrip_index.htm.

5.1.2 Training

STL is committed to furthering the professional and technical development of employees at all levels. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for STL employees are outlined in Job Descriptions.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency.

The QA section in conjunction with the Human Resources section are responsible for maintaining documentation of these activities.

Each laboratory section is required to maintain documentation associated with analytical training (e.g., training records, IDOCs, CDOCs, and controlled documents). The QA department maintains documentation of method proficiency (e.g., IDMPs, MDLs, MDLVs, PT Sample Tracking, QC Control Limits/Data). This information is available to managers and staff for planning and evaluation.

The following evidence items are maintained in the employees technical training file for each technical employee:

- An Ethics Agreement signed by each staff member (renewed each year).
- A Confidentiality Agreement signed by each staff member (renewed each year).
- Initial Demonstration of Capability (IDOC)
- The employee has read and understood the latest version of the laboratory's quality documentation.
- The employee has read and understood the latest, approved version of all test methods and/or SOPs for which the employee is responsible.
- Annual evidence of continued DOC that may include successful analysis of a blind sample on the specific test method; a similar test method; an annual DOC; or four successive and acceptable LCSs.
- Documentation of external training courses attended
- All training regarding QA policies and procedures

The Human Resource department maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Table 8. STL Employee Minimum Training Requirements

Specialty	Experience
General Chemistry and Instrumentation	Six months
Gas Chromatography	One year
Atomic Absorption	One year
Mass Spectrometry	One year
Spectra Interpretation	Two years

Required Training	Time Frame [1]	Employee Type
Environmental Health & Safety	Month 1	All
Ethics	Month 1	All
Data Integrity	Month 1	Technical and PMs
Ethics Refresher	Annually	All
Quality Assurance	Quarter 1	All
Initial Demonstration of Capability	Prior to unsupervised method	Technical

(IDOC)	performance	

[1] From the date of initial employment unless otherwise indicated.

The quality assurance training includes an overview of regulatory programs and program goals, discussions about data integrity and data misrepresentation and an overview of laboratory quality control procedures and purposes.

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

Technical training is accomplished by the Operations Manager, Department Supervisor or a senior analyst to ensure method comprehension. All new personnel are required to demonstrate competency in performing a particular method by successfully completing an Initial Demonstration of Capability. IDOCs are performed by the analysis of four replicate QC samples. Results of successive LCS analyses can be used to fulfill the IDOC requirement. The accuracy and precision, measured as average recovery and standard deviation (using n-1 as the population), of the 4 replicate results are calculated and compared to those in the test method (where available). If the test method does not include accuracy and precision requirements, the results are compared to target criteria set by the laboratory. The laboratory sets the target criteria such that they reflect the DQOs of the specific test method or project. A IDOC Certification Statement is recorded and maintained in the employee's training file. Tabulated results summary and raw data are completed and signed by the analyst and section manager with the proper entries made onto the analysts training record. The data are submitted to the QA department for approval and entry into the master IDOC spreadsheet and filing. Figure 4 shows an example of a IDOC Certification Statement.

On an annual basis, each analyst's method capabilities must be evaluated. The requirement that a CDOC (Continued Demonstration of Capability) be completed for each method currently being analyzed must be presented for approval to QA in the same format as the IDOC discussed above.

Further details of the laboratory's training program are described in the SOP related to Laboratory Personnel Training *(AQA-TRAIN-10)*.

5.1.3 Ethics Policy

Establishing and maintaining a high ethical standard is an important element of a Quality System. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times; STL has established an Ethics Policy (P-L-006) and an Ethics Agreement (Figure 5). Each employee signs the Ethics Agreement, signifying agreed compliance with its stated purpose. The ethics agreement is required to be re-signed on an annual basis.

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

Ethics is also a major component of STL's quality and data integrity systems. Each employee is trained in ethics within two weeks of hire and quality training within three months of hire. Annually, ethics refresher training will be provided. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. A data integrity hotline is maintained by STL and administered by the Corporate Quality Director.

Figure 4. Demonstration of Capability Certification Statement

SEVERN TRENT STL

DOC Cert. Statement
Revision 6
October 12, 2005

SEVERN TRENT LABORATORIES - BUFFALO

TRAINING & DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

Employee: _____ Page _____ of _____

Method Number: _____ Date: _____

Parameters or Analytes: _____

Initial Demonstration of Capability: ☐

SOP Number: _____ Revision # _____ Date Read _____

Trained By: _____

Date training began: _____ Date training completed: _____

Continued Demonstration of Capability: ☐

SOP Number: _____ Revision # _____ Date Read _____

I CERTIFY that I have read and understand the SOP identified above. I have also submitted data associated with the demonstration of capability.

_____ _____
Employee Signature Date

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific Sops are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at this facility, and that the associated information is well organized and available for review by authorized assessors.

John Schove _____ _____
Operations Manager Signature Date

Verl Preston _____ _____
Quality Assurance Manager Signature Date

Figure 5. STL Ethics Agreement

I understand that STL is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:

- I will not intentionally report data values that are not the actual values obtained;
- I will not intentionally report the dates, times, sample or QC identification, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;
- I will not intentionally misrepresent another individual's work;
- I will not intentionally report data values that do not meet established quality control criteria as set forth in the Method and/or Standard Operating Procedures, or as defined by Company Policy;
- I agree to inform my Supervisor of any accidental reporting of non-authentic data by me in a timely manner; and I agree to inform my Supervisor of any accidental or intentional reporting of non-authentic data by other employees; and
- If a supervisor or a member of STL management requests me to engage in or perform an activity that I feel is compromising data validity or quality, I will not comply with the request and report this action immediately to a member of senior management, up to and including the President of STL.

As a STL employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination. In addition, I understand that any violation of this policy which relates to work under a government contact or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE:_____ Date: _____

Supervisor/Trainer:_____ Date:_____

5.2 Facilities

The laboratory is a secure facility with controlled and documented access. Access is controlled by keyless entry access cards, locked doors, and a staffed reception area. All visitors sign in and are escorted by STL personnel while at the facility. The laboratory is locked at all times.

The facility is designed for efficient, automated high-quality operations. The laboratory is equipped with Heating, Ventilation, and Air Conditioning (HVAC) systems appropriate to the needs of environmental testing laboratories. Environmental conditions in the facility, such as hood flow, are routinely monitored and documented.

The facility is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. STL also provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc..

5.3 Test Methods

Routine analytical services are performed using standard EPA-approved methodologies. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices.

5.3.1 Method Selection

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

Most of the test methods performed at STL originate from test methods published by a regulatory agency such as the US EPA and other state and federal regulatory agencies. These include, but are not limited to, the following published compendiums of test methods. A listing of methods in which the laboratory is capable of performing is listed in the laboratory's Master Methods Index and Preservation Table *(SOP ASR-Psrv-07).*

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water.

Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-003, February 1999.

Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.

Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.

Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.

NIOSH Manual of Analytical Methods, 4th ed., August 1994.

Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88-039, December 1988, Revised July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992.

Statement of Work for Inorganics Analysis, ILM04.2, ILM05.2 and ILM05.3 USEPA Contract Laboratory Program Multi-media, Multi-concentration.

Statement of Work for Organics Analysis, OLM04.2 (with OLM04.3 update) and OLC02.1, USEPA Contract Laboratory Program, Multi-media, Multi-concentration.

New York State Department of Environmental Conservation, Analytical Services Protocol, NYSDEC ASP.

Standard Methods for the Examination of Water and Wastewater, 18th/19th/20th edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and establishes an implementation schedule. As such, the laboratory strives to perform only the latest versions of each approved method.

5.3.2 SOPs

STL maintains a Master Index of SOPs *(SOP Master Index)* for both Method and Process SOPs. Method SOPs are maintained to describe a specific test method. Process SOPs are maintained to describe function and processes not related to a analytical testing (e.g., administrative procedures).

Method SOPs contain the following information, but not necessarily in the order listed:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 6).

1. Identification of Test Method
2. Applicable Matrix
3. Scope and Application, including test analytes
4. Summary of the Test Method
5. Reporting Limits
6. Definitions
7. Interferences
8. Safety
9. Equipment and Supplies
10. Reagents and Standards
11. Sample Collection, Preservation and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Calculations
16. Method Performance
17. Data Assessment and Acceptance Criteria for Quality Control Measures
18. Corrective Actions for Out-of-Control Data
19. Contingencies for Handling Out-of-Control or Unacceptable Data
20. Waste Management/Pollution Prevention
21. References
22. Tables, Diagrams, Flowcharts and Validation Data
23. Changes From Previous Revision

Process SOPs contain the following information, but not necessarily in the order listed.

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 6).

1. Scope
2. Summary
3. Definitions
4. Responsibilities
5. Procedure
6. References
7. Tables, Diagrams, and Flowcharts
8. Changes from Previous Revision

The QA Department is responsible for maintenance of SOPs, archival of SOP historical revisions, maintenance of an SOP Master Index, and records of controlled distribution. SOPs, at a minimum, undergo annual review (12 months). Where an SOP is based on a published method, the laboratory maintains a copy of the reference method.

Figure 6. Proprietary Information Statement

This documentation has been prepared by Severn Trent Laboratories, Inc. (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to STL upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

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SOP Interim Change Form

The SOP Interim Change Form is used for implementation, documentation, and authorization of changes to SOPs *(Procedure for Writing, Reviewing and Revising SOPs, AQA-SOP-55)*. Immediate changes in SOPs may be necessary to accommodate improvements; to implement acceptable changes in practices; or to correct potential errors in the existing version. The reason for the change will be identified and a detailed description of the procedure change will be presented. Since this form will become part of the referenced SOP, until such time that the SOP is updated, it must be legible and comprehensible. The Interim Change Form must provide an exact description and identify the affected sections.

Once this form is completed and changes are authorized, it becomes an official part of the SOP for which it revises, and is subject to all document control and records management policies.

5.3.3 Method Validation

Laboratory developed methods are validated and documented according to the procedure described in Section 5.3.5.

5.3.4 Method Verification

Method verification is required when a validated standard test method or a method modification is implemented. The level of activity required for method verification is dependent on the type of method being implemented, or on the level of method modification and its affect on a method's robustness. Method modification often takes advantage of a method's robustness, or the ability to make minor changes in a method without affecting the method's outcome.

It is the responsibility of the Operations Manager to present to the QA Manager all applicable method validation studies for review and approval. The documented approval by the Operations Manager, Department Supervisor and QA Manager must be applied to all applicable validation records before the method is released for use. Method verification may require some, but not all, of the activities described in Section 5.3.5.

5.3.5 Method Validation and Verification Activities

Before analyzing samples by a particular method, method validation and/or method verification must occur. A complete validation of the method is required for laboratory developed methods. While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

Determination of Method Selectivity
Method selectivity is demonstrated for the analyte(s) in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

Determination of Method Sensitivity
Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. The laboratory determines MDLs are described in Section 4.4.3.6 and the corporate procedure for *MDL Policy, (S-Q-003).*

Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)
An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system.

When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data are to be reported in this region, they must be done so with a qualification that denotes the semi-quantitative nature of the result.

Determination of Interferences
A determination that the method is free from interferences in a blank matrix is performed.

Determination of Range
Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

Demonstration of Capability
DOCs are performed prior to method performance.

Determination of Accuracy and Precision
Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

Documentation of Method
The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Appendix describing the specific differences in the new method is acceptable in place of a separate SOP.

Continued Demonstration of Method Performance
Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS and Method Blanks.

5.3.6 Data Reduction and Review

Analytical data are entered/downloaded directly into LIMS or recorded on pre-formatted bench sheets that are paginated and bound into laboratory logbooks. These logbooks are issued and controlled by the laboratory's QA Section. A unique document control code is assigned to each book to assure that chronological record keeping is maintained. Analytical data may also be electronically stored as a secure .pdf file.

Analytical data are referenced to a unique sample identification number for internal tracking and reporting. Both LIMS entries and logbook pages contain the following information, as applicable: analytical method, analyst, date, associated sample numbers, standard concentrations, instrument settings, and raw data. Entries are in chronological order and maintained so as to enable reconstruction of the analytical sequence.

The analyst is responsible for entering / recording all appropriate information, and for signing and dating all logbook entries daily. All entries and logbook pages are reviewed for completeness by a supervisor, peer reviewer or the analyst themselves. Data review checklists document the analytical review of the LIMS entries, logbook and associated QC indicators. Copies of instrument outputs

(chromatograms, mass spectra, etc.) are maintained on file or electronically with the analyst's signature/initials and date.

5.3.6.1 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data are reduced by the analyst and updated to the LIMs. Both the data entry and raw data are then verified by the department supervisor or alternate analyst. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the data and manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the STL Corporate SOP entitled *Acceptable Manual Integration Practices (S-Q-004).*

Copies of all raw data and the calculations used to generate the final results, such as bound logbooks, are retained on file for a minimum of 5 years or as otherwise requested by the client/project.

Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

5.3.6.2 Data Review

All data, regardless of regulatory program or level of reporting, are subject to a thorough review process. The individual analyst continually reviews the quality of the data through calibration checks, quality control sample results and performance evaluation samples. Data review is initiated by the analyst during, immediately following, and after the completed analysis.

All levels of the review are documented on Data Review Checklists that are specific to each laboratory section *(Technical Data Review; AGP-DataReview-21).*

Primary Review

The primary review is often referred to as a "bench-level" review. In most cases, the analyst who generates the data (e.g., logs in, prepares and/or analyzes the samples) is the primary reviewer. In some cases, an analyst may be reducing data for samples run by an auto-sampler set up by a different analyst. In this case, the identity of both the analyst and the primary reviewer is identified in the raw data.

One of the most important aspects of primary review is to make sure that the test instructions are clear, and that all project specific requirements have been understood and followed.

Once an analysis is complete, the primary reviewer ensures, where applicable, that:

- Sample preparation information is complete, accurate, and documented.
- Calculations have been performed correctly.
- Quantitation has been performed accurately.
- Qualitative identifications are accurate.

- Manual integrations are appropriate.
- Data flags to indicate manual integrations are recorded.
- Manual integrations are authorized by a date and signature or initials of primary analyst.
- Client specific requirements have been followed.
- Method and process SOPs have been followed.
- Method QC criteria have been met.
- QC samples are within established limits.
- Dilution factors are correctly recorded and applied.
- Non-conformances and/or anomalous data have been properly documented and appropriately communicated.
- COC procedures have been followed.
- Primary review is documented by date and initials/signature of primary analyst.
- All unused portions of hardbound logbooks are 'Z'ed out; corrections are made with a single line drawn through the error and are dated and initialed

Any anomalous results and/or non-conformances noted during the Primary Review are documented on the Data Review Checklist and on a Job Exception ; and are communicated to the Supervisor and the Project Manager for resolution. Resolution can require sample reanalysis, or it may require that data be reported with a qualification. Non-conformances are documented per Section 4.9. Case narrative comments are generated by the primary reviewer for any unresolved anomalous results or non-conformances.

Secondary Review

The secondary review is also a complete technical review of a data and is performed by the Supervisor, peer analyst or data specialist. The secondary review is documented on the same Data Review Checklist as the primary review.

The following items are reviewed:

- Qualitative Identification
- Quantitative Accuracy
- Calibration
- QC Samples
- Method QC Criteria
- Adherence to method and process SOPs
- Accuracy of Final Client Reporting Forms
- Manual Integrations – Minimal requirement is to spot-check raw data files for manual integration, as verified by date and initials or signature (hardcopy or electronic) of secondary data reviewer. Some regulatory programs require 100% secondary review of manual integrations.
- Completeness
- Special Requirements/Instructions
- Review and approve case narrative comments

If problems are found during the secondary review, which are documented on the data review checklist, the reviewer must work with the appropriate personnel to resolve them. If changes are made to the data, such as alternate qualitative identifications, identifications of additional target analytes, re-quantitation, or re-integration, the secondary reviewer must contact the laboratory analyst and/or primary reviewer of the data so that the primary analyst and/or reviewer is aware of the appropriate reporting procedures.

Completeness Review
The completeness review includes the review of the case narrative which outlines anomalous data and non-compliances using project narrative notes, Job Exceptions and DQRs generated during the primary and secondary review. The completeness review addresses the following items:

- Is the project report complete?
- Does the data meet with the client's expectations?
- Were the data quality objectives of the project met?

Are QC outages and/or non-conformances approved and appropriately explained in the narrative notes?

The laboratory Department Supervisor, Data Reporting personnel and the Project Manager contribute to the completeness review.

5.3.7 Data Integrity and Security

This section details those procedures that are relevant to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data.

Security and Traceability
Access to the laboratory's LIMS system that collects, analyzes, and processes raw instrumental data, and those that manage and report data is both controlled and recorded. System users are granted access levels that are commensurate with their training and responsibilities.

Control of the system is accomplished through limitation of access to the system by users with the education, training and experience to perform the task knowledgeably and accurately. System users are granted privileges that are commensurate with their experience and responsibilities.

Computer access is tracked by using unique login names and passwords for all employees that have access to the computer system. Entries and changes are documented with the identity of the individual making the entry, and the time and date. Where a computer system is processing raw instrumental data, the instrument identification number as described in Section 5.4.1 is recorded. The system has the capability of maintaining audit trails to track entries and changes to the data. This function is activated on any computer system that has that capability (e.g., Enviroquant, Chemstation, TotalChrom).

Verification
All the LIMS software programs have been verified prior to use and prior to the implementation of any version upgrades. Verification involves assessing whether the computer system accurately performs its intended function. Verification generally is accomplished by comparing the output of the program with the output of the raw data manually processed, or processed by the software being replaced. The verification of LIMS software programs are conducted by the Information Technology Manager with the assistance of the QA Manager, Operations Manager and the Department Supervisors. The IT Manager documents the approval of the program verifications. All records of the verification are retained as QC records.

Validation

Software validation involves documentation of the verification of final calculated results. Software validation is performed by the QA manager on all in house programs. Records of validation include original specifications, identity of code, printout of code, software name, software version, name of individual writing the code, comparison of program output with specifications, and verification records as specified above. Records of validation are retained as QC records.

The QA manager must retain documentation of the validation process as defined above. The designated LIMS methods administrator at the laboratory has the responsibility to validate any LIMS methods, calculations or criteria codes prior to use for sample analysis.

Auditing
STLs LIMS System Managers continually review the control, security, and tracking of IT systems and software.

Version Control
The laboratory maintains copies of outdated versions of software and associated manuals for all software in use at the laboratory for a period of 5 years from its retirement date. The associated hardware, required to operate the software, is also retained for the same time period.

5.4 Equipment

5.4.1 Equipment Operation

STL is committed to routinely updating and automating instrumentation. The laboratory maintains state of the art instrumentation to perform the analyses within the QC specifications of the test methods. The laboratory maintains an Equipment List *(STLBuffEquipList)* that documents the following information:

- Identity
- Date In Service
- Manufacturer's Name, Model Number, Serial Number
- Current Location

All equipment is subject to rigorous checks upon its receipt, upgrade, or modification to establish that the equipment meets with the selectivity, accuracy, and precision required by the test method for which it is to be used. All manufacturer's operations and maintenance manuals are kept up to date and accessible for the use of the equipment operator. Documentation of equipment usage is maintained using analytical run and maintenance logbooks.

5.4.2 Equipment Maintenance

STL employs a system of preventative maintenance in order to ensure system up time, minimize corrective maintenance costs and ensure data validity. All routine maintenance is performed as recommended by the manufacturer and may be performed by an analyst, instrument specialist or outside technician. Maintenance logbooks are kept on all major pieces of equipment in which both routine and non-routine maintenance is recorded.

Any item of equipment or instrumentation that has been subjected to overloading or mishandling, provides suspected results, has been shown by verification or otherwise to be defective, is new or

not been used for an extended period of time, is taken out of service and tagged as "OUT-OF-SERVICE", *(AGP-OutofService-65)*

Any instrumentation that is brought back on-line must have MDLs and DOCs performed and have acceptance within prescribed criteria; or calibrated by a certified agency (e.g., balances or Class S weights) and tagged as being within calibration specifications; and proven to provide consistent measurements (e.g., refrigerators, eppendorf pipettes, ovens).

The return to analytical control following instrument repair is documented in the maintenance logbook. Notation of the date and maintenance activity is recorded each time service procedures are performed. Maintenance logbooks are retained as QA records.

Maintenance contracts are held on specific pieces of equipment where outside service is efficient, cost-effective, and necessary for effective operation of the laboratory. Table 9 lists STL's major equipment and the suggested maintenance procedures.

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check tubing for wear	Daily
	Fill rinse tank with 10% HCl	Daily
	Change dryer tube	As Needed
	Fill reductant bottle with 10% Stannous Chloride	Daily
ICP & ICP/MS	Check pump tubing	Daily
	Check liquid argon supply	Daily
	Check fluid level in waste container	Daily
	Check re-circulator levels	Monthly
	Clean or replace filters	As required
	Check torch	Daily
	Check sample spray chamber for debris	Monthly
	Clean and align nebulizer	Monthly
	Change pump oil	Monthly
	Change Cones	As required
	Change printer cartridge	As required
	Replace pump tubing	As required
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Precision check/alignment of flow cell	As required
	Wavelength verification check	Annually
Auto Analyzers	Clean sampler	Daily
	Check all tubing	Daily
	Clean inside of colorimeter	Daily
	Clean pump well and pump rollers	Quarterly
	Clean wash fluid receptacle	Weekly
	Oil rollers/chains/side rails	Weekly
	Clean optics and cells	Quarterly

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Agilent GC/MS	Pump oil-level check	Monthly
	Pump oil changing	Annually
	Analyzer bake-out	As required
	Analyzer cleaning	As required
	Resolution adjustment	As required
	COMPUTER SYSTEM AND PRINTER:	
	Air filter cleaning	As required
	Change data system air filter	As required
	Printer head carriage lubrication	As required
	Paper sprocket cleaning	As required
	Drive belt lubrication	As required
Gas Chromatograph	Compare standard response to previous day or since last initial calibration	Daily
	Check carrier gas flow rate in column	Daily via use of known compound retention
	Check temp. of detector, inlet, column oven	Daily
	Septum replacement	As required
	Glass wool replacement	As required
	Check system for gas leaks with SNOOP	W/cylinder change as required
	Check for loose/frayed power wires and insulation	As Required
	Bake injector/column	As Required
	Change/remove sections of guard column	As Required
	Replace connectors/liners	As Required
	Change/replace column(s)	As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63)	Semi-annually
	Detector cleaning	As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings	As required
	Clean lamp window	As required
HPLC	Change guard columns	As required
	Change lamps	As required
	Change pump seals	Semi-annually or as required
	Replace tubing	As required
	Change fuses in power supply	As required
	Filter all samples and solvents	Daily
	Change autosampler rotor/stator	As required
Balances	Class "S" traceable weight check	Daily, when used
	Clean pan and check if level	Daily
	Field service	At least Annually

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Conductivity Meter	0.01 M KCl calibration Conductivity cell cleaning	Weekly As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Check conductivity Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Daily Daily Daily As required As required
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Weekly As required
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Daily Monthly
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed

5.4.3 Equipment Verification and Calibration

All equipment is calibrated prior to use (Initial Calibration) to establish its ability to meet the QC guidelines contained in the test method for which the instrumentation is to be used. All sample measurements are made within the calibrated range of the instrument and in compliance with method requirements. The calibration data, which includes instrument conditions and standard concentrations, is documented in pre-formatted instrument injection logs or within LIMS itself. The preparation of all reference materials used for calibration is documented in standards preparation logbooks in accordance with *SOP AGP-STD-14 (Standards Traceability and Preparation Logbooks).*

Once an instrument is calibrated, ongoing instrument calibration is demonstrated (Continuing Calibration) at the appropriate frequency as defined in the test method. Refer to the STL Corporate Policy *Selection of Calibration Points (P-T-001),* for guidance on using calibration data. Any instrument that is deemed to be malfunctioning is clearly marked and taken out of service. When the instrument is brought back into control, acceptable performance is documented.

5.4.3.1 Instrument Calibration

Specific instrument calibration procedures for various instruments are summarized further in this section, and detailed in the respective analytical methods. Typically, more than one analytical method is available for an analysis. These various methods and other program requirements (e.g., U.S. EPA CLP, AFCEE, USACE, QAPPs, contracts, etc.) may specify different calibration requirements. Therefore, calibration details as specified in the respective laboratory SOPs, Technical Profiles, QAPP, program requirements, and contracts supersede the general instrument calibration procedures are described further in Table 10. Complete details are provided in each method SOP.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
Metals (ICAP)	Initial Calibration	Following a period of time sufficient to warm up the instrument, the ICP is calibrated prior to each analytical run or minimally every 24 hours. Calibration standards are prepared from reliable reference materials and contain all metals for which analyses are being conducted. Working calibration standards are prepared fresh daily. Prior to an analytical run, the instrument is calibrated using appropriate standards. An Initial Calibration Verification (ICV) standard is analyzed immediately after standardization, followed by an Initial Calibration Blank (ICB). The ICV is from a source other than that used for initial calibration and the ICB must be free of target analytes at and above the value to be reported or appropriate corrective action must be taken. ICP Interference Check Samples (ICSAB) are analyzed at the frequency described in each method SOP.
	Continuing Calibration	The initial calibration is verified during the analysis sequence by analysis of a Continuing Calibration Verification (CCV) standard and a Continuing Calibration Blank (CCB). The response of the CCV must be within the SOP-specified criteria (e.g., ± 10% recovery of the true value). The CCB must be free of target analytes at or above the value to be reported or appropriate corrective action must be taken. If any ICVs/CCVs or blanks exceed their acceptance criteria, appropriate corrective action must be taken.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
Inorganic Colorimetric Methods	Initial Calibration	A full initial standard calibration curve will be prepared for all colorimetric analyses. Working standards to define this curve will include a minimum of five (5) concentrations which cover the anticipated range of measurement, plus a calibration blank. At least one of the calibration standards will be at a concentration which will enable verification of instrument response near the reporting limit as defined in Section 8.6 or a level suitable for meeting specific program requirements. The requirement for an acceptable initial calibration is described in the analytical SOP. If the criteria are not met, appropriate corrective action must be taken. Calibration data, e.g., correlation coefficient, is entered into the laboratory notebook, or associated instrument printouts, and retained with the sample data. If the initial curve is not analyzed that day, a daily calibration verification must be analyzed. This daily calibration will at a minimum consist of a blank and a mid-range standard. Results must be within SOP-specified criteria. If not, reanalysis of the standards may be done once to verify the readings; otherwise, a new curve will be developed.
Inorganic Colorimetric Methods (cont'd.)		For procedures that require pretreatment steps, a minimum of one standard shall be prepared with the pretreatment. If the pre-treated standard is within SOP-specified criteria, the curve will be used. If the pre-treated sample is not within the criteria, the reason will be determined. If it is determined that the difference between the curves is inherent in the procedure, the curve will be based on the standards prepared and carried through the pretreatment. An ICV will be analyzed immediately after the standardization, followed by an ICB. The ICV must be from a source other than that used for initial calibration. The ICV must be within SOP-specified criteria and the ICB must be free of target analytes or appropriate corrective action must be taken.
	Continuing Calibration	The initial calibration is verified during the analysis sequence by analysis of a CCB and a CCV. If any ICVs/CCVs or blanks exceed their acceptance criteria, analysis is terminated, and the instrument is recalibrated. All samples since the last valid calibration verification are evaluated for acceptability or reanalyzed. (If the CCV is elevated and the samples are ND, the data are deemed acceptable.)
Ion Chromatography	Initial Calibration	The ion chromatograph will be calibrated every three months or sooner if calibration verification can not be achieved. Calibration standards will be prepared from appropriate reference materials and will include a blank and a minimum of three concentrations to cover the anticipated range of measurements. At least one of the calibration standards will be at a concentration which will enable verification of instrument response near the reporting limit. If SOP-specified calibration criteria cannot be achieved, appropriate corrective action must be taken. Calibration data, e.g., correlation coefficient, will be archived with sample raw data.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Continuing Calibration Verification	A calibration verification standard and blank will be analyzed each day prior to sample analysis, throughout the sequence at a frequency of 10% and at the end of the analysis shift. The response calculated as a percent recovery of the standard must meet SOP or program-specific criteria. The response of the blank must be less than the concentration to be reported for samples analyzed.
GC/MS	All GC/MS instrumentation is calibrated to set specifications prior to sample analysis. These specifications vary depending on the requirements of the analytical program and the designated analytical method.	
	Tuning and Mass Calibration	Mass spectrometers are calibrated with perfluorotributylamine (FC-43) or perfluorophenanthrene (FC- 5311) as required to ensure correct mass assignment. In addition, at the beginning of the daily work shift, the GC/MS system must be tuned with decafluorotriphenylphosphine (DFTPP) for semivolatiles analysis and 4-bromofluorobenzene (BFB) for volatiles analysis, and calibrated to target compounds. Autotunes are run with PFTBA(perfluorotributylamine),which is encased in a vial inside the mass spec. DFTPP and BFB are run daily (12 hours where appropriate) for SVOA and VOA respectively.
GC/MS (cont'd.)	Tuning and Mass Calibration	Laboratory work using SW-846 protocols, defines the work shift as a 12-hour period initiated by the injection of DFTPP or BFB. For drinking water programs (500 series methods), a 12-hour work shift is specified in the method for calibration frequency. For wastewater programs (600 series methods), the tune expires when the day's analytical sequence is complete; however, no time limit is given for the length of the daily GC/MS work shift; therefore a maximum of 24 hours for 624 and 625 is used. Ion abundances will be within the windows dictated by the specific program requirements.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Initial Calibration	After an instrument has been tuned, initial calibration curves (generally 3-5 points) are generated for the compounds of interest. The low level standard must be at a concentration which will enable verification of instrument response near the reporting limit or at a concentration acceptable to meet program requirements. The other standards must extend through the linear working range of the detector. The parameters requiring quantitation must meet SOP or program-specified criteria prior to initiation of sample analysis. Any sample extracts containing parameters of interest which exceed the concentration of the high level standard, must be diluted to bring the parameters within the range of the standards. Instrument response to these target compounds are evaluated against SOP-specified criteria. Linearity is verified by evaluating the response factors (RF) for the initial calibration standards against SOP-specified criteria. Once an acceptable calibration is obtained, samples may be analyzed up until the expiration of the tune. At that time, the instrument must be re-tuned prior to further analysis. After acceptable tuning, a continuing calibration standard may be analyzed in lieu of a full multi-point calibration if the SOP-specified criteria are met. The majority of compounds analyzed for GC/MS comprise EPA's Target Compound List (TCL) or Priority Pollutant List (PPL). For add-on compounds not on the current TCL or PPL, initial calibration may be performed using a single point calibration of the additional compound(s), unless prior arrangements are made for a full three-to-five point calibration. Calibration data, to include linearity verification, will be maintained in the laboratory's records of instrument calibrations. 3 to 5 point curves for all GCMS analytes, special list. The only case where single point standards are used is for quantitation of PCBs other than Arochlor 1016 or 1260.
	Continuing Calibration	During each operating shift, a single calibration standard may be analyzed to verify that the instrument responses are still within the initial calibration determinations, as defined in the specific SOPs. If criteria cannot be met, appropriate corrective action must be taken.
GC and HPLC	Gas chromatographs and high performance liquid chromatographs will be calibrated prior to use as described in analytical SOP or program requirements. Calibration standard mixtures will be prepared from appropriate reference materials and will contain analytes appropriate for the method of analysis or program requirements	
GC and HPLC (cont'd.)	Initial Calibration	Initial calibration will include three or more calibration standards covering the anticipated range of measurement. The low level standard must be at a concentration which will enable verification of instrument response near the reporting limit or at a concentration acceptable to meet program requirements. The other standards must extend through the linear working range of the detector. The parameters requiring quantitation must meet SOP or program-specified criteria prior to initiation of sample analysis. Any sample extracts containing parameters of interest which exceed the concentration of the high level standard, must be diluted to bring the parameters within the range of the standards.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Continuing Calibration	The response of the instrument will be verified for each analysis sequence by evaluation of a daily calibration verification standard at a mid-range concentration. In order to demonstrate that the initial calibration curve is still valid, the calibration check standard must be within SOP or program-specified acceptance criteria for the compounds of interest or the instrument must be recalibrated. For multi-analyte methods, this check standard may contain a representative number of target analytes rather than the full list of target compounds. Optionally, initial calibration (e.g., the full range of concentration levels) can be performed at the beginning of the analysis sequence. Within the analysis sequence, instrument drift will be monitored by analysis of a mid-range calibration standard every ten samples or 12 hour sequence (depending on the method protocol), including external QC. If the SOP or program-specified calibration criteria are not met for the compounds of interest, appropriate corrective action must be taken.

5.5 Measurement Traceability

5.5.1 General

Traceability of measurements is assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard is subject to ongoing certifications of accuracy.

At a minimum, these include procedures for checking specifications for balances, thermometers, temperature, De-ionized (DI) and Reverse Osmosis (RO) water systems, automatic/eppendorf pipettes and other volumetric measuring devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards [with the exception of class A glassware (including glass microliter syringes that have a certificate of accuracy)].

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balances are calibrated on each day of use. All thermometers and temperature monitoring devices are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use

Laboratory DI and Elga water systems have documented preventative maintenance schedules and the conductivity of the water is recorded on each day of use

Procedures for maintenance and record keeping of support equipment are defined in SOP *Support Equipment: Maintenance, Record Keeping and Corrective Actions of Analytical Balances, Temperature Control Devices and Reagent Water (AGP-SupportEquip-02)*

5.5.2 Reference Standards

The receipt of all reference standards is documented in the departmental Chemical History Logbook. Standards are obtained from commercial vendors and sources may vary depending upon the availability of mixes and solutions from vendors. Each production unit is responsible to ensure, when available, that all standards are traceable to EPA, NIST or A2LA and are accompanied by a Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis.

The receipt of each dry chemical, purchased stock solution or reference material to be used as a standard is assigned a unique ID number. The chemical name, manufacturer, lot number, date received, expiration date, date opened and initials of the analyst who opened the chemical are documented. The expiration dates for ampulated solutions shall not exceed the manufacturer's expiration date. Expiration dates for laboratory-prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials shall be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory labels all standard and QC materials with expiration dates.

The preparation of all daughter solutions, whether a single or multiple-component stock, intermediate, or working standard solution, is documented in a standard solution preparation logbook. This documentation references the Standard ID of the respective parent solution(s) used in its preparation, providing a solid trail back to the solution or chemical received from the vendor. These records include the standard name, final volume, matrix, final concentration, analyst initials, prep date and expiration date. A daughter solution should not have an expiration date which post-dates any of the parent solutions used in its preparation.

References standards are labeled with a unique Standard Identification Number, date received, and the expiration date. All documentation received with the reference standard or documentation of standard purity is retained as a QC record and references the Standard Identification Number. All efforts are made to purchase standards that are $\geq$ 97.0% purity. If this is not possible, the purity is used in performing standards calculations.

The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a different lot is acceptable for use as a second source. The appropriate QC criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an ICV or LCS is used as the second source confirmation.

Storage conditions, such as shelf life, ambient or chilled, controlled or restricted access, wet or desiccated, etc., are in conformance with the specifications set in the associated method, the program requirements, or the manufacturer's recommendation, as appropriate.

5.5.3 Reagents

Reagents are, in general, required to be analytical reagent grade unless otherwise specified in method SOPs. Reagents must be, at a minimum, the purity required in the test method. The date

of reagent receipt and the expiration date as well as the date of reagent preparation (where applicable) are documented in the standards preparation logbooks.

5.6 Sampling

Sample representativeness and integrity are the foundations upon which meaningful analytical results rely. Where documented and approved SAPs and/or QAPPs are in place, they must be made available to the laboratory before sample receipt, and approved by laboratory management before sample receipt.

5.7 Sample Handling, Transport, and Storage

5.7.1 General

COC can be established either when bottles are sent to the field, or at the time of sampling. STL can provide all of the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory. Complete details for sample container preparation are contained within *Sample Container Preparation and Shipment SOP (ASR-Bottle-03)*. A summary of sample receipt is as follows with complete details available within the *Receipt of Analytical Samples SOP (ASR-Receipt-05).*

Samples are received at the laboratory by the designated sample custodians and a unique LIMS job number is assigned. The following information is recorded for each sample shipment:

- Client/Project Name.
- Date and Time of Laboratory Receipt.
- Laboratory Job Number
- Signature or initials of the personnel receiving the cooler and making the entries.

Upon inspection of the cooler and custody seals, the sample custodian opens and inspects the contents of the cooler, and records the cooler temperature. If the cooler arrival temperature exceeds the required or method specified temperature range by $\pm 2^{\circ}C$ (for samples with a temperature requirement of $4^{\circ}C$, a cooler temperature of just above the water freezing temperature to $6^{\circ}C$ is acceptable); sample receipt is considered "compromised" and the procedure described in Section 4.7.1 is followed. All documents are immediately inspected to assure agreement between the test samples received and the COC.

Any non-conformance, irregularity, or compromised sample receipt as described in Section 4.7.1 is documented in an Analytical Receipt Resolution Form (ARRF) and brought to the immediate attention of the Project Manager for resolution with the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the permanent project record.

Samples that are being tested at another STL facility or by an external subcontractor are repackaged, iced, and sent out under COC.

Following sample labeling as described in Section 5.7.2, the sample is placed in storage. Refrigerated storage coolers are maintained at $4 \pm 2^{\circ}C$ and the temperatures are monitored daily.

All samples are stored according to the requirements outlined in the test method, and in a manner such that they are not subject to cross contamination or contamination from their environment.

Access to the laboratory is restricted to laboratory personnel or escorted guests as described in Section 5.2. Therefore, once sample possession is relinquished to the laboratory, the sample is in a designated secure area (e.g., the laboratory facility) accessible only to authorized personnel. Locked storage coolers are available for protocol that require internal COC procedures.

5.7.2 Sample Identification and Traceability

The sample custodian organizes the sample containers, COCs, and all pertinent information associated with the samples. The sample identity is verified against all associated sample information. Any inconsistencies are documented via an ARRF and forwarded to the Project Manager for resolution with the client prior to identifying the sample(s) into LIMS.

Each sample container is assigned a unique Sample Identification Number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label.

All unused portions of samples, including empty sample containers, are returned to the secure sample control area, unless it has been documented that the container was disposed.

5.7.3 Sub-Sampling

Taking a representative sub-sample from a container containing a soil or solid matrix is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation.

After thoroughly mixing the sample within the sample container or transfer to a suitable plastic bag, a sub-sample from various quadrants and depths of the sample are taken to acquire the required sample weight. Any non-homogenous looking material is avoided and noted as such within the sample preparation record.

5.7.4 Sample Preparation

Sample preparation procedures vary for each matrix and analytical method are as referenced in the laboratory SOPs.

5.7.5 Sample Disposal

Samples are retained in STL storage facilities for 30 days after the project report is sent unless prior written arrangements have been made with the client. Samples may be held longer or returned to the client per written request. Unused portions of samples are disposed of in accordance with federal, state and local regulations. The laboratory removes or defaces sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). Complete details on the disposal of samples, digestates, and extracts is available within the *Sample Disposal SOP (ASR-DISP-33) and Hazardous Waste Management SOP (AWM-HazMg-01).*

5.8 Assuring the Quality of Test Results

5.8.1 Proficiency Testing

The laboratory analyzes Proficiency Test (PT) samples as required for accreditation and as outlined in NELAC. The laboratory participates in the PT program semi-annually for each PT field of testing for which it is accredited, according to the NELAC PT field of testing published guidelines. This includes drinking water, wastewater and solid/soil matrices.

The laboratory also participates in various client PT programs, when submitted.

PT samples are handled and tested in the same manner (procedural, equipment, staff) as environmental samples. Results of PT samples are distributed to the laboratory line management for review and action, if required. Any required response to deficiencies are submitted to the QA department for review and are filed with the PT study records. PT test sample data are archived using the requirements for project and raw data record retention.

5.8.1.1 Double Blind Performance Evaluation

The laboratory participates in an annual double blind performance evaluation study. An external vendor is contracted to submit double blind samples to the laboratory. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor, who provides a detailed report to the Corporate Quality Director and to the laboratory. This is administered as a double blind program in order to assess all facets of the laboratory's operations.

5.8.2 Control Samples

Control samples (e.g., QC indicators) are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity, selectivity, and interferences. Control samples must be uniquely identified and correlated to unique batches. Control samples further evaluate data based upon (1) Method Performance, which entails both the preparation and measurement steps; and (2) Matrix Effects, which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch.

Control sample types and typical frequency of their application are outlined Sections 5.8.2.1 through 5.8.2.5 and Tables 11 through 15. Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method and regulatory program control samples are as listed in Sections 7 and 8 typically of each method SOP.

5.8.2.1 Method Performance Control Samples: Preparation Batch

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment.

Control samples are added to each prep batch to monitor method performance (Table 11) and are processed through the entire analytical procedure with investigative/field samples.

Table 11. Preparation Batch Control Samples

Control Sample Type	Details	
Method Blank (MB)	Use	Monitors for potential contamination introduced during the sample preparation and analytical processes.
	Typical Frequency [1]	1 per batch of ≤ 20 samples per matrix type per sample extraction or preparation method.
	Description	Organics: Laboratory pure water for water samples or a purified solid matrix for soil or solid samples (when available or when requested); solid matrices commonly include sodium sulfate, vendor or agency supplied soil or solid, or purchased sand; these solids may require purification at the laboratory prior to use. Inorganics: Laboratory pure water for both water and soil or sediment samples. Volume/weights are selected to approximately equal the typical sample volume/weight used in sample preparation; and final results in a soil/solid batch may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison to actual field samples.
Laboratory Control Sample (LCS)	Use	Measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects.
	Typical Frequency [1]	1 per batch of ≤ 20 samples per matrix type per sample extraction or preparation method. For multi-analyte methods, the LCS may consist of surrogates in the blank matrix, and or a representative selection of target analytes/internal standards.
	Description	Prepared from a reference source of known concentration and processed through the preparation and analysis steps concurrently with the field samples. Aqueous LCS's may be processed for solid matrices unless a solid LCS is requested; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the actual field samples.
Known QC Sample	Use	Comply with regulatory requirements; check the accuracy of an analytical procedure; troubleshoot method performance problems; verify an analyst in training's ability to accurately perform a method; to verify the return-to-control after method performance problems; and may also be used as an LCS.
	Typical Frequency [1]	As defined by the client or QAPP.
Known QC Sample (cont'd.)	Description	Obtained from outside suppliers or agencies; generally require preparation from concentrated materials by dilution into a standard matrix; contain known analytes or compounds; acceptance limits are provided by the vendor.

[1] Denotes an STL required frequency.

Field blanks, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. However, a field blank should not be selected for matrix QC, as it does not

provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB".

5.8.2.2 Method Performance Control Samples: Matrix

Matrix control samples include sample duplicates (MD), sample matrix spikes (MS), and sample surrogate spikes. These control samples help monitor for potential physical and chemical effects which may interfere with the precision and/or accuracy of the selected analytical method. Since interferences can enhance or mask the presence of target analytes, matrix control samples measure the degree of interference and are used to assist in the interpretation of the analytical results. The laboratory avoids performing matrix QC on known field blank samples, such as trip blanks and rinsates, since these samples are not indicative of the sample matrix.

Table 12. Matrix Control Samples

Control Sample Type	Details	
Matrix Duplicate (MD)	Use	Monitors the effect of site matrix on the precision of the method; and of the reproducibility of laboratory preparation and measurement techniques. **Note:** Precision may also be affected by the degree of homogeneity of the sample, particularly in the case of non-aqueous samples or aqueous samples with particulates. Sample homogeneity and matrix effect should be considered when field samples are used to assess reproducibility. **Note:** A field duplicate, when received, measures Representativeness of sampling and the effect of the site matrix upon precision.
Matrix Duplicate (MD) (cont'd.)	Typical Frequency [1]	1 per 20 samples per matrix or per SAP/QAPP [2].
	Description	Performed by analyzing two aliquots of the same field sample independently; analyzed for each associated sample matrix (e.g., when requested by the client or the analytical method).
Matrix Spike (MS)	Use	Measures the effect of site sample matrix on the accuracy of the method.
Matrix Spike (MS) (cont'd.)	Typical Frequency [1]	1 per 20 samples per matrix or per SAP/QAPP.
	Description	Aliquot of a field sample which is spiked with the analytes or compounds of interest; analyzed for each associated sample matrix (when requested by the client or analytical method). The determination of MS percent recovery (% R) requires an analysis of a fortified sample and a non-fortified sample under the same procedural conditions (e.g., sample volumes, dilutions, procedural conditions, etc.). The concentration determined in the non-fortified sample is subtracted from the fortified sample concentration before determining the %R. The degree of homogeneity of the sample, particularly in the case on non-aqueous samples or samples with particulates, may affect the ability to obtain representative recoveries.
Matrix	Use	Measures effect of site sample matrix on precision of method.

Table 12. Matrix Control Samples

Control Sample Type	Details	
Spike Duplicate (MSD)	Typical Frequency [1]	1 per 20 samples per matrix, when requested by the client or the analytical method, or per SAP/QAPP [2].
	Description	Alternative to sample duplicate. Generally, inorganic protocols specify an MD/MS and organic protocols specify an MS/MSD.
Surrogate Spike	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Every QC and analytical sample.
	Description	Compounds similar to the target analytes in structure, composition and chromatography, but not typically found in the environment, are added to each QC and analytical sample, prior to preparation (e.g., extraction). If the surrogates in an analytical batch do not all conform to established control limits, the pattern of conformance in investigative and control samples is examined to determine the presence of matrix interference or the need for corrective action.
Internal Standards	Use	Monitor the qualitative aspect of organic and inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] Denotes an STL required frequency.
[2] Either an MSD or an MD is required per 20 samples per matrix or per SAP/QAPP.

5.8.2.3 Matrix QC Frequencies

The frequency of matrix QC indicators depends on regulatory program compliance, a project's data quality objectives, or a client's requirements. The following frequency will be applied to samples when the regulatory programs are known and it does not conflict with project or client requirements.

Table 13. EPA Program Requirements

Program	Description [1]
SDWA	MD performed at a 10% frequency or 1 per preparation batch of ≤10 samples, whichever is more frequent.
CWA	MS (GC methods) and MD is performed at a 10% frequency or 1 per preparation batch of ≤10 samples, whichever is more frequent. For GC/MS Methods, MS is performed at a 5% frequency or 1 per preparation batch of ≤20 samples, whichever is more frequent.
RCRA	MS/MSD or MS/MD is performed at a rate of 5% per client (independent of the preparation batch). For clients submitting less than 10 samples, the method matrix QC requirement may be satisfied by another client's sample within the same prep batch unless the paperwork indicates a client requirement for matrix QC.
U.S. EPA CLP	MS/MSD or MS/MD is performed at a rate of 5% or 1 set per Sample Delivery Group (SDG) per matrix, independent of the prep batch. Samples are processed in simultaneous or continuous batches.

[1] MS, MSD and MD may not be applicable to some analytical protocols because of the nature of the sample or protocol.

5.8.2.4 Method Performance Control Samples: Instrument Measurement

Control samples are used to ensure that optimum instrument performance is achieved. These samples help ensure that the proper identification and quantitation of target compounds or analytes are achieved. The instrument control samples appropriate to each analytical technique are described in laboratory SOPs for each respective method. A brief description of these checks is included in Table 14.

Table 14. Instrument Performance Control Samples

Control Sample Type		Description
		Inorganics
ICV	Use	Calibration standard of known concentration prepared from a source other than that used for the calibration standards.
	Sequence	Analyzed after the standard curve to confirm calibration.
ICB	Use	Blank water or solvent; confirms the calibration and assures that any potential contamination is less than the reporting limit.
	Sequence	Analyzed immediately after the ICV.
ICP Interference Check Samples (ICSA/ICSB)	Use	Verifies the absence of spectral interferences.
	Sequence	Analyzed consecutively at the beginning of each eight hour analytical sequence, after the ICV/ICB, and again at an eight hour frequency following a CCV/CCB. When CLP protocols are followed, the ICSA/B will be analyzed with the analytical sequence, before the final CCV/CCB.
Reporting Limit Verification	Use	Verifies linearity near the reporting limit for CLP metals analyses. (Note: CRI is at a level 2X the CRDL; CRA is near the CRDL).
Standard (CRA and CRI)	Sequence	Performed only when analyzing CLP Samples or as specified by the client or program. Analyzed after the ICB. The CRI is also analyzed at the end of the eight hour analytical sequence, prior to analysis of the final CCV/CCB.
CCV	Use	Confirm that the instrument performance has not significantly changed during the analytical sequence; to verify stable calibration throughout the sequence; and/or to demonstrate that instrument response did not drift over a period of non-use. May be made from a source other than that used for the standard curve, however if the ICV is 2nd source, the CCV may be same source.
	Sequence	Analyzed at 10% or every two hours, whichever is more frequent; also analyzed at the end of the analytical sequence.
CCB	Use	Water blank used to confirm that the baseline has not drifted and to monitor for contamination at the reporting limit.
	Sequence	Analyzed at a rate of 10% for inorganics and at a rate of 1 per 10 readings/injections or every two hours, whichever is more frequent, for CLP metals; also analyzed at the end of the analytical sequence.
ICP Metals Linear Range	Use	Verify linearity and document the upper limit of the calibration range for each element.

Table 14. Instrument Performance Control Samples

Control Sample Type	Description	
Analysis Standard (LRS)	Sequence	Performed quarterly with a blank and a minimum of five standard concentrations to cover the anticipated range of measurement; one of the calibration standards will be at or near the reporting limit. The calibration curve generated must have a correlation coefficient $\geq$ 0.995 in order to consider the responses linear over that range.
ICP Inter-Element Correction (IEC)	Use	Correction factors for spectral interference (particularly due to Al, Ca, Fe, and Mg).
	Sequence	Determined at least annually for all wavelengths used for each analyte reported by ICP; or any time the ICP is adjusted in any way that may affect the IECs.
Organics		
GC/MS Tuning & Performance	Use	Ensures correct mass assignment and is monitored through response to target compounds during initial and continuing calibration, with minimum response criteria for specified system performance check compounds (SPCCs), and linearity is verified by evaluating the response factors (RF) for calibration check compounds (CCCs).
	Sequence	Tuned at the beginning of the daily work shift. Throughout the analysis, blanks, internal standard areas, surrogates, chromatographic baseline, resolution of peaks, and overall quality of the chromatography are used collectively to monitor instrument performance.
GC & HPLC Instrument Performance	Use	Monitored through retention time shift evaluation, linearity checks, and degradation checks of selected target compounds (e.g., for Endrin or DDT as appropriate).
GC & HPLC Instrument Performance	Sequence	Continuing calibration verification (e.g., blanks, shifts in chromatographic baseline or retention times, resolution of peaks, and overall quality of the chromatography) throughout the analytical sequence is accomplished through analysis of calibration check standards.

5.8.2.5 Method Performance Control Samples: Analysis Batch

Matrix specific control samples are used to assess the precision and accuracy of the method as applied to the specific sample matrix. These indicators provide information on sample matrix effects that is independent of the efficiency of the preparatory technique. The method performance control samples appropriate to each analytical technique are identified in the respective method. A brief description of these checks is included in Table 15.

These control samples are performed to provide a tool for evaluating how well the method performed for the respective matrix. These values are used by the client to assess the validity of a reported result within the context of the project's data quality objectives. For matrix specific QC results falling outside laboratory control limits which are attributed to matrix affects, no systematic corrective action is taken.

Table 15. Analysis Batch Performance Control Samples

Control Sample Type	Description	
ICP Serial Dilution	Use	5X Dilution of a field sample (performed at the instrument) to check for possible physical and/or chemical interferences.
	Sequence	5% of field samples or 1 per ≤20 samples per batch.
Method of Standard Addition (MSA)	Use	When specified by the analytical protocol or by client request.
	Sequence	When specified by the analytical protocol or by client request.

5.8.3 Statistical Control Limits and Charts

Statistical control limits and control charts are used to establish method performance of a given analysis and to monitor trends of QC results graphically over time. Once a data base of the laboratory results for a method/matrix/QC analyte combination is established, the acceptability of a given analysis of that QC parameter (and of the analytical batch to which it belongs) can be evaluated in light of the laboratory's normal performance. This is intended to help identify problems before they might affect data. Often, patterns of response that are not at all evident in sets of numbers are very distinct when the same values are viewed as a chronological graph.

Establishment of Limits

The purpose of using statistical control limits is to define, for each analyte in a given method/matrix/QC type combination, a range of expected values. This range encompasses the random variation that occurs normally in the laboratory and allows one to evaluate control samples in that context, rather than according to an arbitrary or external set of values. Limits for accuracy and precision are defined below:

Accuracy

As recoveries of a QC analyte in a given matrix are tabulated over time, a mean value for recovery is established, as is the standard deviation (s) of those recoveries. If the analysis is in statistical control (e.g., if the set of QC recoveries over time show random variation about the mean) approximately 99.7% of all recoveries for that QC will fall within three standard deviations (3s) of the mean. Thus, assuming that the mean itself is an acceptable level of recovery, the values corresponding to 3s above and 3s below the mean are defined as the Control Limits. Any single recovery outside these values is assumed to have resulted from some circumstance other than normal variation and shall be investigated.

Roughly 95% of points should fall within 2s of the mean. The values +2s and -2s are the Warning Limits. Any normal result has approximately a 1/20 chance of being between 2s and 3s from the mean, so a result in this region doesn't necessarily warrant corrective action, but attention should be paid to such points.

Precision

Precision is used to indicate matrix variability so that appropriate decisions can be made by the client when repeated analyses vary significantly. The coefficient of variation, expressed as a percentage (e.g., the %RSD) for the data set used to calculate accuracy control limits defines the control limit for precision. Duplicate analyses of the QC samples, such as duplicates or MS/MSD, should have an RPD less than or equal to this established precision control limit to be considered free of matrix interferences.

The laboratory calculates statistical control limits on an annual basis, or more frequently if change have been made to the analytical process which affects the chemistry of the method. Such limits are available on a project or QAPP-specific basis.

5.8.4 Calibration

Calibration protocols are method-specific, are briefly described in Table 10 and are defined in the Sections 6 & 7 of the method SOPs.

5.8.5 Glassware Cleaning

All glassware is thoroughly cleaned prior to use to ensure that sample integrity is not affected from artifacts caused by contaminated glassware.

A summary of general cleaning procedures follows with details provided in the *Laboratory Glassware Cleaning* SOP *(AGP-Glass-04):*

General laboratory glassware is cleaned with a low- or non-phosphate detergent, followed by thorough rinsing with tap water and deionized water.

Volumetric flasks and pipettes used for inorganics (method dependent), test tubes and caps used for micro-COD procedures, phosphate glassware, and metals-related glassware include an acid-washing step.

BOD glassware, includes use of EPA approved disposable plastic bottles or cleaning with a nitric or sulfuric acid and/or a NOCHROMIX-washing step.

Organic glassware includes a solvent-wash.

5.8.6 Permitting Departures from Documented Procedure

Where a departure from a documented SOP, test method, or policy is determined to be necessary, or unavoidable, the departure is documented in a Job Exception and reported in the case narrative. In most cases, these departures can be made with the approval of the Department Supervisor, Project Manager and the client. Issues of serious concern, as determined by the Operations Manager, Department Supervisor or Project Manager, will be brought to the attention of the Laboratory Director and/or QA Manager. In some instances, it is appropriate to inform the client before permitting a departure. The Project Manager, in consultation with the QA Manager, will make the determination as to the degree of notification required by the client.

On rare occasions, special analytical techniques will be requested for research, project specific requirements, or client needs. In these instances, SOPs may not be available, however, the analyst will thoroughly record the analytical steps and observations within a bound preformatted logbook.

5.8.7 Development of QC Criteria, Non-Specified in Method/Regulation

Where a method or regulation does not specify acceptance and/or rejection criteria, the laboratory must examine the data user's needs and the demonstrated sensitivity, accuracy and precision of the available test methods in determining appropriate QC criteria.

Data users often need the laboratory's best possible sensitivity, accuracy, and precision using a routinely offered test method, or are unsure of their objectives for the data. For routine test methods that are offered as part of STL's standard services, the laboratory bases the QC criteria on statistical information such as determination of sensitivity, historical accuracy and precision data, and method verification data. The method SOP includes QC criteria for ongoing demonstration that the established criteria are met (e.g., acceptable LCS accuracy ranges, precision requirements, method blank requirements, initial and continuing calibration criteria, etc.).

In some cases, a routine test method may be far more stringent than a specific data user's needs for a project. The laboratory may either use the routinely offered test method, or may opt to develop an alternate test method based on the data user's objectives for sensitivity, accuracy, and precision. In this case, it can be appropriate to base the QC criteria on the data user's objectives, and demonstrate through method verification and ongoing QC samples that these objectives are met.

For example, a client may require that the laboratory to test for a single analyte with specific DQOs for sensitivity, accuracy, and precision as follows: Reporting Limit of 10 ppm, Accuracy $\pm$25%, and RSD of <30%. The laboratory may opt to develop a method that meets these criteria and document through the Method Blank results, MDL study, and LCS results that the method satisfies those objectives. In this case, both the method and the embedded QC criteria have been based on the client's DQOs.

In some cases, the data user needs more stringent sensitivity, accuracy, and/or precision than the laboratory can provide using a routine test method. In this case, it is appropriate that the laboratory provide documentation of the sensitivity, accuracy, and precision obtainable to the data user and let the data user determine whether to use the best available method offered by the laboratory, or determine whether method development or further research is required.

5.9 Project Reports

The SOP for data package assembly and reporting formats is *ARP-Report-125* and a summary of this procedure follows.

Analytical reports comprise final results (uncorrected for blanks and recoveries unless specified), methods of analysis, levels of reporting, surrogate recovery data, and method blank data. In addition, special analytical problems will be noted in the case narratives. The number of significant figures reported are consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two (2) or three (3) significant figures. Data are normally reported in units commonly used for the analyses performed.

Concentrations in liquids are expressed in terms of weight per unit volume (e.g., milligrams per liter, mg/L). Concentrations in solid or semi-solid matrices are expressed in terms of weight per unit weight of sample (e.g., micrograms per kilograms, ug/kg). Reporting limits take into account all appropriate concentration, dilution, and/or extraction factors, unless otherwise specified by program requirements (e.g.,IRPMS reports).

A client report is generated with various steps of approval prior to printing of the final version. If any analytical anomalies were encountered during the analyses, e.g., an out-of-control matrix duplicate, it is documented in a case narrative. The case narrative is prepared by the respective operating unit, project manager, or other designated personnel and inserted in the final report.

The final report forms are printed, data packages are organized, a glossary of flags and acronyms is added, and reports are paginated.

5.9.1 General

The criteria described in Section 5.9.2 apply to all Project Reports that are generated under NELAC requirements. The criteria described in Section 5.9.3 and 5.9.4 apply to all Project Reports.

5.9.2 Project Report Content

- Title
- Laboratory name, address, telephone number, contact person
- Unique Laboratory Project Number
- Name and Address of Client
- Client Project Name (if applicable)
- Laboratory Sample Identification
- Client Sample Identification
- Matrix and/or Description of Sample
- Dates: Sample Receipt, Collection, Preparation and/or Analysis Date
- Definition of Data Qualifiers
- Reporting Units
- Test Methods
- Report Paginated

The following are required where applicable to the specific test method or matrix:

- Solid Samples: Indicate Dry or Wet Weight
- Whole Effluent Toxicity: Statistical package used
- If holding time $\leq$ 48 hours, Sample Collection, Preparation and/or Analysis Time
- Indication by flagging or narrative comment where results are reported below the quantitation limit.

5.9.3 Project Narrative

A Project Narrative and/or Cover Letter is included with each project report and, at a minimum, includes an explanation of any and all of the following occurrences:

- Listing of any subcontracted analyses and subcontractor location
- Non-conformances

- "Compromised" sample receipt (see Section 4.7.1)
- Method Deviations
- QC criteria failures
- Any authorized SOP deviations, non-conformances and QC failures must be covered in the case narrative, cover letter or within the report.

Project Release

The Project Manager or his designee authorizes the release of the project report with a signature.

Where amendments to project reports are required after issue, these are documented in the form of a DQR (refer to Section 4.8) and can be in the form of a separate document and/or electronic data deliverable resubmittal. The revised report is clearly identified as revised with the date of revision and the initials of the person making the revision. Specific pages of a project report may be revised using the above procedure with an accompanying cover letter indicating the page numbers of the project revised. The original version of the project report will be kept intact and the revisions and cover letter included in the project files.

5.9.4 Subcontractor Test Results

Subcontracted data are clearly identified as such, and the name, address, and telephone number for the laboratory performing the test is included in the project report. Subcontracted results from laboratories external to STL are not reported on STL report forms or STL letterhead. Test results from more than one STL facility are clearly identified with the name of the STL facility that performed the testing, address, and telephone number for that facility. Data from subcontractors' reports may be added to an STL electronic deliverable.

Data subcontracted within STL may be reported on the originating laboratory's report forms provided the following mandatory requirements are met:

- The name, address, and telephone number of the facility are provided.
- Analytical results produced by the STL intra-company subcontractor are clearly identified as being produced by the subcontractor facility.
- The intra-company subcontractor's original report, including the chain of custody is retained by the originating laboratory.
- Proof of certification is retained by the originating laboratory.
- All information as outlined in Section 5.9.2 is included in the final report where the report is required to be compliant with NELAC, for both the originating and subcontracting laboratory.

5.9.5 Electronic Data Deliverables

Electronic Data Deliverables (EDD) are routinely offered as part of STL's services. STL offers a variety of EDD formats. EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process in Section 4.4.1. Once the laboratory has committed to providing diskettes in a specific format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained as a QC record.

EDDs are subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory demonstrates that it can

routinely generate that EDD without errors. Any revisions to the EDD format are reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

5.9.6 Project Report Format

STL offers a wide range of project reporting formats, including EDDs, short report formats, and complete data deliverable packages modeled on the Contract Laboratory Protocol (CLP) guidelines. Regardless of the level of reporting, all projects undergo the levels of review as described in Section 5.3.6.

Appendix. List of Cited SOPs and Work Instructions

Cited Section No(s).	Status	Description	Document No.
1.1	Buff	Certification Listing	STLBuffCertList
1.6 5.7.1	Buff	Container Management: Process Operation/Bottle Order Set-Up	APM-Bottle Order-03
1.6 4.4.2	Buff	Project Management: Project Planning Process/Project Information Requirements	APM-ProjInfo-20
4.1.1 5.4.1	Buff	Capital Equipment Listing	STLBuffEquipList
4.1.2.9	Buff	Computer System Account and Naming Policy Computer System Password Policy Software Licensing Policy Virus Protection Policy	P-I-003 P-I-004 P-I-005 P-I-006
4.3.1.1 5.3.2	Buff	SOP Master Index	STLBuff_SOPIndex
4.3.2 4.12.3	Buff	Data Management: Record Storage and Retention	AGP-RecordStorage-56
4.4.2	Buff	Project Kick-Off Meetings	APM-ProjInfo-20
4.6	Buff	Procurement of Laboratory Supplies and Services	APH-Supply-08
4.6.1	STL	Testing Solvents and Acids	S-T-001
4.7.2	Buff	Client Confidentiality	APM-ProjInfo-20
4.8	Buff	Data Quality Request	AQA-DQR-65
4.8	Buff	Preventative or Corrective Action	AQA-CA-35
4.8 4.11	Buff	Job Exception Report (Non-conformance Report)	AQA-CA-35
4.11	Buff	Quality Systems Management Review	AQA-Management Review-45
4.11	Buff	Preventive Action Measures	AQA-CA-35
4.13	STL	Systems Audits	S-Q-002
5.1.2	Buff	Laboratory Personnel Training	AQA-Train-10
5.1.3	STL	Ethics Policy	P-L-006
5.3.1	Buff	Methods Capabilities & Index	ASR-Prsv-07
5.3.2	Buff	SOP Interim Change	AQA-SOP-55
5.3.5	STL	MDL Policy	S-Q-003
5.3.6.1	STL	Acceptable Manual Integration Practices	S-Q-004

Appendix. List of Cited SOPs and Work Instructions

Cited Section No(s).	Status	Description	Document No.
5.3.6.2	Buff	Data Review Checklists / Technical Data Review GC Extractables / HPLC GC Volatiles GC/MS: Volatiles and Semi-Volatiles Metals Wet Chemistry	AGP-DataReview-21
5.4.2	Buff	Instrument and Equipment Out-of-Service Tagging	AGP-OutofService-65
5.4.3	STL	Selection of Calibration Points	P-T-001
5.4.3	Buff	Standards Traceability and Preparation	AGP-STD-14
5.5.1	Buff	Balance Calibration, Care and Use	AGP-SuppEquip-02
5.5.1	Buff	Thermometer Calibrations	AGP-SuppEquip-02
5.5.1	Buff	Water Quality	AGP-SuppEquip-02
5.7.1	Buff	Sample Receipt: Handling and Processing	ASR-Receipt-05
5.7.1	Buff	Sample Container Preparation and Shipment	ASR-Bottle-03
5.7.5	Buff	Laboratory Waste Disposal Procedures	ASR-Disp-33 and AWM-Haz.Mg-01
5.8.5	Buff	Glassware Cleaning Procedures	AGP-Glass-04
5.9 5.9.6	Buff	Data Management: Reporting	ARP-Report-125

Appendix J
Microseeps Quality Systems Manual

(Document on CD-ROM)

MICROSEEPS, INC.

QUALITY SYSTEMS MANUAL

Controlled Copy No. _____

Signatures of Final Approval:

Ruth Welsh — 6-20-05

Ruth Welsh
Laboratory Manager
Date

Patrick McLoughlin — 6-20-05

Patrick McLoughlin Ph.D.
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1.0 Introduction, Purpose, and Scope

1.1 Introduction

Microseeps, Inc. (Microseeps) recognizes its crucial role in providing reliability and excellence in the environmental analytical industry. The laboratory provides information necessary for engineering, industrial, and regulatory clients to make informed judgments and applicable policy decisions. The laboratory's analytical services also assist clients in complying with major environmental regulations. Microseeps' management acknowledges that uncompromising dedication to quality is fundamental to remaining a competitive force in the analytical services market.

1.2 Purpose

The purpose of this Quality Systems Manual is to outline a program of policies, procedures, and documentation, which assures that our analytical services meet a defined standard of quality on an ongoing basis. This document defines the standards under which all laboratory operations will be performed. As supplemented by Standard Operating Procedures (SOPs), the Quality Systems Manual describes the laboratory's organization, objectives, and operating philosophy.

Microseeps Quality Systems Manual contains references to the laboratory's policies and operational procedures that have been established in order to meet the quality requirements of the NELAC Standards and the following Quality Policy Statement.

1.2.1 Microseeps' Quality Policy Statement

Microseeps is an organization that provides information and environmental services to a very diverse client base. In all processes from initial customer contact to project completion, Microseeps aim is to offer clients a reliable product of the best quality that is delivered in a timely manner at a reasonable price. This Policy Statement represents a top level management commitment and shall be achieved using the following goals:

1. Production of accurate information.
2. Adherence to an ethical standard that demands continuous honesty and integrity.
3. Achievement of customer satisfaction.
4. Establishment of a documented trail to support the results.
5. Maintenance of a pleasant working environment where employees are treated equitably and fairly.

These goals are fundamental to all of Microseeps' actions.

The Standard Operating Procedures and Microseeps Quality Systems Manual detail the procedures for achieving these goals. Management personnel are charged with ensuring all applicable procedures are completed in the spirit of the Quality Policy. They are available to all employees to assist in applying these principles and to advise appropriate action. It is the responsibility of each Microseeps employee to consistently act as directed by the five goals of quality. Further, it is their responsibility to consult their immediate superior for direction if an issue arises for which the response is unclear.

If Microseeps' Director Level Management realizes that these goals are not achievable but the validity and appropriateness of client results is not jeopardized, we will inform affected clients of the situation and allow them to alter their employ of Microseeps' services accordingly. If the client's data are to be negatively impacted where the validity of the results is in question, or a clients' project is operationally or legally jeopardized, the following procedures shall be followed:

1. Concerned operations will be suspended.

2. Affected clients will be notified.

3. Alternative procedures for securing the requested services will be employed until the corrective actions are completed to a level that is consistent with Microseeps' documented procedures and is satisfactory to management.

To achieve these quality goals, all laboratory data must be properly documented, legally defensible, and supported by statistically defined and verifiable confidence limits. Falsification of data under any circumstance is unacceptable and is grounds for termination. Microseeps has an Employee Handbook which provides policies and procedures to help employees avoid involvement in any activities that would diminish confidence in Microseeps competence, impartiality, judgment and/or operational integrity.

Microseeps uses EPA-approved methodologies such as those found in Standard Methods and SW-846, whenever methods are available. If an EPA-approved method has not been specified, Microseeps will select an industry recognized and validated method for use or will develop an internal method based upon thorough research and good scientific methods. In all instances of scientific innovation, Microseeps recognizes the value of a firm commitment to quality and integrity.

1.3 Scope of Quality Systems Manual

This document serves as both the Microseeps, Inc. Quality Systems Manual and the Microseeps Laboratory Quality Assurance Plan. It contains both quality assurance policies and quality control procedures that are followed to ensure and document the quality of analytical data. This manual provides detail concerning quality management requirements employed at Microseeps

for the documented acquisition of samples, analysis of those samples via specific tests, the reporting of that data to the client, and the ultimate disposal of samples.

1.4 Scope of Services

Microseeps offers a comprehensive scope of laboratory analytical services and field sampling activities to environmental consultants, industries, governmental agencies, and municipalities. The scope of services include:

Wastewater, storm water, solid waste, and hazardous waste analyses

- Metals analyses via ICP or Cold Vapor Atomic Absorption
- Semivolatile analysis via:

  GC/MS

- Volatile analyses via:

  GC/MS
  GC PID
  GC ELCD
  GC ECD

- Ion analysis via Ion Chromatography
- Wet Chemistry analyses of 32 different analytes via a range of wet chemistry techniques.

Intrinsic Bioremediation Analysis

Mobile Analytical Services

Field Sampling Services

- Hand Sampling for soil gas
- Hand Sampling for Natural Attenuation
- Direct Push sampling for Natural Attenuation, soil gas, or compliance analysis.

Training Seminars

Soil Vapor Extraction Analysis

1.5 Certifications

Microseeps holds the following certifications:

- National Environmental Laboratory Accreditation Program (NELAP): Pennsylvania
- Florida
- Connecticut
- West Virginia
- South Carolina
- Louisiana
- New York
- New Jersey
- Kentucky

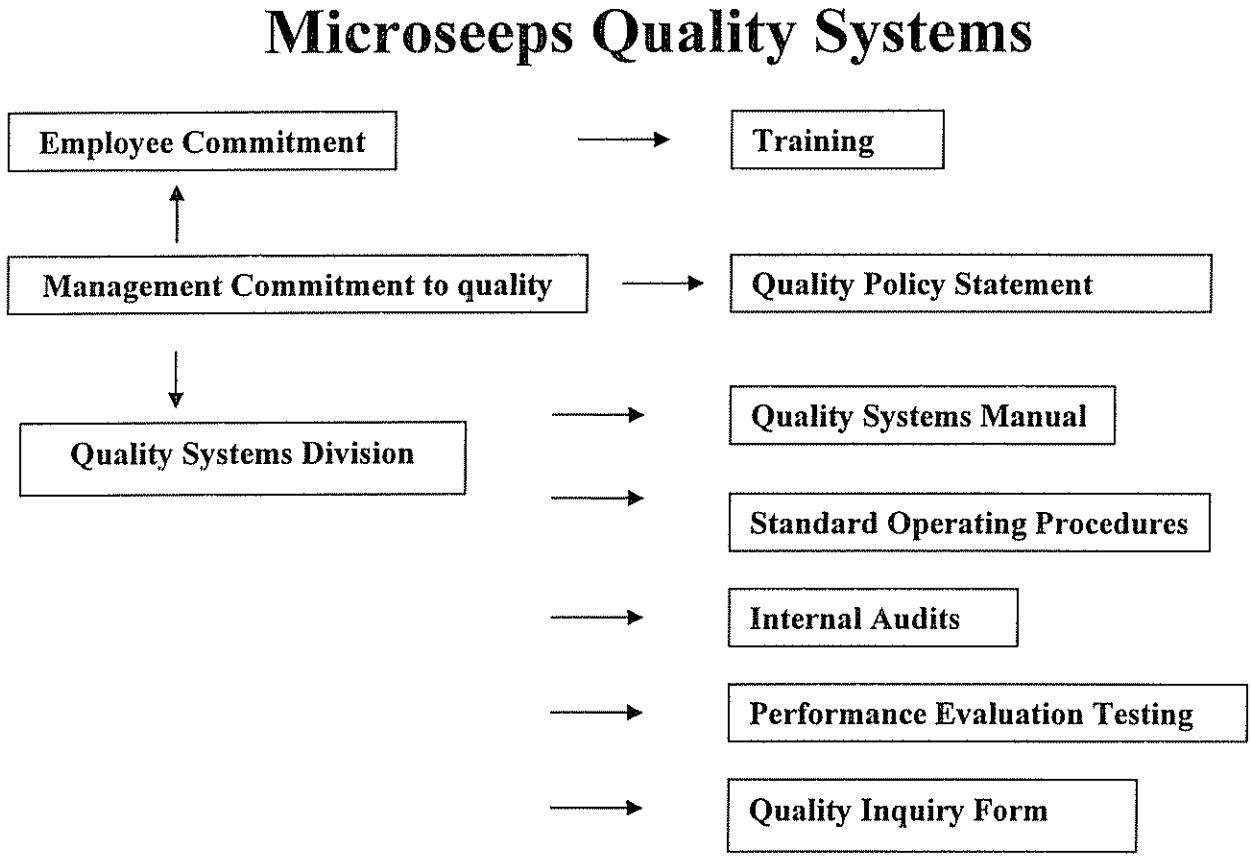
Specific parameter lists for the various certifications are available from the Customer Service Department upon request.

2.0 Quality Systems – Establishment and Audits

2.1 Establishment of a Quality System

Microseeps has established a quality system based upon the fundamentals of good laboratory practices and the requirements outlined in Chapter Five of the National Environmental Laboratory Accreditation Program. Although Microseeps provides a variety of environmental services, Microseeps' Quality System has been designed according to the type, range, and volume of analytical testing activities undertaken in the laboratories. This section describes Microseeps' Quality System and outlines the policy and procedures for implementing corrective action when non-conforming work or departures from policies and procedures occur.

2.2 Elements of Microseeps Quality System



This flowchart shows the general elements of Microseeps Quality Systems. Each area of this flowchart is addressed or referenced in this Quality Systems Manual.

2.2.1 Quality Manual Review

The Quality Systems Division reviews Microseeps Quality Systems Manual annually for accuracy and applicability. These records are maintained in the Quality Systems Coordinator's Office. All revisions will be conducted in accordance with Microseeps Standard Operating Procedure for Document Control.

2.2.1.1 Document Control

Document control procedures are specified in Microseeps Standard Operating Procedure for Document Control SOP-ADM 5. The Standard Operating Procedure outlines document control procedures for generating, formatting, revising, approving, tracking, distributing, indexing, archiving, and destroying controlled documents, including the Quality Systems Manual.

2.2.2 Internal Audits

Technical audits serve to verify compliance with method-specific procedures including operations related to test methods. Any audit that is conducted on an operation that is involved with data generation and the assurance of its quality is a technical audit. System audits function to verify compliance with the laboratory's quality system. Types of procedures that would be reviewed as a part of a systems audit could include: (1) response to complaints; (2) sample tracking methodologies; and (3) sample acceptance policies.

The Quality Systems Department will conduct all technical and system audits. Audits will be conducted under the direction of the Technical Director and shall be conducted by individuals who are independent of the activity to be audited. All internal auditors shall be trained and qualified in the areas in which they will be conducting the audit.

2.2.2.1 Audit Frequency

Internal Audits are scheduled and conducted by the Quality Systems Division and may either be scheduled or unannounced. Microseeps conducts internal technical audits and internal system audits at least monthly. The audits are conducted to insure that Microseeps' operations continue to comply with the Quality Systems requirements specified in the Quality Systems Manual and Standard Operating Procedures. The Quality Systems Coordinator maintains an audit schedule to ensure that all laboratory groups and programs are audited.

2.2.2.2 Procedures for Internal Audits

Audits will be conducted on analytical groups or systems according to the annual audit schedule posted in the Quality Systems Coordinator's Office. Immediately prior to starting the audit the Laboratory Director will be notified. Microseeps' Standard Operating Procedure ADM-4 for Internal Laboratory Audits outlines the specific procedures for conducting an internal audit.

According to the SOP, the audit is performed using the appropriate audit check sheet as a guide. The audit check sheet will be selected and used as the auditing guide for the area that is to be audited. The audit check sheets delineate the activities and records that will be reviewed. The sheet is completed and additional notes are made by the auditor based upon observations, interviews, and record reviews.

2.2.2.3 Internal Audit Findings and Corrective Action

Using the audit check sheets and supplementary notes, an Internal Audit Report (Figure 2-1) is prepared by the Quality Systems Coordinator. The Internal Audit Report contains an overall summary of the audit, including both items of a positive nature, as well as deficiencies. The cause of each deficiency is determined using Root Cause Analysis. Root Cause Analysis is the foundation upon which all Corrective Action is based. This process and an example are outlined in Figure 2-2 in this Quality Systems Manual.

Corrective Action based upon the root cause analysis is indicated on a Corrective Action Report (see Figure 2-3). A Corrective Action Report is prepared for each deficiency listed on the Internal Audit Report. The Internal Audit Report and Corrective Action Report are forwarded to the appropriate Department Head for corrective action. A follow-up audit is conducted.

If the audit was of a technical nature, the Audit Report will be forwarded to the Laboratory Director. The Laboratory Director and/or the Assistant Laboratory Director will meet with the Analytical Division the first business day following the audit. The audit will be discussed along with the recommendations for corrective action. Corrective action is expected to take place immediately or as soon as possible following the audit. A follow-up audit of any deficient area(s) will be conducted within one week of audit completion, or as soon as corrective action is completed in order to monitor the effectiveness of corrective action.

Where any audit findings or defective measuring or test equipment may cast doubt on the correctness or validity of the laboratory's calibrations or test results, Microseeps shall take immediate corrective action as specified in Microseeps' Quality Policy Statement. If the subsequent investigation shows that laboratory results have been affected, the affected client shall be notified in writing by the Customer Service Office.

2.2.2.4 Work Stoppage

The Technical Director has the authority to stop any work that is found to be unsatisfactory or to prevent reporting of unjustifiable results. The work will not be ordered to commence until corrective action is taken that insures satisfactory work and reliable results according to the Technical Director's discretion.

2.2.3 Performance Evaluation Audits

Performance evaluation audits enable Microseeps to measure the precision, accuracy, and comparability of laboratory-generated data through the use of blind reference materials. Microseeps participates in performance evaluation studies required by the EPA and several state agencies. Performance evaluation studies are completed as required to maintain necessary certifications, and as required as an integral part of an internal audit at the Technical Director's discretion.

Performance evaluation studies for all accredited parameters are planned for testing twice each year. The Quality Systems office maintains EXCEL spreadsheets of results so that trends are easily noticeable.

In situations where the analyst is to know that a performance evaluation is being conducted, the analyst prepares the samples according to the instructions provided. If the PE is to be a blind audit, the Quality Systems Department prepares the samples. The samples are analyzed as soon as possible after opening the vials to avoid sample deterioration. Prior to reporting the results to the appropriate agency, the Laboratory Director and the Quality Systems Manager evaluate results of the performance evaluation samples.

2.2.3.1 Performance Evaluation Findings and Corrective Action

Once the evaluation report from the Performance Evaluation Provider or appropriate agency is received, the Quality Systems Coordinator forwards the findings to the Technical Director. The Laboratory Director is informed of the report and unacceptable results are investigated to determine the root cause of the failures. The following points are addressed during the investigation:

- Potential for reporting/calculation errors
- Preparation of calibration standards
- Evaluation of quality control data associated with the analysis
- Evaluation of analytical technique and instrument performance

Additional Performance Evaluation samples may be submitted for analysis if the Technical Director and Quality Systems Manager determine it is necessary to ensure that an analytical method, technique, or instrument performance problem is corrected.

2.2.4 External Audits

External Audits are conducted as necessary to retain laboratory certifications or at a client's request. The Technical Director is the liaison between Microseeps and an external auditor. The Technical Director is responsible for notifying the laboratory staff of upcoming audits. This notification will include the agency that will perform the audit, the reason for the audit, the dates involved, and the areas of concern.

During the audit, laboratory personnel will be available to the auditor as requested, as will any documentation necessary for the auditor to obtain sufficient information to effectively evaluate the laboratory. If laboratory information of a proprietary nature is necessary to complete an audit, the auditor will be required to complete and sign Microseeps Confidentiality Agreement.

2.2.4.1 External Audit Findings and Corrective Action

The Technical Director will prepare a report summarizing the findings of the post audit meeting. Following review of this report, any deficiencies noted by the auditor that affect the validity of data in any area will be addressed immediately. Corrective action will be taken to eliminate the cause of a deficiency and to prevent recurrence. The Corrective Action process shall identify and implement corrective actions to eliminate the root cause of the deficiency. The development and implementation of a corrective action plan shall not be contingent upon the receipt of the external auditor's report.

When the External Audit Report is received at Microseeps, deficiencies will be addressed in the order of their priority as determined by the auditor. The Technical Director will prepare a response plan for correcting the reported deficiencies and will submit a Corrective Action Plan to the auditing agency. Corrective action will be taken according to the timetable outlined by the Technical Director.

2.2.5 Managerial Review

Laboratory Management shall conduct a review of the Quality System and its testing and calibration activities. This review shall be conducted to ensure the Quality System's suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. This review shall be conducted annually by department level management or higher.

The review will be conducted in the same manner as an Internal Systems Audit, but the auditors will be made up of department level management. Microseeps' Standard Operating Procedure ADM-4 includes an Audit Check Sheet for the Managerial Review Process, which outlines the review procedure.

2.2.5.1 Managerial Review Findings and Corrective Action

The review findings shall be documented on an Audit Report Form and submitted to the Technical Director for review. The Technical Director shall review the Audit Report with the Quality Systems Department. Corrective Action Reports will be generated for the audit deficiencies and will be resolved as necessary. A timeline for resolution is specified on the Corrective Action Report and the review findings become an item for discussion at the monthly Managers meeting where management ensures the corrective actions are completed.

2.2.5.2 Manager Meetings

Regular discussions of quality assurance issues are necessary to provide a forum in which upper management are informed of problems and changes that affect the laboratory operations. The Technical Director meets with the President, Senior Vice President Director of Operations, Laboratory Director, and Director of Sales, Customer Service, and Field Services each month to discuss quality issues. These meetings serve as a general review of factors affecting quality and will include the following topics at a minimum:

- Personnel changes
- Instrument changes
- Internal and External Audit findings
- Certification changes
- Testing and calibration activities
- Quality System implementation activities and progress

2.2.5.3 Documentation

At the end of the meeting, the Technical Director will document the results of the meeting, prepare a summary of the discussion, and set an agenda for additional follow-up, if required. This report shall serve as documentation of management commitment to the Quality System. A report of each meeting will be kept on file in the Quality System Coordinator's Office.

2.3 Additional Quality Control Checks

In addition to periodic audits, Microseeps ensures the quality of results provided to clients by implementing additional checks to monitor the quality of the laboratory's analytical activities. Some of these checks are as follows:

- Use of certified standards in many of our quality control samples
- Replicate testing using the same and different test methods
- Correlation of results for different but related analysis of a sample
- Participation in Proficiency Testing

2.4 Audit and Review Documentation

All audit records shall be kept on file in the Quality Systems Coordinator's Office. These records shall be available for external auditors, as well as, individuals involved in a Managerial Review of the Microseeps Quality System. This documentation will include:

- Audit Check Sheets
- Audit Report Forms
- Corrective Action Reports

2.5 Quality Inquiry Forms

Microseeps has instituted a procedure for reporting when departures from documented policies, procedures, and quality control have occurred. The Quality Inquiry Form QIF (see Figure 2-4) is a means for anyone in the company to communicate quality concerns to the Technical Department. These forms are a unique part of Microseeps' Quality System, which provide an avenue for all employees to reflect a genuine concern for quality throughout the company. The QIF is also used to document Customer Complaints and initiate the process of resolving those complaints. All items pertaining to the Customer complaint shall be recorded on the QIF including investigation results and resolution to the satisfaction of the client. All QIFs that deal with Customer Complaints shall be kept on file in the Customer Service Office.

Note: If the quality concern can be corrected by the person who discovered the quality problem, a CAR should be completed in lieu of a QIF.

2.6 Corrective Action

An integral part of Microseeps' Quality Systems Program is the system for identifying, reporting, and correcting deficiencies in the laboratory operation. There are several areas in the laboratory that may require corrective action. It is the responsibility of every employee to be aware of potential problems and to notify the appropriate personnel of situations requiring corrective action.

2.6.1 Problem Isolation and Identification

Identification and isolation of problems in the laboratory are not always easy tasks. The need to perform corrective action may become apparent at any point of the analytical process. Corrective action should be initiated and documented as soon as a problem becomes evident. The analyst at the bench detects some situations such as malfunctioning equipment. Corrective action for these situations takes the form of repairing the instrument, either internally or through the use of a service call. The corrective action is documented in the instrument maintenance log and the data obtained just prior to the failure is closely scrutinized for acceptability.

Other situations may not be easily identifiable. For example, systematic drift or sensitivity fluctuations may not be identified until the time that data is validated. Other occurrences that may trigger the need for corrective action include the following:

- Recoveries for surrogates, matrix spike, matrix spike duplicates, and laboratory control standards outside of acceptance limits.
- Percent differences for duplicate analyses outside acceptance limits.
- Trends noted in quality control data.

Out of control events that concern sample analysis and data generation must be documented in a Case Narrative, which becomes a permanent part of the client's project file and final data report.

2.6.2 Sample Handling Problems

Problems involving sample handling may include missing or broken containers, discrepancies between the chain of custody and actual shipment, improperly preserved bottles, insufficient volume, and missed holding times. When one of these problems is identified, a Non-Conformance Form is completed and acted upon in accordance with the Standard Operating Procedure for Sample Receiving (SOP-S2). If necessary, the client is contacted to discuss possible resolutions to the problem. The Non-Conformance Form becomes a part of the client's permanent file.

2.6.3 Sample Analysis Problems

Problems incurred during sample analysis may bring procedures and data into question. These problems may include the following:

- Unacceptable calibration
- Improper procedures
- Unacceptable blank, LCS, and/or surrogate recovery
- Quantitation error
- Required QC not performed
- Retention time shifts

Resolving these problems may include preparing and analyzing the samples a second time, re-calibrating the instrument, or making new standard solutions and reagents. Standard Operating procedures detail corrective action steps that are specific to an analytical procedure. The documentation becomes part of the client's permanent file.

The person identifying the problem documents the situation and identifies possible sources of the problem. If this individual can immediately correct the situation, for example, by re-calibrating the instrument, they will do so and document the action that was taken in the case narrative. If the problem cannot be corrected immediately, the person documents the situation and notifies the Laboratory Director. The Laboratory Director is responsible for ensuring that the appropriate corrective actions are followed.

2.6.4 Corrective Action Reports

A Corrective Action Report (CAR) will be completed for each audit deficiency, legitimate issue reported on a Quality Inquiry Form, and any systemic out of control event that occurs. The Quality Systems Coordinator is responsible for maintaining a supply of forms. To track the corrective action, each CAR will be given a unique identifier in the form of YY-XXXX where:

- YY = the month in which the CAR was initiated and
- XXXX = is the sequential number of the record starting with 0001

2.6.4.1 Corrective Action Report Initiation and Procedures

A Corrective Action Report may be generated by anyone in the company who discovers and can correct a systemic out of control event. There are two types of situations in which an employee will generate a CAR. First, when the root cause is obvious to the person who discovered the out of control event, and secondly when the root cause is not obvious.

In the first situation, the person who discovers an out of control event shall institute and document corrective action by completing the top portion of the CAR (above the dotted line). They will enter their name, the current date, a description of the event that needs corrective action, and the specific corrective action that was taken. The Lead Analyst or Department supervisor shall be notified of the event and the corrective action necessary to solve the non-conformance. The person who completes the corrective action shall sign the form and forward it to their immediate supervisor for a signature. The form will then be forwarded to the Quality Systems Department for cataloging. If additional action is required, the form shall be returned to the department concerned and returned to the Quality Department when corrective action is complete.

If the root cause is not immediately obvious, the Quality Systems Department shall be notified immediately to assist in Root Cause Analysis and determining the appropriate corrective action.

The Quality Systems Department will forward a copy of the CAR to the Laboratory Director. If it is determined that the client needs to be notified of the incident, the Quality Systems Department shall forward a copy of the CAR to the Customer Service Department for further action.

2.6.4.2 External Audits and Performance Evaluation Studies

The Technical Department will initiate CAR(s) in response to third party audits and deficiencies on performance evaluation studies. In the case of third party audits, if the resolution to the finding is easily identifiable, the Technical Director will discuss the situation with the Laboratory Director and the corrective action will be implemented. If the finding concerns a procedural change or is interdisciplinary, the Technical Director will form a task team to investigate the problem and develop an appropriate course of action. The task team's findings will be presented to the Technical Director for discussion and possible implementation. Once a corrective action plan is implemented, the situation will be monitored for a reasonable period of time to ensure that the action has been effective.

The Technical Director will prepare a report for management on a monthly basis that summarizes all corrective actions (See Figure 2-5). The report will provide a brief description of the problem, the steps that are being taken to correct the situation, and the status of the item.

2.7 Preventive Actions

The preferred course of laboratory quality and improvement is to identify opportunities for improvement rather than react to the occurrence of problems or complaints. Microseeps is

continually seeking ways to improve its performance and product. When these areas are identified, a plan is developed by the staff, usually the Technical Director, Laboratory Manager, or Manager of Technical Systems. Preventative actions are implemented according to the time table specified in the plan and monitored at weekly Operations Meetings. Preventive action procedures include follow-up actions and applications of controls in order to ensure effectiveness.

2.8 Management Arrangements for Permitting Departures from Documented Procedures or Standard Specifications

It is Microseeps management's intent to ensure that documented procedures are followed. Rarely, a situation may occur that requires a departure from documented quality procedures. When this type of situation occurs, the Technical Director and Laboratory Director, and any other manager whose department may be affected, will discuss and unanimously agree upon the action to be taken. The departure will be documented in memo form and kept on file in the Quality System Coordinator's Office. Corrective action will be taken as soon as possible to prevent the necessity of the departure from reoccurring.

Figure 2-1

Internal Audit Report

Audit Date: _____ Department: _____

Division: _____ Auditors: _____

Lead Analyst/Supervisor: _____

Employees Present: _____

_____

Audit Summary:

Overall Rating (circle one): **Excellent Good Fair Poor**

Areas of Excellence:

Deficiencies:

(Listed by sequential numbers)

Technical Director Signature: _____ Date: _____

Figure 2-2

Root Cause Analysis

Deficiencies are always analyzed from the non-conformance back to the root cause as follows:

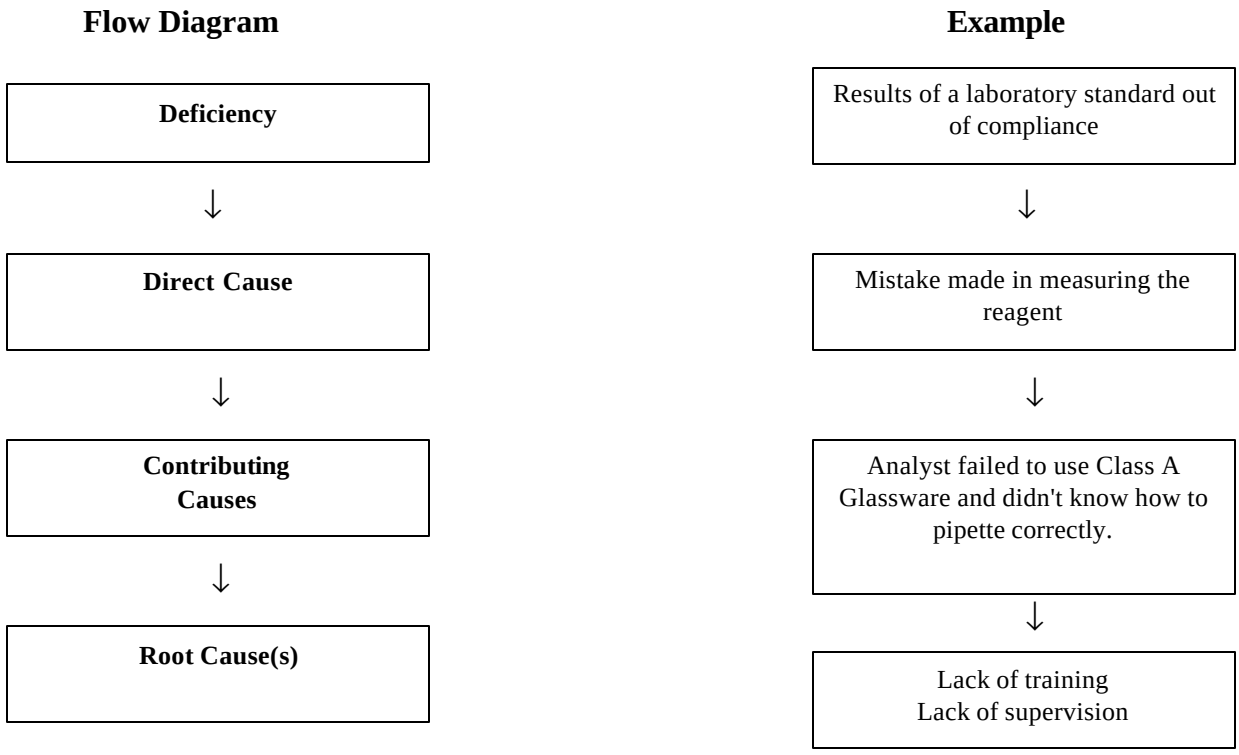


Figure 2-3

Corrective Action Report

This form is to be initiated by the person who discovers an event that needs corrective action. Please complete only the portion of the form above the dotted line. When complete, forward to the Quality Systems Department.

Initiated by: _____ **Date:** _____
Print Name

Description of the event needing corrective action and corrective action required.

Root Cause: _____

This Corrective Action must be acted upon in the time frame indicated below:

Immediately ___ One Week ___ Two Weeks ___ Other _____
Est. Date

Corrective Action Taken: **Due Date:**

Action Completed by: _____ **Date:** _____

Supervisor Review: _____ **Date:** _____

Project #: _____ **Sample Numbers:** _____

Additional Action: is___ is not ___ necessary. Customer Contact: is___ is not ___ necessary.

Action Closed: _____ **Date:** _____

cc: Laboratory Director, Technical Director **CAR #** _____

Figure 2-4

Quality Inquiry Form

This form is to be used as a means to communicate quality issues within the company and as a means of documenting customer complaints. The first resource to resolving any internal concern is direct communication. This form is provided as a means to pass inquiries to the Technical Department when direct communication is inappropriate or ineffective. Please fill out as completely as possible and forward to the Technical Director.

Date of inquiry _____

Concerned Department:	Customer Service	Organics	Bottle Preparation	Risk Vapors
	Metals	Extractions	Wet Chemistry	Risk Waters
	General	Management	Quality Systems	Receiving

Please categorize the issue (Please circle one of the following numbered items):

Procedure related

1) Failure to follow SOP
2) Improper SOP
3) No SOP known

Record keeping related

4) Items not or improperly recorded
5) Records improperly stored
6) Records improperly handled (lost, insecure, removed from building)

Client Communication related

7) Client was knowingly promised the unachievable
8) Client's request was disregarded
9) Information was inappropriately kept from client
10) Report was incomplete or inaccurate
11) Other client complaint (describe below)

Internal Communication related

12) Internal chain of command (as per organizational chart) not followed
13) Client's request not passed to appropriate personnel
14) Supervisor was unresponsive
15) Redundant efforts were caused by poor communication among analysts

Please describe the issue and means taken to resolve (use other side if necessary):

_____

_____

_____

_____

Figure 2-5

Quality Systems Update and
Corrective Action Summary Report
To Management

1. Brief Description of audit deficiencies and quality inquiry forms:

CAR #	Date	Deficiency	Resolution	Update

2. Steps being taken to remedy the deficiency: (Obtain information from other Management Team members who have unresolved CAR's and update them on status of Technical Directors'.)

3. Other items for discussion as they pertain to quality:

- **Personnel changes**
- **Instrument changes**
- **Upcoming Audits**
- **Certifications**
- **Quality System implementation activities and progress**

file:C\My Documents\Forms\Corrective Action Summary Report

3.0 Organization and Management

Microseeps is a small business as defined by Standard Industrial Classification 8734. Since Microseeps is a business that must compete in a marketplace characterized by merger and consolidation, it is even more important to have an efficient organization with well-defined responsibilities. The purpose of this section is to describe the organization and management structure of Microseeps, interdepartmental and intradepartmental relationships, and to detail employee responsibilities and qualifications.

3.1 Microseeps Organization

Organizations must have a framework within which to achieve their objectives. Organizational structure determines the configuration of positions, duties, and channels through which performance is controlled and authority is delegated. It is crucial that a company use a structure that enables it to realize competitive advantages.

In order for Microseeps to achieve its stated objectives for the quality of its analytical services, all employees must be able to function within a structure that provides an emphasis on quality from a technical standpoint. A functional organizational structure is specifically suited to this purpose.

3.1.1 Organizational Structure

Microseeps' organizational structure is functional. This type of structure is centralized, which restricts decision-making authority to higher levels of management. Company organization is departmental and is specialized and arranged according to function. Because of the nature of the individual departments, some authority is delegated to individuals who lead smaller groups within the larger departments.

The Quality Systems Department Manager reports directly to the President and CEO. Sales, Finance, and other administrative departments are overseen by the Board of Directors. Microseeps' Organizational Chart is depicted in Figure 3-1.

3.1.2 Interdepartmental Relationships

Responsibility and dedication to quality laboratory practices and procedures begin at the highest level of management. It is the duty of the President to assure that the framework is in place to provide quality systems guidelines. It is the duty of Microseeps managers to implement the policies and procedures and to see that they drive the activities of the laboratory.

3.1.2.1 Operations Department

The Vice President of Operations oversees the following departments: 1) Operations; 2) Field Services; 3) Technical Services; 4) Customer Service; and 5) Information Technology. The Laboratory Manager oversees the analytical work groups in the laboratory. The Manager of

Customer Service oversees all of the Customer Service functions. The Manager of Field Services oversees all aspects of field sampling and the Manager of Technical Services is responsible for equipment maintenance and regulatory method development.

3.1.2.2 Quality Systems Department

The Quality Systems Department operates outside of the scope of the Operations Department in lines of authority. Responsibility for the Quality Systems Department falls upon the Quality Systems Manager who reports directly to the President. The Quality Systems Manager is responsible for the following work:

1) Quality Systems
2) Research and Development

The Quality Systems Coordinator is administratively responsible for quality assurance and quality control, and may serve as the Quality Systems Manager's representative in his absence.

3.1.3 Intradepartmental Relationships

Managers' Meetings are held monthly between the department heads, and senior level management. This meeting is the forum in which company operations are discussed, problems are resolved, and departments communicate with one another.

The Quality Systems department has a quality assurance and quality control role to fulfill within each department at Microseeps. Discrepancies in quality in any department will be relayed from the Quality Systems Manager to the appropriate Department Head using a format that is appropriate for the type of discrepancy and recommending an appropriate corrective action. This may take the form of using an electronic “Help Desk” forum, using a Corrective Action Form, or taking action from a determination made during a Manager’s Meeting.

3.2 Personnel Qualifications

All of Microseeps employees are responsible for complying with the applicable job specific quality assurance and quality control requirements. All staff are to familiarize themselves with the quality documentation and implement the policies and procedures contained in this Manual in their work. Each staff member, including contracted and additional technical and key support personnel should they be required, must demonstrate a combination of formal education and experience to satisfactorily perform their particular function, as well as, a general knowledge of laboratory operation, quality assurance, quality control, test methods, and records management. All personnel are placed in a work group or department with adequate supervision that ensures the employee works in accordance with the laboratory’s Quality System. Documentation of employee proficiency for specific job functions and test methods is maintained in the Quality Systems Coordinator's Office.

The Laboratory Manager, based upon specific educational and experience requirements, makes laboratory job assignments. Laboratory analysts are assigned a job classification according to the level of formal education and related laboratory experience they possess.

Basic duties of key staff (those included in Figure 3-1) are discussed below. Complete job descriptions for key staff are contained in Appendix A of this Quality Systems Manual. Complete documentation of all employee job descriptions is kept on file in Employee Personnel Files in the Senior Vice President's Office.

3.2.1 President and Chief Executive Officer

The President and Chief Executive Officer is responsible for overall company performance. He is responsible for new business development and the financial integrity of the company. The President and Chief Executive Officer represents Microseeps at trade shows and environmental conferences.

3.2.2 Senior Vice President

The Senior Vice President works closely with the President to provide overall direction for the laboratory operation. He is responsible for identifying potential new markets and shares the responsibility for the financial integrity of the laboratory with the President. Additionally, he approves capital expenditures and evaluates current market conditions to maintain the laboratory's competitiveness.

3.2.3 Vice President of Sales and Marketing

The Vice President of Sales and Marketing is responsible for meeting target sales goals, overseeing sales staff, and assists the President and Senior Vice President in developing sales and marketing strategies.

3.2.4 Vice President of Operations

The Vice President of Operations is responsible for implementing and enforcing laboratory policies and procedures. The Vice President of Operations delegates responsibility to the Department Heads, is responsible for the daily operation of each department of the laboratory, except Quality Systems, and is responsible for overseeing the routine expenditures and for maintaining the laboratory's budget.

3.2.5 Laboratory Manager

The Laboratory Manager is responsible for ensuring that Microseeps' goals of providing accurate and verifiable analyses are met. It is the Laboratory Manager's responsibility to ensure that all Operation's Department personnel have the required qualifications and training for their positions. Once qualified personnel are in place, the Laboratory Manager, in conjunction with the Quality

Department, will be responsible for assuring that all employees are thoroughly familiar with the Quality Systems Manual and accepted laboratory practices. The Laboratory Manager oversees the daily management of the laboratory staff. A major component of this particular responsibility is the integrity of laboratory reports. The Laboratory Manager's qualified designee will review and approve all outgoing reports. The Laboratory Manager reports to the Vice President of Operations. In the absence of the Laboratory Manager, the assistant Laboratory Manager will assume these responsibilities.

3.2.6 Quality Systems Manager

In laboratory documents, the title Technical Director is used synonymously with Quality Systems Manager. The Quality Systems Manager is ultimately responsible for ensuring that the data produced by the laboratory are technically sound and of the highest quality possible. The Quality Systems Manager serves as the focal point for quality assurance and quality control and is responsible for the oversight and/or review of quality control data. This position notifies laboratory management of deficiencies in the quality system and monitors corrective action. The Quality Systems Manager reports directly to the President.

3.2.7 Manager of Technical Systems

The Manager of Technical Systems ensures that technical problems within the laboratory are resolved. This position is also responsible for ensuring instrument repairs are conducted in a timely and cost effective manner. The Manager of Technical Systems implements plans for method development and assists clients in data interpretation. The Manager of Technical Systems reports to the Vice President of Operations.

3.2.8 Manager of Customer Service

The Manager of Customer Service provides direction and supervision to ensure that clients receive the best service possible. This position reviews proposal submittals and proposes pricing strategies for potential projects. This position reports to the Vice President of Operations.

3.2.9 Manager of Field Operations

The Manager of Field Operations provides laboratory and field support and direction for the Field Services Operations of Microseeps. This position reports to the Vice President of Operations.

3.2.10 Manager of Information Technology

The Manager of Information Technology (LIMS Administrator) has overall responsibility for the Information Systems in the Laboratories and Administrative Offices. This position troubleshoots systems to ascertain where problems may be occurring and either repairs them or coordinates and schedules repairs with a vendor. The Manager of Information Technology makes software and hardware recommendations that will best serve the company and arranging for purchasing and

ordering information systems equipment. This position reports to the Vice President of Operations.

3.2.11 Quality Systems Coordinator

The Quality Systems Coordinator Assists the Quality Systems Manager to ensure that Microseeps Quality Systems Policies and Procedures are being followed. This is accomplished by: (1) Reviewing data validation procedures; (2) Alerting the analysts should the need for corrective action exist; (3) Performing internal audits; (4) Establishing a periodic schedule for analyzing performance evaluation samples; and (5) Maintaining Quality Control records. The Quality Systems Coordinator reports to the Quality Systems Manager.

3.2.12 Assistant Laboratory Manager

The Assistant Laboratory Manager assists the Laboratory Manager in reviewing the work performed in the laboratories, in scheduling work within the laboratory, and overseeing data collection for final reports. The Assistant Laboratory Manager also interfaces with Project Managers to ensure that projects requiring special consideration are completed according to specification. This position is responsible for notifying the Project Manager of any situation that may negatively impact a client project so that the client may be notified. The Assistant Laboratory Manager reports to the Laboratory Manager.

3.2.13 Procurement Coordinator

The Procurement Coordinator (Sample Custodian) is responsible for properly receiving and logging-in all samples received at Microseeps and ensuring that storage and documentation requirements are met. This position also unpacks and marks the samples with the correct internal laboratory identification number so that the client's samples can be tracked through the laboratory. The Procurement Coordinator is responsible for documenting all discrepancies between samples received and accompanying chains of custody and for notifying customer service so that the client may be contacted. This position also has the responsibility for ordering laboratory supplies and maintaining packing slip/receipt documentation. The Sample Custodian reports to the Laboratory Manager.

3.2.14 Lead Analyst

The Lead Analyst assists in the daily supervision and training of staff analysts and technicians. Lead Analysts ensure that safety policies are followed and that any unsafe conditions are reported to the Company Health and Safety Officer. The Lead Analyst performs a broad variety of duties within a specific group or division. The Lead Analyst ensures that analysts strictly adhere to procedures outlined in the Quality Systems Manual and Microseeps Standard Operating Procedures. The Lead Analysts report directly to the Laboratory Manager.

3.2.15 Laboratory Analyst

Laboratory Analysts are responsible for retrieving samples from Sample Receiving at the direction of the Lead Analyst and observing all internal custody requirements. The analysts shall ensure that aliquots analyzed are representative of the entire sample. All analyses shall be conducted according to Microseeps Standard Operating Procedures. Responsibilities also include following good laboratory practices in carrying out duties assigned, and complying with all safety regulations applicable to their respective laboratories. Analysts report directly to their Lead Analyst.

3.2.16 Project Manager/Customer Service Representative

The Project Manager (Customer Service Representative) is responsible for overseeing in-house analytical projects. It is the Project Manager's responsibility to accurately communicate project requirements to the Laboratory Manager so that projects are logged-in, analyzed, and reported in the format, timeframe, and within the project-specific protocols required by the client. This position reports to the Manager of Customer Service.

3.2.17 Field Services

Field Services personnel are responsible for direct push and other types of sampling services. It is their responsibility to represent Microseeps to the client, collect valid samples, document activities appropriately, order and inventory supplies for field projects. The Field Services Personnel report directly to the Manager of Field Operations.

3.2.18 Bottle Preparation

The Bottle Preparation Technician is responsible for accurately preparing sample containers in a timely and safe manner according to client specification. The Bottle Preparation person also maintains records of standing orders and ensures they are prepared for the courier and for shipment when appropriate. This position also prepares purchase orders for ordering supplies as needed. The Bottle Preparation person also maintains the Bottle Preparation room and storage area in a neat and orderly manner. This position reports to the Manager of Customer Service.

3.3 Contract Review, Design Control, and Quality Planning

The Customer Service Department shall use the flow diagram shown in Figure 3-2 as an initial review of all new work that comes in to the laboratory. A project manager shall be assigned to the project if so designated by the flow diagram. When the contract or scope of work specifies two or more of the requests on the left side of the flow diagram, the assigned project manager shall initiate a Contract Review Checklist (see Figure 3-3) and forward it to the departments concerned.

The contract and/or scope of work shall be reviewed by each department concerned to determine if Microseeps has adequate facilities and resources to complete the work in the contract appointed time frame. Each department is to indicate on the form whether their department can or cannot

meet the contract requirements, initial and date the form, and return it to the Project Manager within 24 hours of receipt of the checklist. If a department cannot meet the requirements of the contract or scope of work, the department head is to indicate the reason(s) for that decision on the back of the Checklist prior to returning it to the Project Manager.

The client shall be informed and a resolution discussed if the laboratory review of capability indicates any potential conflict, deficiency, lack of accreditation status, or inability on the laboratory's part to complete the clients work. Any differences shall be resolved to the satisfaction of the laboratory and the client prior to commencement of work.

All records of Contract Reviews, including any significant changes, are maintained in the Customer Service Contract files. Customer service also maintains records of discussions with the client relating to the client's requirements and the results of the work during the period of the execution of the contract. Correspondence with the client pertaining to laboratory activities for a project are maintained in the Client Project File. The client shall be notified of any deviation from the contract, including accreditation changes that affect the contract.

In the event that the contract needs amended after work has commenced, the same contract review procedures shall be repeated and any changes shall be communicated to all affected personnel.

Figure 3-1
Microseeps' Organizational Chart

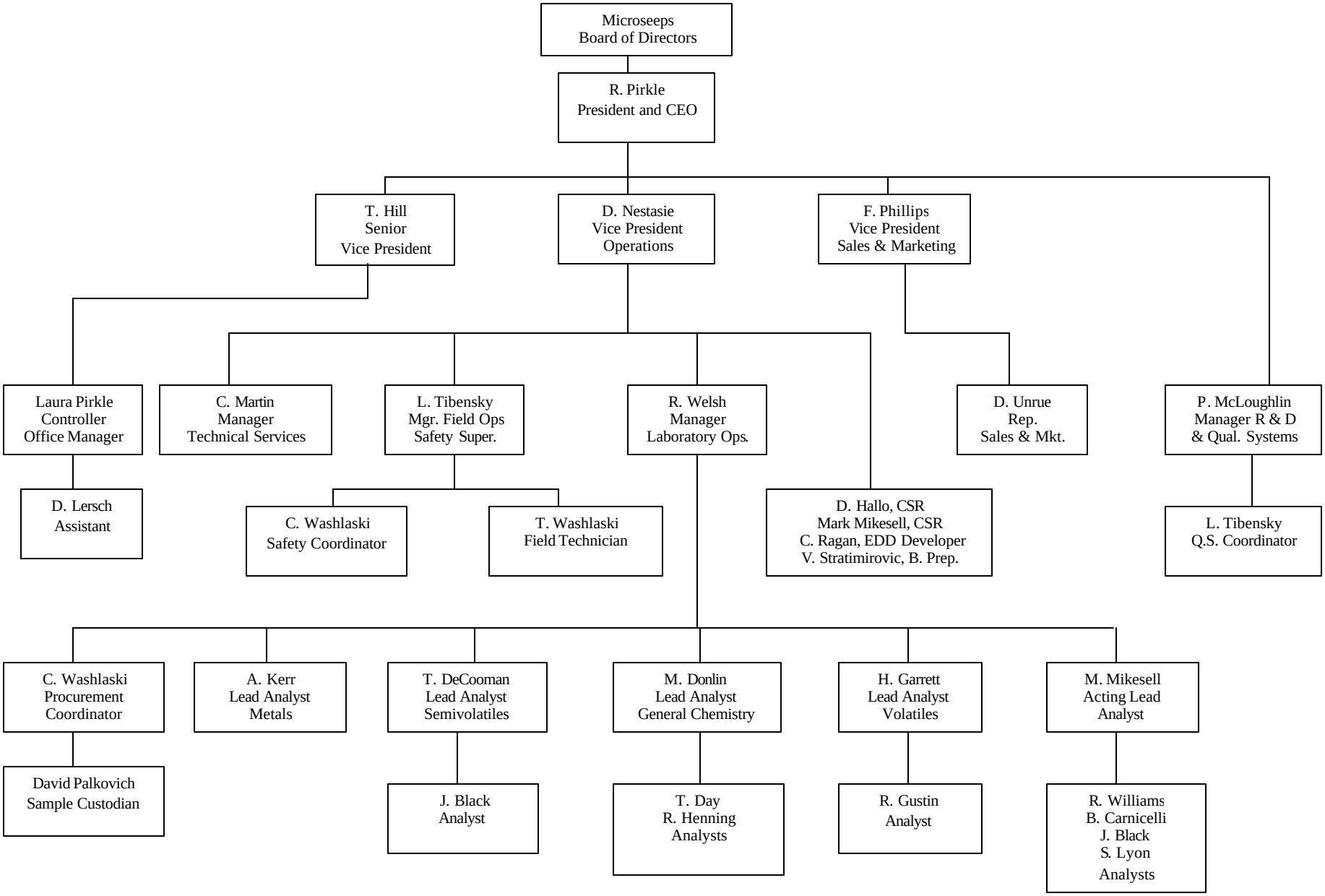


Figure 3-2

Flow Diagram for Contract Review, Design Control, and Quality Planning

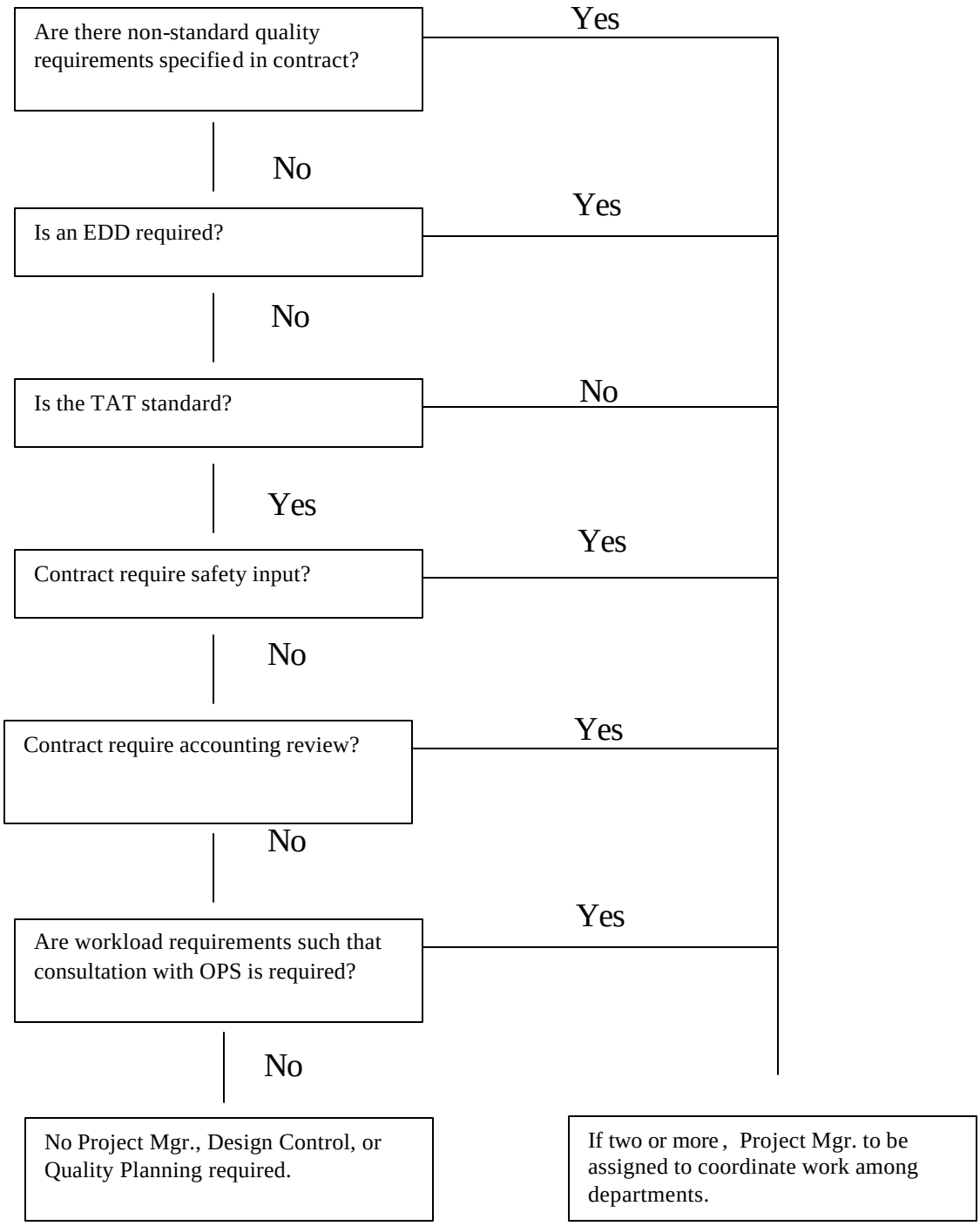


Figure 3-3

Contract Review Checklist

Please review the designated portion of the attached contract/scope of work and determine if your department/work group can or cannot meet all of the requirements of the contract.

Place an X in either the Can or Cannot column, date, and initial where indicated and return this form to the Project Manager within 24 hours of receipt.

Client Company: _____

Contact: _____

Microseeps PM: _____

Date Submitted	Department	Can	*Cannot	Date	Initials
	Operations: • Methods • Personnel • Detection Limits				
	Information Systems • EDD Format				
	Quality Systems • Certifications				
	Accounting • Payment terms				
	Safety • H & S Worksheet				
	Waste • Special requirements				
	Field				

***Please note on the back of this page the specific reasons why your department may not be able to meet the requirements of the contract.**

4.0 Sample Management

The purpose of this section is to outline and reference procedures for the type and use of sample containers, the preservation of the samples, sample shipping, sample receipt, sample custody, sample log in, sample disposal, and analytical subcontracting. A list of analytical procedures conducted by Microseeps is found in Tables 4-1 through 4-3 at the end of this section. Certifications are found in Tables 4-4 and 4-5.

4.1 Sample Containers and Preservation

In order to provide the best possible service for the client and to obtain the required sample volume, Microseeps prefers to provide the sampling containers. Sample containers are constructed of either polyethylene or glass. Containers are purchased that are pre-cleaned and ready for use. Certified clean containers can be provided if project specifications require. Preservatives are added to most sample containers prior to shipping to the client. Clients are encouraged to completely fill each container in order to provide adequate sample volume.

4.2 Sample Packing and Shipping

Samples are either hand carried to the laboratory or shipped using commercial carriers. Samples that have limited holding times should always be shipped to the laboratory daily using a reliable overnight shipping service.

The chain of custody is used to establish the identity of samples and to provide proof of possession of the samples by Microseeps. Chains of custody are supplied to the client with the sample bottle order. The following information should be recorded, by the client, on the chain of custody.

- Client Name, Address, and Phone Number
- Project Name
- Project Number
- Sample identification, Collection Time, Number of Containers
- Specific Analytical Requirements
- Sample Matrix
- To whom the analytical data shall be submitted
- Relinquish signature

Ice should be added so that a temperature of $4^{o}C \pm 2^{\circ}$ is maintained during shipment. Samples should be packed inside a large bag and the bag should be sealed. Bags of ice should be placed on top of the bag and should not be in direct contact with the sample containers. Cooler temperatures are taken electronically and recorded upon receipt by the laboratory.

The chain of custody should be enclosed in the zip lock bag provided by Microseeps, and taped to the inside lid of the cooler.

A copy of Microseeps' Sample Acceptance Policy is included with each client bottle order (see below). In order to expedite the sample log in process, please be sure to include and accurately complete all paperwork that should accompany samples to the laboratory.

Sample Acceptance Policy

1. Samples that are shipped to Microseeps must be accompanied by proper, full, and complete documentation. This documentation shall be marked on a chain of custody and shall include: sample identification, the location, date and time of collection, sampler's name, preservation type, sample type, specific parameters to be analyzed, and any special remarks concerning the sample.

2. Sample labels shall be supplied by Microseeps or the client. Those labels must be water resistant and completed using indelible ink. Each sample label must include a unique identification number that links it to the chain of custody documentation.

3. Samples shall be in the proper containers with the preservatives that are specific to the type of analysis required.

4. All samples must be received within specified holding times. Clients are requested to notify a Microseeps' Customer Service Representative if samples with short holding times are being shipped.

5. Samples must arrive at Microseeps with sufficient volume to conduct the requested analyses. All bottles should be filled completely.

6. When problems with samples or documentation are found during the sample receiving process, a Non-Conformance Form is completed by the Procurement Coordinator and forwarded to the Customer Service Office. A Customer Service Representative will make every attempt to contact the client as soon as possible to make decisions concerning those discrepancies. The Non-Conformance Form is kept as a permanent part of the project file.

7. If the client cannot be reached, a message will be left either on voice mail or with a receptionist for the client to return the phone call. The samples will be placed in a storage refrigerator and held until a Microseeps' Customer Service Representative gets a response from the client. (Exceptions will be made when samples are received that have short holding times and the samples are from a client with whom Microseeps has regular and frequent dealings. Or when the samples have short holding times and the samples are from a client with whom Microseeps has a signed contract, work order, or purchase order.)

4.3 Sample Custody

Microseeps takes custody of the samples when they are received at the laboratory or picked up at a client site by a Microseeps' employee. All samples are maintained in access-controlled areas until

work is started. The person responsible for either the sample preparation or analysis will retrieve the sample(s) from the storage area and return them when the function is complete.

4.3.1 Samples Requiring Secure Storage

For samples requiring locked storage and strict custody protocols (e.g. samples as evidence), the samples are locked in a temperature controlled sample storage cooler that is separate and distinct from other samples. The Procurement Coordinator is the only person with keyed access to this area. When evidentiary samples are to be analyzed, the Procurement Coordinator initiates a sample tracking record (Figure 4-2), removes the required samples from secure storage, and the analyst signs them out using the complete sample number as generated by the LIMS to identify the samples taken.

After analysis, all remaining sample, sample extracts, or the empty sample container are returned to secure storage, signed back in on the sample tracking record, and placed back in the secure storage area until the client authorizes their disposal. Entries are made to the form each time a sample is removed and returned to the storage areas. Whenever sample preparations are completed, i.e. organic extractions and metals digestions, the sample preparation group adds them to the tracking record. All records of evidentiary samples are maintained until the client authorizes destruction.

4.3.2 Sample Receipt Protocols

The Procurement Coordinator or designee signs for each shipment and a copy of the shipping documents is retained. Specific sample receipt procedures are addressed in Microseeps Standard Operating Procedure for Sample Receipt (SOP-S2). A brief outline follows:

- Shipment containers are inspected, opened, and monitored for temperature, if applicable.
- Temperature is recorded on the Chain of Custody.
- Shipment containers are unpacked and samples resolved with the chain of custody.
- Chain of Custody is signed.
- Non-conformances are resolved with the client.
- Samples are logged in to Microseeps LIMS system.

Specific sample log in procedures are outlined in Microseeps Standard Operating Procedure for Sample Log-In using the Laboratory Information Management System (LIMS). Microseeps LIMS assigns a unique internal project number and sequential sample numbers. These numbers are used to track the project through the laboratory. The sample numbers are transferred to each sample container using a computer-generated label. These numbers are documented on the chain of custody form and verified by the sample custodian. The computer generates a cooler receipt form, which is printed and placed in the project file along with the chain of custody and other related documentation.

All documentation relating to the project is maintained in the project file and retained in the laboratory for five years.

4.3.3 Resolution of Non-conformances

Sample receipt non-conformances are resolved according to Microseeps Standard Operation Procedure for Sample Receipt (SOP-S2). In nearly all cases, the client is contacted for the resolution decision and documentation.

Several possibilities may exist for resolving sample receipt problems. All decisions are the client's responsibility. Once a resolution is determined, the solution is noted on the non-conformance form and one or more of the following actions will occur:

- The log-in process will continue
- Written documentation will be requested from the client
- The sample(s) will be returned
- The sample(s) will be disposed

Throughout the problem resolution process, the sample will either be kept in a secure area or will be in view of the sample receipt personnel. All records generated during this process become a part of the client's permanent file.

When a client decides to proceed with analyses of samples that do not meet acceptance criteria, that decision shall be fully documented on the non-conformance form and the analysis data shall be "qualified" using a narrative and/or a data qualifier on the final data report.

4.4 Sample Storage and Recovery

Samples requiring refrigeration are placed into temperature-controlled coolers that are maintained at 4°C ± 2°. The cooler temperature is recorded each morning and afternoon. Temperature logs are maintained for each cooler.

All samples for volatile analysis are segregated in a cooler away from other samples. All samples are stored separately from standards, reagents, food, and other potentially contaminating sources.

4.5 Sample Disposal

Once samples are analyzed, they are moved from a primary to a secondary storage area. Samples are stored in secondary storage areas for thirty days following the date an analytical report is generated. Samples are disposed or returned according to the procedures outlined in SOP-ADM 14, Microseeps Standard Operating Procedure for Waste Disposal.

4.6 Subcontracting Analytical Samples

Microseeps' Laboratory Manager or his designee will notify the Project Manager when samples or extracts need to be sent to a subcontract laboratory, the number of samples to be sent, and the duration of the need for subcontract services. The Project Manager or Customer Service Representative will notify the client in writing of the intent to subcontract samples. The Assistant Laboratory Manager will schedule the work with the subcontract laboratory and notify Sample Receiving about the specifics of shipping the samples.

The Assistant Laboratory Manager shall monitor the progress of the analytical work and receive the analytical data from the subcontract laboratory.

In the event of expedited turnaround that cannot be met by Microseeps, the Project Manager shall initiate the subcontract laboratory procedure in order to meet the client's need.

4.6.1 Subcontract Laboratory Approval

The following procedures are in place to ensure that laboratories that are to be used for subcontracting analytical samples meet minimum requirements for quality as specified by the Quality Systems Department. Prior to approval of a subcontract laboratory, the Quality Systems department shall request the following information from the laboratory:

- List of current certifications and expiration dates of each.
- Copy of the Quality Assurance Plan for the subcontract facility.

Once this material has been received, it shall be reviewed by the Quality Systems Department and a decision will be made concerning the approval of the subcontract laboratory.

4.6.2 Client Notification of Subcontract Laboratory

There are various reasons why a subcontract laboratory may be used including, but not limited to, an expedited turnaround time, laboratory capacity, and special analysis. All clients shall be notified in writing when any samples are to be sent to a subcontract laboratory.

Where the laboratory subcontracts any part of the testing that is covered under NELAP, this work shall be subcontracted to a NELAP accredited laboratory. The laboratory performing the subcontracted work is indicated on the final report and non-NELAP accredited work is clearly identified.

TABLE 4-1

Container, Preservation, and Holding Time Requirements
(EPA Methods for Chemical Analysis of Water and Wastes Table I and SW-846 3rd ed. Revision 1, Table 2-21)

Parameter	Method	Container [1]	Preservative [2][3]	Maximum Holding Time [4]
Acidity	EPA 305.1	P	Cool to 4°C	14 days
Alkalinity	EPA 310.1 SM2320B	P	Cool to 4°C	14 days
Anions by IC	SW846-9056	G, VOA with TLS	Cool to 4oC	48 hours for NO_2, NO_3 28 days for other anions
BOD CBOD	EPA 405.1 SM5210	P	Cool to 4°C	48 hours
Bromide	EPA 320.1	P	Cool to 4°C	28 days
Chloride	SM 4500	P	Cool to 4°C	28 days
Chromium, Hexavalent	SM 3500	P	Cool to 4°C	24 hours
COD	EPA 410.4	P	Cool 4°C;H_2SO_4 to pH <2	28 days
Coliform, Fecal and T	SM 9222D	P	Pre-preserved	8 hours
Color	EPA 110.2	P	Cool to 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	EOA 335.2 SW846-9010 SW846-9014	P	Cool to 4°C; NaOH to pH >12[5]	14 days
Fluoride	EPA 340.2	P	Cool to 4°C	28 days
Hardness	EPA 130.2	P	Cool to 4ºC	6 months
MBAS (Surfactants)	EPA 425.1	P	Cool to 4°C	48 hours
Mercury	SW846-7470 EPA 245.2	P	Cool to 4ºC, HNO_3 to pH <2	28 days
Metals, except Cr^{+6} and Mercury	SW846-6010 EPA 200.7	P	Cool to 4ºC, HNO_3 to pH <2	6 months
Total Organic Nitrogen Ammonia Kjeldahl	SM 4500-N-C EPA 350.2 EPA 351.3	P	Cool 4ºC, H_2SO_4 to pH< 2	28 days
Nitrate	EPA 353.3	P	Cool 4ºC, H_2SO_4 to pH< 2	28 days
Nitrite	EPA 354.1	P	Cool to 4ºC	48 hours
Nitrate/Nitrite	EPA 353.3	P	Cool 4ºC, H_2SO_4 to pH< 2	28 days
Odor	EPA 140.1	P	Cool to 4ºC	24 hours
Oil and Grease	(40 CFR) 1664	AG	Cool 4ºC, HCl to pH< 2	28 days
TOC/DOC	SW846-9060 EPA 415.1	G	Cool 4ºC, H_2SO_4 to pH< 2	28 days
pH	EPA 150.1 SW846-9040	P	Cool to 4ºC	Immediate
Phenols	EPA 420.1 SW846-9065	AG	Cool 4°C; H_2SO_4 to pH <2	28 days

TABLE 4-1, Continued

Parameter	Method	Container[1]	Preservative[2][3]	Maximum Holding Time[4]
Phosphorus: o-phosphate	EPA 365.3 EPA 365.1	P P	Filter immediately; H_2SO_4 to pH <2 Cool to 4°C	28 days 48 hours
TDS TSS TS Percent Solids TVS	EPA 160.1 EPA 160.2 EPA 160.3 EPA 160.3 EPA 160.4	P, G	Cool to 4°C	7 days
Conductivity	EPA 120.1 SW846-9050A	P	Cool to 4°C	28 days
Flashpoint	SW846-1010	P	Cool to 4° C	None specified
Sulfate	EPA 375.4 SW846-9038	P	Cool to 4°C	28 days
Sulfide	EPA 376.1 SW846-9030B SW846-9031	P	Cool, 4°C; Zinc Acetate & NaOH to pH>9	7 days
Sulfite	EPA 377.1	P	Cool to	Immediate
Turbidity	EPA 180.1	P	Cool to 4°C	48 hours
Purgeable Halocarbons	SW846-5030 8260B & 8021 EPA 624 EPA 601	G with TLS	HCl to pH<2, Cool 4°C	14 days
Purgeable Aromatics	SW846-5030 8260B & 8021 EPA 624 EPA 602	G with TLS	HCl to pH<2, Cool 4°C	14 days
PCBs	SW846-3510 SW846-8082	G with TLC	Cool to 4°C	7 days to extract then 40 days.
Pesticides	SW846-3510 SW846-8081	G with TLC	Cool to 4°C	7 days to extract then 40 days.
BNAs	SW846-3510 SW846-8270C EPA 625	AG with TLC	Cool to 4°C	7 days to extract then 40 days.
Diesel Range Organics	SW846-3510 SW846-8015	G with TLC	1:1 HCl - 5 ml Cool to 4°C	7 days to extract then 30 days.
Gasoline Range Organics	SW846-8015	G with TLS	HCl to pH<2, Cool 4°C	14 days

1. AG - amber glass, G - glass, P - polyethylene, TLC - Teflon-lined cap, TLS - Teflon-lined septum
2. Sample preservation should be performed immediately upon sample collection. Composite samples may be preserved by maintaining at 4°C until sample splitting and collection is completed.
3. If the dissolved content is to be measured, samples should be filtered on site immediately before adding preservatives.
4. The holding times listed are the maximum times that samples may be held before analysis and still be considered valid under EPA regulations. Holding times are measured from sampling.
5. For CN analysis, add 0.6 g ascorbic acid/liter sample if residual chlorine is present.
6. pH should be 6-9.
7. Add $Na_2S_2O_3$ if residual chlorine is present (0.008 percent).

TABLE 4-2

Container, Preservation and Holding Time Requirements for Soil, Sediment, Sludge

Parameter	Method	Container[1]	Preservative	Maximum Holding Time
Volatiles	SW846-5035 8260B	G with TLC Encore	Cool 4º C MeOH & $Na(SO_4)_2$	14 days
Semi-volatiles	SW846-3550B 8270C	G with TLC	Cool 4º C	14 days until extraction. 40 days after extraction.
Ammonia	Mod. 4500B	G with TLC	Cool 4º C	28 days
Cyanide amenable	EPA 335.1 Mod.	G with TLC	Cool 4º C	14 days
DRO	SW846-3550B Mod.8015	G with TLC	Cool 4º C	14 days until extraction 40 days after extraction
Flashpoint	SW-846-1010	G with TLC	Cool 4º C	Not specified
Mercury	SW846-7471	G with TLC	Cool 4º C	28 days
Oil and Grease	SW846-1664	G with TLC	Cool 4º C	28 days
Paint Filter	SW846-9095	P	Cool 4º C	Not specified
PCB solid or wipe	SW846-3550 SW846-8082	G with TLC	Cool 4º C	7 days to extract then 40 days
Percent Solids	EPA 160.3	G with TLC	Cool 4º C	7 days
pH 1:1	SW846-9045C	G with TLC	Cool 4º C	Immediate
Phenol	SW846-9065	G with TLC	Cool 4º C	28 days
Cyanide Reactive	SW846-9014	G with TLC	Cool 4º C	14 days
Cyanide Total	SW846-9010B/ 9014	G with TLC	Cool 4º C	14 days
Sulfide Reactive	SW846-9034	G with TLC	Cool 4º C	7 days
Specific Gravity	SM2710 F	G with TLC	Cool 4º C	28 days
Mercury TCLP	SW846-1311/ 7470	G with TLC	Cool 4º C	28 days from collect. to extract. 28 days from extract. to analysis
Metals TCLP	SW846-3010/ 6010B	G with TLC	Cool 4º C	180 days from collect to extract. 180 days from extract. to analysis
Metals Total	SW846-3050/ 6010B	G with TLC	Cool 4º C	6 months
Pesticides TCLP	SW846-8081	G with TLC	Cool 4º C	40 days
Pesticides	SW846-8081	G with TLC	Cool 4º C	40 days
Semi-volatiles TCLP	SW846-8270C	G with TLC	Cool 4º C	14 days from collect. to extract. 7 days from extract. to prep. 40 days from prep. to analysis
Volatiles TCLP	SW846-5030B/ 8260B	G with TLC	Cool 4º C	14 days from collect. to extract. 14 days from extract. to analysis
Total Kjeldahl Nitrogen	$SM4500N_{org}C$	G with TLC	Cool 4º C	28 days
Total Organic Nitrogen	SM4500-N-C	G with TLC	Cool 4º C	28 days
Total Volatile Solids	EPA 160.4	G with TLC	Cool 4º C	7 days
Semivolatile, Pesticide and PCB Prep. Methods	EPA 3510C/ 3550B/3580A	NA	NA	7 days

1. TLC = Teflon-lined cap, G = Glass

Table 4-3

Bioremediation Indicator Parameters and Risk Analysis Test Methods
These parameters are analyzed using Microseeps' Methods.

Parameter	Method	Container	Preservative	Maximum Holding Time
Hydrogen by Bubble Strip	SM9/AM20Gax	22 cc vapor vial with stopper septum	None	14 days
Light hydrocarbons by Bubble Strip	SM9/AM20Gax	22 cc vapor vial with stopper septum	None	14 days
Permanent gases by Bubble Strip	SM9/AM20Gax	22 cc vapor vial with stopper septum	None	14 days
Light hydrocarbons in water: Methane, Ethane Ethene	PM01/AM20Gax	40 ml Amber VOA vial with mylar septum	Trisodium Phosphate & Cool to 4ºC	14 days
Permanent gases in water: Oxygen, Nitrogen, Carbon Dioxide	PM01/AM20Gax	40 ml Amber VOA vial with mylar septum	Benzalkonium Chloride & Cool to 4ºC	14 days
Light hydrocarbons in vapor	AM20Gax	22 cc vapor vial with flat septum	None	14 days
Permanent gases in vapor	AM20Gax	22 cc vapor vial with flat septum	None	14 days
Hydrocarbons in vapor	AM4.02	22 cc vapor vial with flat septum	None	Unspecified
Chlorinated hydrocarbons in vapor	AM4.02	22 cc vapor vial with flat septum	None	Unspecified
Fuel Oxygenates in water	AM22G	40 ml clear VOA with teflon septum	Trisodium Phosphate and Cool to 4ºC	14 days
Total inorganic carbon in water	PM01/AM20Gax	40 ml Clear VOA vial with mylar septum	Cool to 4ºC	14 days
Volatile Fatty Acids	AM21G	40 ml Clear	Cool to 4°C	21 days
Low Level VFA	AM23G	40 ml Amber with teflon septum	Cool to 4°C Benzalkonium chloride	14 days
AMIBA	WC-43	BAFeIII: 8oz plast. All by direct push: 2 oz glass soil jar All by drill. rig: 2 – 40 ml amber 2 oz glass soil jar	Freeze	3 months

Table 4-4
STATE CERTIFICATIONS

Parameter	Method	State Certification
Alkalinity	EPA 310.1, SM2320	WV, SC, CT
Chloride	SM4500 Cl B; SW846-9056	WV, SC, CT
Hexavalent Chromium	SM3500 Cr D, SW846-7196A	WV, CT
Cyanide	SW846-9010B & 9014	WV, CT
Hexane Extractable Material	EPA 1664 A	WV
Specific Conductance	SW846-9050	WV, CT
Sulfate	EPA 375.4, SW846-9056	WV, SC, CT
Sulfide	EPA 376.1, SW846-9034	WV, SC, CT
Orthophosphate & total Phosphorus	EPA 365.3	WV, CT
Temperature	EPA 170.1, SM2550 B	WV, CT
BOD	EPA 405.1	WV, CT
CBOD	EPA 405.1; SM5210B	WV, CT
COD	EPA 410.4, SM 5220 D	WV, CT
TOC	SW846-9060	WV, SC, CT
Ammonia	EPA 350.2, SM4500 NH_3 B & E	WV, CT
Nitrate	SW846-9056	WV, SC, CT
Nitrite	EPA 354.1, EPA 300.0; SW846-9056	WV, SC, CT
TDS	EPA 160.1; SM2540C	WV, CT
TSS	EPA 160.2; SM2540D	WV, CT
Total Solids	EPA 160.3; SM2540B	WV, CT
Mercury	EPA 245.2, SW846-7470 & 7471A	WV, CT, SC
Metals (TAL)	EPA 200.7, SW846-6010C	WV, CT, SC
Volatiles (TCL)	SW846-8260B; EPA 624	WV, CT, SC
BNAs	SW846-8270C; EPA 625	WV, CT, SC
Pesticides	SW846-8081A	WV, CT, SC
PCBs	SW846-8082	WV, CT, SC
Extractions	SW846-3510C, 3550B, 5030B, 5035	WV, CT, SC
Digestions	SW846-3010, 3050	WV, CT, SC

Check with Microseeps Customer Service Office for state specific parameter lists.

Table 4-5

NELAP Accredited Parameters/Methods

Primary NELAP: Pennsylvania
Secondary NELAP: FL, NY, NJ, LA
(Not all states accredit all parameters.)

Parameter	Method
Alkalinity	EPA 310.1, SM2320B
Chloride	SW846-9056; SM4500 Cl-B
Nitrate	SW846-9056
Hardness	EPA 130.2
Nitrite	EPA 354.1, SW846-9056
HEM	EPA 1664A
Orthophosphate	EPA 365.3
Sulfate	EPA 375.4, SW846-9056
Sulfide	EPA 376.1
BOD	EPA 405.1
CBOD	EPA 405.1, SM 5210B
TOC	SW846-9060
Metals	SW846-6010B & EPA 200.7
Mercury	SW846-7470
Volatile Organics	SW846-8260B
Fuel Oxygenates	SW846-8015D & 8260

Call Customer Service Office for state-specific analyte list.

Figure 4-1
Chain of Custody Form

CHAIN - OF - CUSTODY RECORD

Company : _____
Co. Address : _____
Proj. Manager : _____
Proj. Location : _____
Proj. Number : _____
Phone # : _____ **Fax # :** _____

Sampler's signature : _____

Parameters Requested

Results to : _____

Invoice to : _____

Cooler ID	Cooler Temp.

Sample ID	Sample Description	Date	Time	Comp.	Grab	# Cont.	Remarks

Relinquished by :	Company :	Date :	Time :	Recived by :	Company :	Date :	Time :
Relinquished by :	Company :	Date :	Time :	Recived by :	Company :	Date :	Time :
Relinquished by :	Company :	Date :	Time :	Recived by :	Company :	Date :	Time :

WHITE COPY : Accompany Samples YELLOW COPY : Laboratory File PINK COPY : Submitter

Figure 4-2
Sample Tracking Record

Sample Storage Location: _____

Samples placed in storage by: _____

Circle Bottle Type:		T-Metals	Sulfide	Cyanide	D-Gas	Hydrogen
IC-VOA	Nutrient	D-Metals	BNA	Bacteria	Vapor	G. Chemistry
HEM	Phenol	TOC/TOX	VOA	Soil	VFA	Fuel-Oxygenate
TIC	D-Gases	GRO	DRO	Pesticide	PCB	LLVFA

LIMS generated Sample Numbers	**Removed from Storage**			**Returned to Storage**		
	Initials	**Date**	**Time**	**Initials**	**Date**	**Time**

5.0 Facilities, Instrumentation, and Materials Procurement

5.1 Facility Description

Microseeps is located in the University of Pittsburgh's Applied Research Center (UPARC). The complex consists of 58 separate buildings, which house approximately 130 companies. The Oxford Development Company provides management for the UPARC facility including building maintenance and safety support. Oxford Development's maintenance personnel are on-site and respond quickly and efficiently to all internal environmental or air quality issues. Specialized maintenance staff is on-call to respond to ventilation, heating, cooling, lighting, or other problems that may occur.

Laboratory temperatures are controlled by the permanent heating and air-conditioning systems in the UPARC complex. Where the cooling systems have not been efficient enough to maintain correct temperature and humidity requirements for proper instrument function, Microseeps has installed additional units as needed.

When environmental conditions jeopardize the results of environmental tests, the Lead Analyst shall notify the Laboratory Manager immediately of the problem. If the problem cannot be resolved immediately the Laboratory Manager shall order a stoppage of all work until either the problem is resolved, or another area of the laboratory can be utilized to continue testing.

Microseeps is located in Building B-1 and has offices and laboratories on the first, second, and fourth floors.

5.1.2 Laboratory Areas

The following table specifies the locations of the individual laboratories and workspaces in the building in which Microseeps is housed.

First Floor

- Bottle Preparation — Room 110
- Sample Receiving — Room 115/117
- Volatiles Laboratory — Room 126/128

Second Floor

- Dissolved Gas Laboratory — Room 220/222
- Semi-volatiles Laboratory — Room 223
- Vapor Laboratory — Room 221
- Metals Laboratory — Room 213
- LIMS Center — Room 218

Fourth Floor

- Wet Chemistry Laboratory Room 415
- Metals Preparation Room 423
- Semi-Volatiles Preparation Room 425
- Instrument Laboratory Room 430

5.1.3 Building and Laboratory Security

Employee access into the UPARC complex is controlled through key-card turn-styles where each individual that works in the complex has a unique code for entry. UPARC Security is aware of who is on-site or off-site at any given time. Microseeps' laboratory areas are controlled through keyed entry to prevent employees from other firms housed in the complex from gaining access to Microseeps' laboratories. Each employee is issued a key that will open doors to rooms occupied by Microseeps. Those keys do not access accounting or CEO offices. During normal working hours, the laboratory areas are kept unlocked. After normal business hours the rooms are locked to prevent unauthorized personnel entry.

A UPARC Security Force monitors the facility twenty-four hours a day with a series of video cameras. The guards also make rounds by foot and vehicle during afternoon and night shifts. Visitors cannot gain access to the complex except through the Main Security Gate. All visitors are required to register at the main gate and obtain a visitor's pass before entering the complex. UPARC Security notifies Microseeps upon the visitor's arrival to verify admittance. Visitors are directed to Microseeps Reception Office. The visitor is then escorted, by a Microseeps' employee to the Microseeps' employee or laboratory they intend to visit.

5.2 Instrumentation

Instrumentation must be properly calibrated and maintained to produce reliable and reproducible results. This section of the Quality Systems manual defines minimally acceptable standards for installation, calibration, and maintenance of analytical instruments used in the laboratory. Fully detailed procedures are instrument specific and are available in the individual instruments' Operator Manuals.

A list of Microseeps' Instrumentation is included in Exhibit 5-1. This list is updated whenever equipment is placed in service or removed from service.

5.2.1 Installation and Set-up

All new instrumentation must be included in the Quality Systems program prior to being used for sample analysis. When new equipment is ordered, the Laboratory Manager and the Manager of Technical Services determine the preparations that the laboratory must make to accommodate the equipment. This plan includes descriptions of facility modifications that may be required,

personnel responsible for installation (manufacturer or Microseeps employee), performance criteria that need to be met, and training procedures that will be followed.

Data generated during installation and set-up will be included in the maintenance log for the instrument. This data may become important later for troubleshooting and diagnostic checks. Operator manuals supplied by the manufacturer are maintained in the laboratory for reference.

5.2.2 Calibration

Calibration procedures for instrumentation are thoroughly documented and routinely followed to provide assurance that the data produced are reliable and accurate. Specific calibration procedures and frequency are detailed in the individual Standard Operating Procedures.

Initial calibration involves comparing instrumental response to various concentration levels of the analytes of interest. The calibration curve will contain a minimum of three points, excluding a blank. The lowest point of the curve should be approximately 2 - 5 times the instrument detection limit. The most concentrated standard should be below but near the upper concentration limit of the linear range. All standards are prepared from solutions of certified concentrations. All calibrations are checked against a "second source" obtained from, preferably a different vendor, but at least a different preparation. All calibrations are followed by an initial calibration blank to verify that system contaminants and carry-over are not present. For organic analyses, surrogates are added to each blank and standard. In addition, internal standards are also added for some organic analyses.

If the initial instrument calibration results are outside established acceptance criteria, corrective actions are performed. These criteria and the corrective action are specified in individual Standard Operating Procedures. Data associated with unacceptable initial instrument calibration is not reported.

5.2.2.1 Calibration Verification

If an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration is verified prior to sample analysis by running continuing instrument calibration verification standards with each analytical batch. Continuing instrument calibration verifications must be run at the beginning and end of each analytical batch.

5.2.3 Instrument Maintenance

Maintenance of analytical instruments is carried out under the direction of the Manager of Technical Services and may include regularly scheduled preventive maintenance, or maintenance on an as-needed basis due to instrument malfunction. Maintenance activities for instrumentation are documented in Instrument Maintenance Logs. This documentation becomes a part of the laboratory's permanent records.

Regular maintenance of support equipment, such as balances, thermometers, and fume hoods is conducted annually and more often if required. Maintenance on other support equipment, such as ovens and refrigerators is conducted on an as needed basis. The Lead Analysts are responsible for ensuring that temperatures of ovens and refrigerators are checked and recorded twice daily. The Laboratory Manager is notified if the temperature is outside of the range of use of the specific piece of support equipment, and the equipment is scheduled for maintenance.

Records of maintenance to support equipment are also documented in Maintenance Logbooks. Each piece of support equipment does not necessarily have it's own logbook. Maintenance logbooks may be shared with equipment that is housed in the same laboratory area.

5.2.3.1 Out of Service Instruments

In the event that an instrument cannot be calibrated or is determined to be out of order, an out of service or out of calibration tag is placed on it. The Lead Analyst is responsible for ensuring the equipment is tagged. This tag shall be removed when the instrument is repaired and ready for use.

5.2.3.2 Instrument Repair

Unexpected repairs resulting from instrument failure are scheduled immediately after the malfunction is observed. Instrument failures are detected through direct observations and by evaluation of the response of verification standards throughout the analytical run. The Manager of Technical Services is responsible for deciding if laboratory personnel can make the repair or if an outside contractor is required.

Data obtained during instrument failure are not entered into the LIMS for reporting to the client. Complete records of the repairs are maintained in the Instrument Maintenance Logbooks. These records may include notes taken by laboratory personnel during repair and a copy of the service call record. Acceptable instrument performance must be verified before samples can be analyzed.

5.3 Materials Procurement

The purpose of this section is to define requirements for the procurement of materials needed to support laboratory operations.

5.3.1 Purchase and Control of Standards, Reagents, and Materials

The Laboratory Manager reviews and approves all material orders. The quality of purchased standards and reagents is determined as specified in approved analytical methods and further specified in Microseeps' Standard Operating Procedures. All standards and reagents are to be handled and treated in accordance with the Standard Operating Procedure for Analytical Standards and Reference Materials (SOP-ADM 15).

5.3.2 Placing Orders

The employee specifies the materials that need to be ordered on a Purchase Order Form that can be obtained from the Procurement Coordinator. The Purchase Order form is completed by filling in a suggested vendor's name and catalogue number, a description of the item, the quantity requested, the date of the request, their name, and the accounting category. The form is forwarded through the Lead Analyst or Department Head for approval. The Laboratory Manager has the discretion to delay the order, obtain equivalent materials from another supplier, or adjust the quantity if it is in the best interest of the laboratory to do so. It is the manager's responsibility to choose an acceptable vendor. Once the order is approved, the Procurement Coordinator places the order.

5.3.3 Receipt of Materials

Whenever supplies or materials are received, the Procurement Coordinator compares the materials received to the packing list. Any discrepancies are noted on the packing list. A call is placed to the vendor to resolve any problems.

Once the order is reconciled with the purchase order, the packing list(s) is attached to the purchase order and is kept on file by the Procurement Coordinator. The materials and associated documentation (i.e. Certificates of Analysis or Purity) are forwarded to the Department that placed the order for storage until use.

All Material Safety Data Sheets are forwarded to Microseeps' Safety Coordinator for cataloging.

Figure 5-1
Microseeps' Instrument and Equipment List

Gas Chromatography/Mass Spectrometry

Volatiles

Hewlett Packard Chemstation Data System with NIST Chemical Structures; Hewlett Packard 6859Series II GC (3/05) with EPC and 5973 Mass Spectrometer; Varion Archon Autosampler (Serial 14288) (3/05) and Tekmar Velocity XPT Concentrator (Serial US04159007) (3/05).

Hewlett Packard Chemstation with Extended MS Software; Varian Archon Autosampler (Serial 14288); Hewlett Packard Gas Chromatograph Model 5890 Series II, with Hewlett Packard 5971 Series Mass Selective Detector, Tekmar Velocity Concentrator (Serial VS4244003).

Semi-volatiles

Hewlett Packard 5890 Series II GC (Serial 3501A02409) with Hewlett Packard 5972 Mass Spectrometer (Serial 2950A45623) with Hewlett Packard 7673 Autosampler (Serial 3323A35213)

Gas Chromatography

Pesticides and Polychlorinated Biphenyls

Hewlett Packard 5890 Series II Gas Chromatograph with dual ECDs (Serial 3310A48870) with Hewlett Packard 7673 Auto-injector and sample tray (Serial 3009A21045).

Fuel Oxygenates

Hewlett Packard 5890 Series A Gas Chromatograph (Serial 2536A05842) with Tekmar 7000 Autosampler (Serial 91099014/91135007).

Agilent 6890 Gas Chromatograph (Serial US10347026) with Agilent G1888 Headspace Autosampler (serial IT40220036).

Vapors

Hewlett Packard 5890 Series A Gas Chromatograph (Serial 2536A05842) with Tekmar 7000 Autosampler (Serial 91099014/91135007)

Hewlett Packard 5890 Series II Plus (Serial 3336A53505)

Volatile Fatty Acids

(LLVFA) Dionex Ion Chromatograph, Model LC25 (Serial 97040068); Autosampler Model AS40-1 (serial 97050241); Eluent Generator Model EG50 (Serial 94050039); Conductivity Generator Model CD20-1 (Serial 97050142); Gradient Pump Model GP40-1 (serial 97030784).

Varian 3400 Gas Chromatograph (Serial 10272) with Varian 8100 Autosampler (Serial 1371)

Mobile Laboratory

Hewlett Packard 5890 Series II (Serial 3336A51836) with Tekmar 7000/7050 Autosampler (Serial 91346008/91346016)

Dissolved Gases and Hydrogen

Three Proprietary Gas Chromatographs

Metals

Varian Vista AX CCD Simultaneous ICP-AES (SPSS) (Serial EL99063568) with Auto Sampler (Serial EL99063365) (8/99)

Perkin Elmer Flow Injection Mercury System (FIMS100) (Serial 509550-1410) with AS 90 Auto Sampler (Serial 508540-3767) (12/99)

CPI Mod Block centrifuge type Digestion Unit (7/99)

Fisher Versa-Bath (pre 1992)

Mettler PE-4000 Balance (Serial C39640) (pre 1992)

Extraction Laboratory

Horizon Speed-Vap II 9000 Solvent Evaporation System (Serial 02-1801) (4/02)

Wet Chemistry

Dionex 500 Ion Chromatograph with dual Autosamplers, columns and ovens, with electrochemical and UV-Vis Detectors.

Thermo Electron Hipertoc Model TOC Analyzer (12/04).

Spectronic 20G Colorimeter

Spectronic 20D Colorimeter

Variety of pH and Conductance Meters, balances, and glassware.

Orion 601A pH Meter

Acumet Model 50 pH Meter

Buchi EL131 Rotovaporator

Fisher Scientific Flash Point Tester

Denver A-200D Analytical Balance

Sartorius Model 1612 Analytical Balance

HF Scientific Instruments DRT-15C Turbidity Meter

6.0 Data Quality

6.1 Data Quality Objectives

Microseeps conducts analysis on environmental samples for clients who rely on the data in order to make decisions concerning environmental problems, environmental monitoring, and in many cases to investigate the feasibility of cutting edge remedial technologies. The necessity of high quality data is essential so that the best decisions can be made in the interest of both the environment and the client. Microseeps believes data quality objectives are applied to a project from the initial sampling to the final data validation process. On a laboratory scale, Microseeps' data quality objectives are reflected in individual Technical Standard Operating Procedures as quality control acceptance criteria.

Quality control sample acceptance criteria is generated using one of following three methods:

- A minimum of twenty data points are manually collected and entered into an EXCEL spreadsheet. The average percent recovery and/or relative percent difference (RPD) is calculated, whichever is applicable. Acceptance criteria are generated using the standard deviation of the average percent recovery and RPD. Three standard deviations comprise the acceptance range around the average percent recovery and above the RPD.
- Calculated electronically by the LIMS database and expressed in the Control Chart Program.
- Taken from EPA method-specific recommendations in the absence of laboratory-generated criteria.

Acceptance criteria generated from the implementation of control charts, either manually or LIMS generated are evaluated annually or more often. If it is determined that the acceptance criteria has changed to a broader set of limits, the reason for the change is evaluated for error to ensure the analytical method is still in control. When warranted, corrective action shall be instituted by the Quality Systems office or the Operations Department.

All analytical data including quality control results are checked in accordance with SOP-ADM 16 Standard Operating Procedure for Data Integrity, Review and Validation. Performance Evaluation studies are conducted twice a year and the results are reviewed by management. All failed PE samples are followed up with Corrective Actions.

To maintain the quality of laboratory data and to ensure that laboratory procedures are under control, a variety of internal batch quality control samples are analyzed. The data from those samples are used to calculate statistics that help determine precision, accuracy, and to track potential bias. In addition, performance evaluation studies are conducted regularly as well as random submission of blind quality control samples. Certified reference standards are used, as well, to ensure that quality is maintained.

6.2 Internal Batch Quality Control

Batch quality control sample types and frequencies are recommended in published methods and specified in Microseeps Technical Standard Operating Procedures. In general, a batch of samples consists of twenty samples or fewer (as recommended by analytical methods) that are analyzed at the same time. Typically, a set of internal quality control samples is analyzed once for each batch of clients' samples. The types of internal batch quality control samples are discussed below.

6.2.1 Initial and Continuing Calibration Verification Standards

Initial and continuing calibration standards verify the ratio of instrument response to the analyte amount. Typically, initial calibration verification and continuing calibration standards are made from stock solutions, which are different from the stock used to prepare the initial calibration standards.

6.2.2 Calibration Blank

A calibration blank is a 'clean' sample made from an appropriate matrix and/or solvent. The calibration blank is analyzed to insure that there is no contamination in any part of the analytical system, or to establish a baseline if that "contamination" is expected and known to be of consistent concentration. Calibration blanks are analyzed after each initial, continuing, and calibration verification standard. The analytical result of the blank must be below the laboratory's quantitation limit in order for analysis to proceed. If the result of the analysis is above the quantitation limit, the source of contamination must be identified and eliminated. The one exception involves the presence of common laboratory solvents as defined by the EPA.

6.2.3 Method (Preparation) Blanks

Method blanks are reagent water or, for solid/waste matrices, sand or an appropriate solvent is carried through the entire analytical process to monitor potential contamination that may or may not be introduced during sample preparation and processing. For organic analyses, surrogates and internal standards are added to the method blank.

Method blanks are analyzed at the beginning of the batch and prior to sample analysis. If the analytes of interest are below the laboratory's quantitation limit, sample analysis can proceed. If analyte concentrations are found above the quantitation limits, the source of the contamination must be identified and corrected. The reagents used for sample preparation must be checked for contamination and the samples associated with the method blank must be prepared again and reanalyzed if necessary.

6.2.4 Duplicates

Duplicates are analyzed to assess precision of the analytical procedure. Samples for batch duplicate analyses are selected at random, ensuring that the selection is rotated among client samples so that various matrix problems may be noted and/or addressed. If the sample requires

that an aliquot be removed and placed in another container in order to conduct the duplicate analysis, a representative aliquot is collected using one of the following options discussed below in Subsection 6.3.4.

Precision of the analyses may vary due to the matrix effects of the sample. If the precision is outside established control limits, the duplicate analysis is repeated. If the precision is still outside established control limits and all other quality control checks are within control, a matrix effect is assumed.

6.2.5 Matrix Spike/Matrix Spike Duplicates

Matrix spike and spike duplicate samples are analyzed to determine the extent of matrix bias or matrix interference on analyte recovery and to determine sample-to-sample precision. Analytes stipulated by the method, by regulations, or by other requirements must be spiked into the sample. If not supplied by the client, the analyst may choose these samples at random. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

Recovery data is highly dependent upon matrix effects. If acceptable recoveries are observed, it is determined that matrix is having no significant affect on the analytical procedure and that sample preparation and analysis have been performed correctly. Whenever precision, calibration, and system quality control checks are acceptable, large or small matrix spike recoveries are attributed to matrix effects. Because samples are spiked prior to analysis, the concentration of the analyte of interest in the sample may be so high that the spike amount is insignificant. In these cases, spike recovery is meaningless and is not calculated.

6.2.6 Laboratory Control Samples

Laboratory control samples are samples that are spiked with a specific concentration of known reference materials, independent of the calibration standards that are carried through the entire analytical process. These standards are used to assess the accuracy of the analytical process. Where possible, acceptance limits are statistically based upon actual laboratory data. If results are outside acceptance limits, corrective action must be performed before sample analyses can proceed.

6.2.7 Surrogate Standards

Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to organic analysis.

If surrogate recovery is not within acceptance limits, corrective action is instituted and reanalysis of those samples occurs.

6.2.8 Insufficient Sample Volume

When there is insufficient client sample volume in a batch to conduct quality control samples for precision, then a laboratory control sample and laboratory control sample duplicate are analyzed.

6.3 Measurements of Data Quality

Data quality measurements vary from parameter to parameter, are represented as warning and control limits, and are displayed along with associated data on control charts. The following types of measurements are utilized by Microseeps to insure the highest quality data is being provided.

6.3.1 Precision

Precision is a measure of the degree of mutual agreement among individual measurements made under prescribed conditions. Precision of laboratory data is determined through duplicate analyses of samples or matrix spikes and spike duplicates, and is calculated for Microseeps purposes, as either the range or relative percent difference (RPD) of the measurements.

6.3.1.1 Range

Range is defined as the difference between the highest and the lowest value reported for a sample. Rang is used in the laboratory as recommended in published regulatory methods. The formula for calculating range is as follows:

$$Range = HighestValue - LowestValue$$

6.3.1.2 Relative Percent Difference

Relative percent difference (RPD) is used for all of the analytical methods at the laboratory where sample duplicates are analyzed and where both matrix spikes and matrix spike duplicates are analyzed. If one or both measurements are less than the reporting limit, precision is not calculated. The formula for calculating relative percent difference is as follows:

$$RPD = \frac{|A - B|}{\frac{A + B}{2}} \times 100\%$$

6.3.2 Accuracy

Accuracy is the measurement of agreement between a measurement and the true value. It is calculated as the percent recovery of standards and spikes. Accuracy is calculated as the percent recovery of laboratory control samples, matrix spikes, and in organic chemistry surrogate recoveries.

6.3.2.1 Percent Recovery

Percent recovery is used for all of the analytical methods at the laboratory where laboratory control samples, matrix spikes, and/or surrogates are analyzed. The formula for calculating percent recovery is as follows:

$$\%\,\mathrm{Recovery} = \frac{Measurement}{TrueValue} \times 100\%$$

6.3.3 Bias

Bias is defined as a systematic error due to the experimental method that causes the measured values to deviate from the true value. Bias is determined by plotting the average percent recovery and the average relative percent difference on a control chart. A bias is suspected when seven successive data points are plotted on the same side of the average. This is considered an out of control event.

6.3.4 Representativeness

Representativeness is defined as data that accurately and precisely reflect the sampling points or environmental conditions. Numerous items throughout sampling and sample handling must be controlled to maximize representativeness. These include sample collection, preservation, and holding times. Since Microseeps does not perform sample collection, Microseeps cannot accept responsibility for representativeness of sample collection.

When an aliquot must be removed from the sample container for analysis or for making batch quality control samples, one of the following two options are used in order to obtain a representative aliquot:

1) If the analysis won't be compromised by agitation, then the sample is stirred, mixed, crushed, blended, as needed, and the aliquot is removed from sample container and placed in another container.

2) If the analysis may be compromised by shaking or mixing the sample, then portions of the aliquot are taken from different places within the original sample container ensuring that the aliquot is as representative as possible of the original sample.

In some cases where a sample cannot be homogenized, the client will be contacted and a course of action will be decided upon according to a mutually agreed upon decision.

6.3.5 Comparability

Comparability is the confidence level with which one set of data can be compared to a related set of data. Microseeps uses EPA recommended methodology, whenever feasible, participates in internal and external performance evaluation programs, and uses standard reference materials for sample analysis as means of enhancing comparability.

6.4 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Microseeps validates non standard and laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The laboratory keep records of results obtained, the procedures used, and a statement that the method is fit for the intended use.

Records of method development are kept in the Technical Director's Office.

7.0 Data Handling

Microseeps maintains records that enable the re-creation of the specific conditions under which data are produced, and method specific Standard Operating Procedures outline procedures for data collection, reduction, validation, and reporting. This section discusses, in general, Microseeps data handling procedures.

7.1 Data Collection

All laboratory employees are responsible for maintaining laboratory records and documenting them in sufficient detail to recreate analyses. Manually entered data are made using permanent ink. Corrections are indicated by drawing a single line through the incorrect entry, dating and initialing the correction, and coding the reason for the correction. The use of correction fluid or tape, erasure, or other means of making corrections is prohibited. The following information is recorded at the bench either manually or printed out via the data system interfaced with the analytical instrument.

- Method performed.
- Analysis date.
- Analysis time.
- Analyst signature or initials on computer printouts.
- Instrument Identification and settings.
- Analytical sequence consisting of a chronological listing of the processing for each standard, quality control check, and sample recorded in an analytical sequence, run log, or data sheet.
- The laboratory sample number.
- Quality control sample type.
- Standard identification and volume used for all calibration standards.
- True value and lot number of all spiked quality control samples.
- Dilutions including actual initial and final volumes.
- Sample aliquot and final volume.
- Instrument reading.
- Final results with units.
- Calculations for all quality control checks.
- Narrative describing any unusual observances.

If an analysis extends over more than one shift or day, each person responsible for part of the analysis records the date and time their portion of the analysis was initiated.

7.2 Data Reduction

Reducing raw data into a presentable form is the responsibility of the analyst performing the determination. The actual equations used to calculate final results are found in the analytical methods. The following general rounding rules are used for the calculations:

- Data is not rounded until the final answer is obtained.
- To round a figure, the number of significant figures is determined. If the figure to the right of the immediate right-most significant figure is greater than 5, round up. If this figure is less than 5, truncate the result after the last reportable figure. If this figure is equal to 5 and there are non-zero digits to the right, round up. If the figure is equal to 5 and there are no non-zero digits to the right, round up when the preceding figure is odd, and truncate when the preceding figure is even.

7.3 Data Validation

Each analyst initials and dates the data that is generated in the laboratory. All data generated by the laboratory undergoes either an independent peer review or a review by a lead analyst or other designated individual to ensure compliance with accepted quality control standards prior to data entry. The purpose of this review is to check for precision, accuracy, and completeness. The following items are verified during this review. Not all items are applicable to each test.

- Holding times.
- Proper measurement units.
- Instrument tune and initial calibration criteria.
- Proper number of calibration standards and blanks.
- Surrogate and spike percent recoveries.
- Comparison of quality control sample results to acceptance criteria.
- Corrective action for out of control conditions.

After this review, all data that is not instrument-interfaced directly with the Laboratory Information Management System (LIMS), is manually entered into the database. All manually entered data undergoes a review upon entry. The Laboratory Director attempts to review approximately 10% of all laboratory data. Project Managers complete the data validation process by reviewing final reports for completeness prior to submission to the client.

7.4 Laboratory Information Management System

The LIMS is the point of collection for all of the laboratory data. The integrity of laboratory data is of the utmost importance. The LIMS system has built-in security levels that keep individual access on an "as needed" basis and does not allow for access beyond what is necessary for the completion of individual duties. Specific operation and management of the LIMS system is outlined in LIMS Standard Operating Procedures that are maintained by the Laboratory.

7.5 Report Preparation

After all of the data has been entered into the LIMS, a draft copy of the final report is generated and the following items are reviewed by the Laboratory Manager or designee:

- Client name and address.
- Analytical results, units, and reportable figures.

- Inter-parametric relationships.
- Data reasonableness in respect to sample information

Following the review, if results are acceptable the Laboratory Manager, or designee makes the determination to generate a final report. The report is forwarded to a Project Manager who conducts a general review for completeness, signs the report, and releases it to the client. The draft copy of the report is placed in the client file for storage.

Final Reports are sent to clients through the U.S. Postal Service unless the client requests an electronic copy. Electronic copies are placed into Portable Document Format (pdf) or EXCEL spreadsheets and password protected prior to transmission.

7.5.1 Final Report Modifications

Once a laboratory final report is generated and sent to the client, it can only be modified under the following circumstances:

- A copy of the original unedited report is kept on file in the laboratory. In the case of electronic records and/or files, either a pdf is generated of the original report prior to editing, or a paper copy of the original report is placed in the project file.
- The reissued report must have a statement in the comments section that specifies the modification(s) and indicates the date of report reissue.
- A copy of the reissued report must be filed with the original report.

Data review of report modifications is conducted in accordance with Microseeps Standard Operating Procedure for Data Review and Validation (SOP-ADM 16).

7.6 Record Retention

A signature record of all employees is maintained in the Training Records Manual kept in the Quality Systems Coordinator's Office.

All raw data and reports for analytical projects are kept for a full five (5) years. After this time the records are destroyed. Records are kept onsite in a locked storage area and maintained in accordance with Microseeps Standard Operating Procedure for Document Control (SOP-ADM 05). The area is inspected monthly.

The LIMS database, which retains all electronic records that have been entered from the date of the LIMS inception, is backed up each day. The tape on which this backup is stored is maintained in a secure location off-site.

All critical data on personal computers is backed up on the internal network S drive. The network is backed up on tape and stored at a secure location off-site.

7.7 Service to the Client and Confidentiality

Clients are welcome to an on-site visit to Microseeps Laboratories in order to discuss client needs or requests, and also to monitor the laboratory's performance in relation to ongoing client projects. All efforts are made to maintain client confidentiality while providing service to other clients.

All client data, whether from privately owned or government organizations, and all correspondence is considered confidential information and shall not be released to anyone other than the client without the expressed written permission of the client. These transactions shall be handled by the Customer Service Office.

7.8 Records Dispensation

In the unlikely event that the laboratory transfers ownership or goes out of business, all clients will be notified in writing and requested to notify Microseeps concerning the dispensation of their records.

8.0 Measurement Traceability

Traceability of measurements and standards is insured in the laboratory by using balance calibration weights that are traceable to national standards, calibrating thermometers using an NIST calibrated thermometer, and by the use of certified standard solutions.

8.1 Reagents and Standards

The purity of the materials required in analytical chemistry varies with the type of analysis, the parameter being measured, and the sensitivity of the detection system. In general analytical reagent grade is satisfactory for most inorganic analyses. Other analyses, such as trace organic, may require special ultra-pure reagents. In cases were the method does not specify the purity of the reagent, it is intended that analytical reagent grade be used. The labels on the container are checked and the contents examined to verify that the purity of the reagents meets the needs of the particular method involved.

8.1.1 Reagent and Standard Preparation

Reagents are prepared and standardized with the utmost of care against reliable primary standards. They are re-standardized or prepared fresh as often as required by their stability as specified by method and other reference sources. Stock and working standard solutions are checked regularly for signs of deterioration.

Standard preparation procedures are specific to the analytical determination being made and are defined in detail in specific technical Standard Operating Procedures, regulatory methods, and in the laboratory's Standards Logbooks.

8.1.2 Reagent and Standard Labeling and Storage

Standard solutions are labeled with the compound name, lot number, preparation date, and expiration date. The analysts store reagents and solvents in a manner that prevents contamination and deterioration prior to their use. Standard solutions are stored in compatible containers.

Microseeps Standard Operating Procedure for Analytical Standards and Reference Materials (SOP-ADM 15) gives detailed instructions for handling, storing, labeling, and documenting standards and reference materials.

8.1.3 Standards Preparation Logbook

Standard Preparation Logbooks are issued as controlled documents to every analyst or laboratory in which they will be used. These logbooks are used to document standard preparation procedures, dates, lot numbers, concentrations, manufacturer, expiration date, and any other information that

may be necessary in order to re-create or track a particular standard. The compound or element name and/or formula are documented along with the final concentration or normality. The description of how reagents and standards are prepared may be referenced from a previous description if the exact procedure is used.

8.1.4 Traceability of Standards

The traceability of each purchased stock standard is easily accessible. Certificates of analysis of each standard are maintained in a binder in the laboratory until the standard is depleted or disposed. The certificate is then given to the quality assurance manager for archiving. The traceability of each laboratory prepared standard is entered into standard logbooks.

8.2 Thermometers

All of the laboratory thermometers in use are calibrated annually using a thermometer that is traceable to NIST. Thermometer calibration is outlined in Microseeps Standard Operating Procedure for Calibration of Thermometers (SOP-ADM 12).

8.3 Weights and Balances

All balances are calibrated before use with Class I NIST traceable weights that are calibrated annually. Balances are serviced and calibrated by an outside contractor annually. Additional information is outlined in Microseeps Standard Operating Procedures for Calibration of Weights and Balances (SOP-ADM 11).

9.0 Training Program

Training for laboratory personnel is accomplished at several levels. Areas for which training is conducted include new employee orientation, laboratory safety, specific task training, analytical procedure training, laboratory ethics, and other technical training courses as the need arises.

Analytical training, while addressed in general in this manual, is addressed specifically in the Standard Operating Procedure for Administering and Documenting Training in Laboratory Procedures and Instrumentation (SOP-ADM 02).

9.1 New Employee Orientation

Orientation is conducted to familiarize new employees with company policies, quality system procedures, facilities, coworkers, laboratory ethics, and laboratory safety. The Laboratory Manager is responsible for notifying the various departments of the start date of all new employees.

9.2 Laboratory Safety

Upon hire, each employee is required to read the laboratory's Chemical Hygiene Plan (safety manual), is issued a pair of safety glasses, and is instructed in basic laboratory safety requirements. Safety Meetings are mandatory for all employees and are held on a monthly basis by the Safety Coordinator. Documentation of safety training is updated and maintained by Microseeps' Safety Coordinator.

9.3 Task Training

Task training must be successfully completed for employees to perform the following tasks without direct supervision:

- Sample Receiving
- Bottle Preparation
- Customer Service
- Data Entry

Task training is conducted by the Department Manager of the department concerned. During this training, the trainer works closely with the trainee to ensure that all pertinent points of procedures are addressed. If the procedure is outlined in a Standard Operating Procedure (SOP), the trainee is charged with reading the SOP and the trainer will ensure that the training covers all aspects of the SOP. For training to be considered complete, proficiency in the task must be demonstrated to the trainer.

9.4 Analytical Method Training

Prior to conducting analysis on client samples, all analysts must demonstrate their proficiency through initial technical training. Analytical proficiency must be demonstrated annually thereafter. All analytical method training must be conducted by an analyst who is certified in the method for which training is required.

For individual analyst training in sample preparation and analysis, initial training and proficiency is demonstrated through the analysis of a set of 4 consecutively run mid-range standards or an Initial Demonstration of Proficiency (IDOP). When an analysis involves a work group (i.e. semivolatile preparation and analysis), a team approach to the IDOP applies. When each individual completes their part in the successful analysis of the IDOP, the analytical team is considered competent to conduct the analysis on client samples.

For continued demonstrations of capability for individuals and work groups, one of the following procedures may be used:

1) Another IDOP
2) Four consecutively run laboratory control samples that fall within laboratory/method acceptance criteria.
3) Acceptable analysis of Performance Evaluation samples.
4) Acceptable analysis of blind quality control sample.

Training documentation is maintained in the Quality Systems Office. See Exhibit 9-1 for a flow diagram of analytical method training. A Demonstration of Capability Certification Statement (Exhibit 9-2) shall be maintained for each method in which an employee is certified. This documentation is kept in the Employee Training Records in the Quality Systems Office.

9.5 Laboratory Ethics Training

All employees shall receive initial and annual ethics training. The training shall be conducted by Microseeps' management personnel. The training shall include the contents of the Ethics Program (see Appendix C) in its entirety, including employee and supervisory responsibilities, examples of unethical behavior, disciplinary action for unethical actions, and a means to report unethical actions.

Documentation of Ethics Program Training is kept in the Quality Systems Office. All employees are required to sign and date the Ethics and Data Integrity Agreement (see Exhibit 9-3). This form shall be placed in the employees personnel file.

9.6 Other Technical Training

Other types of technical training are conducted on an as-needed basis and may include training on new instruments, new procedures, or new equipment. Training may be conducted by Microseeps' employees

9.7 External Training

Employees are encouraged to continue their education through the use of symposia and seminars conducted by professional societies, regulatory agencies, and equipment manufacturers. These courses serve as one way for laboratory personnel to remain current on regulatory trends, analytical procedures, and advances in instrumentation. Documentation of external training will be added to the analysts' training records.

Exhibit 9-1

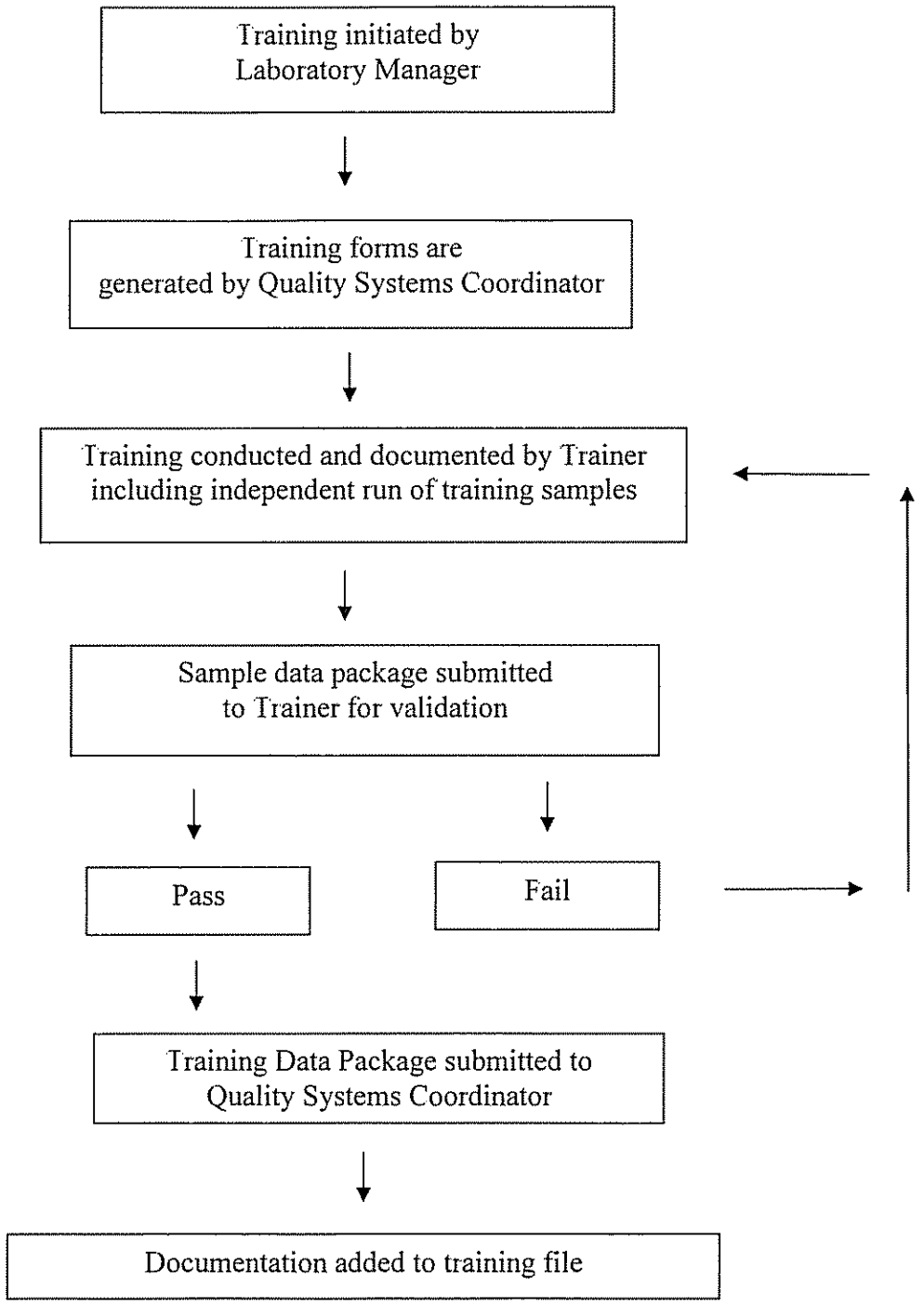

Training initiated by Laboratory Manager
Training forms are generated by Quality Systems Coordinator
Training conducted and documented by Trainer including independent run of training samples
Sample data package submitted to Trainer for validation
Pass
Fail
Training Data Package submitted to Quality Systems Coordinator
Documentation added to training file

Exhibit 9-2

Demonstration of Capability Certification Statement

Date: _____

Microseeps, Incorporated
220 William Pitt Way
Pittsburgh, PA 15238

Analyst Name: _____

Matrix: (laboratory pure water, soil, air, matrix spike)

Analyte (or group)	**Method Number**	**SOP #**	**Revision #**

I, the undersigned, CERTIFY that:

1. The analyst(s) identified above, using the cited test method (s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method was performed by the analyst(s) identified on this certification.

3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration of capability are true, accurate, complete, and self-explanatory.

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well-organized and available for review by authorized assessors.

_____ _____

Patrick McLoughlin, Ph.D. Date
Technical Director

Exhibit 9-3

Microseeps, Incorporated

ETHICS AND DATA INTEGRITY AGREEMENT

I, _____, state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at **Microseeps, Incorporated.**

I agree that in the performance of my duties at Microseeps:

I. I shall not intentionally report data values that are not the actual values obtained;

II. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

III. I shall not intentionally represent another individual's work as my own.

I agree to inform Microseeps of any accidental reporting of non-authentic data by myself in a timely manner.

I agree to inform Microseeps of any accidental or intentional reporting of non-authentic data by other employees.

I have read, acknowledge, and understand my personal ethical and legal responsibilities including potential punishments and penalties for improper, unethical, or illegal actions.

_____ _____

Signature Date

Appendix A

Key Personnel Job Descriptions

President and Chief Executive Officer

General Description: This exempt position is responsible for the overall administrative, technical, and financial direction of the corporation.

Educational Requirements: This position requires a graduate degree and/or fifteen or more years of experience in business management.

Functional Job Description

1. Performs the duties of the Chief Operational Officer as required.

2. Assumes financial responsibility and financial liability for the corporation.

3. Approves company policies and procedures.

4. Represents the company at trade shows.

5. Writes and presents research papers at environmental conferences.

6. Represents the corporation in negotiating major contracts.

7. Researches and considers the growth potential for future markets.

8. Keeping the company's focus on the technical edge of the environmental laboratory market.

9. Serves on the Board of Directors.

10. Committed to insure a framework is in place for the company to provide quality systems guidelines of the highest standard.

11. Other responsibilities as assigned by the Board of Directors.

Senior Vice President

General Description: This exempt managerial position is responsible for providing corporate management of the organization.

Educational Requirements: This position requires a graduate degree and/or ten or more years of experience in corporate management.

Functional Job Description

1. Administers and enforces company policies and procedures.

2. Produces plans and administrative procedures to maximize the efficiency of the corporation within established guidelines.

3. Oversees outsourced Human Resource, Information Technology, and Health and Insurance Benefits contracts.

4. Oversees Finance Department.

5. Maintains budgetary restraints for corporate profitability.

6. Supports the role of Quality Systems within the corporation.

7. Coordinates the administration of the Corporate Ethics Program and Policy.

8. Serves on the Executive Board of Directors.

9. This position has contractual liability for the corporation.

10. Other duties and responsibilities as assigned by the president.

Vice President of Sales and Marketing

General Description: This exempt managerial position is responsible for directing the activities of the Sales and Marketing Staff. This position is ultimately responsible for meeting sales goals and reports directly to the President.

Educational Requirements: This position requires a Bachelor's Degree in Business or a related discipline and a minimum of ten years of business management experience.

Functional Job Description

1. Provide leadership and direction for the Sales and Marketing Staff.

2. Propose the yearly Sales and Marketing Budget to the Executive Committee.

3. Set and communicate reasonable target sales goals for sales staff.

4. Hold sales staff accountable for meeting target sales.

5. Assist the President and Senior Vice-President in developing short and long term sales and marketing strategies.

6. Communicate staffing needs for all related positions so that the sales division can efficiently handle workloads.

7. Responsible for sales staff's code of conduct and disciplinary action.

8. Act as liaison with other departments to ensure interdepartmental communication.

9. Serves on the Executive Board of Directors.

10. Other duties and responsibilities as assigned by the President.

Vice President of Operations

General Description: This exempt managerial position is responsible for providing overall administration of the Operations Department.

Educational Requirements: This position requires a bachelor's degree and/or ten or more years of experience in operations management.

Functional Job Description

1. Oversee the duties and responsibilities of the department managers as required.

2. Administer and enforce company policies and procedures.

3. Develop administrative procedures to maximize the efficiency of operations within established company guidelines.

4. Oversee implementation of procedures to maximize the quality and efficiency of work performed.

5. Oversee effective subordinate development including but not limited to, training and career path development.

6. Maintains budgetary restraints for operational profitability and turnaround schedules.

7. Ensures that all technical assignments are carried out consistently within applicable professional and company quality standards.

8. Maximizes business opportunities within the current client base.

9. Supports the role of Quality Systems within the corporation.

10. Serves on the Board of Directors.

11. Other duties and responsibilities as assigned by the president.

Laboratory Manager

General Description: This exempt managerial position is responsible for overseeing the daily management of the laboratory operations and operations' staff. This position reports to the Vice President of Operations.

Educational Requirements: This position requires a Bachelor's Degree in Chemistry or a related field with a minimum of ten years of experience.

Functional Job Description

1. Provide supervision and direction to all Operations Department personnel.

2. Manage the size of the Operations Department efficiently in accordance with available workload and communicate staffing requirements to the Vice President Operations.

3. Responsible for laboratory code of conduct and staff disciplinary action.

4. Responsible for scheduling work and laboratory personnel to ensure efficient use of time and resources.

5. Assist the Vice President of Operations in developing short and long-range plans for laboratory improvement and growth.

6. Responsible for ensuring that the highest degree of technical quality possible is represented in outgoing final analytical data reports.

7. Ensure that laboratory work assignments are coordinated with work assignments of other groups in the company.

8. Represent the Operations Department in weekly meetings with other managers and Executive Staff.

9. Delegate operational responsibility and duties, as seen fit, to the Assistant Laboratory Manager.

10. Responsible for client liaison when specific questions arise on technique or procedure. Work closely with Technical Services and Customer Service regarding this function.

11. Control operational supplies purchasing and maintain the day to day laboratory budget.

12. Work in conjunction with the Manager of Technical Services to provide input and support for instrumentation needs.

13. Coordinate with the Quality Systems Manager to develop and implement Standard Operating Procedures.

14. Support and enforce the specific methods, policies, and procedures outlined in Microseeps' Standard Operating Procedures and Quality Systems Manual.

15. Assist in the writing and review of laboratory Standard Operating Procedures.

16. Ensure control charts, control limits, MDLs, and PQLs are updated in the LIMS as required.

17. Responsible for the administration of the Chemical Hygiene Plan within the laboratory.

18. Perform other duties and responsibilities as assigned by the Vice President of Operations.

Quality Systems Manager

General Description: This exempt managerial position carries the responsibility for providing leadership and direction to ensure that the laboratory function at the highest level of quality possible in accordance with regulatory and certification requirements. The term Technical Director is use synonymously with the Quality Systems Officer.

Educational Requirements: This position requires a Bachelor's degree in chemistry or a related discipline and a minimum of ten years of laboratory experience.

Functional Job Description

1. Provide leadership and direction for the laboratory's Quality Systems Program.

2. Provide supervision and delegate tasks to the Quality Systems Coordinator.

3. Keep current on regulatory requirements to ensure that Standard Operating Procedures for analyses are in compliance with applicable regulations.

4. Conduct laboratory quality audits according to the annual schedule.

5. Serve as liaison between Microseeps and Regulatory Agencies.

6. Provide leadership and direction for developing and maintaining all Laboratory Standard Operating Procedures and the Quality Systems Manual.

7. Work with the Quality Systems Coordinator and the Laboratory Manager to discern and correct issues leading to poor quality and, if deemed necessary stop unsatisfactory work or prevent reporting of unjustifiable results.

8. Serve as client contact for questions that cannot be answered by the Customer Service Department or the Manager of Technical Services.

9. Other duties as assigned by the President and Chief Operating Officer.

Manager of Research and Development

General Description: The Manager of Research and Development is responsible for directing and managing an ongoing research and development program that insures the laboratory is ready to respond to leading edge technology needs.

Educational Requirements: This position requires a graduate degree in chemistry or a related discipline and a minimum of ten years of laboratory experience.

Functional Job Description

1. Provide assistance with interpretation of client data needs and data reports in cooperation with the customer service department.

2. Assist clients in defining data needs, constructing sampling plans and interpreting data reports via non-binding suggestions and selected references to published documents.

3. Prepare and present at least two presentations per year at seminars, workshops, or conferences.

4. Develop written material for the use of customer service personnel to provide client assistance and education.

5. Identify emerging analytical needs and upon agreement with the Executive Board of Directors, develop conceptual models for analytical methods to support those emerging client needs.

6. Develop conceptual analytical methods into routine procedures for bench chemists to implement.

7. Ensure that new method development is fully documented by compiling a complete package of experimental data in a form that is organized, summarized, and that can be validated.

8. When new analytical methods have become routine procedures, transfer responsibility to the Manager of Technical Services.

9. Establish and maintain an awareness of the literature and the community of scientists and engineers that drive the market identified by the President and Chief Executive Officer.

10. Other duties as assigned by the President and Chief Executive Officer.

Manager Customer Service

General Description: This exempt managerial position is responsible for directing the activities of the Customer Service Staff, Bottle Preparation, and Sample Courier. This position is ultimately responsible for ensuring customer service satisfaction. This position reports directly to the Vice President of Operations.

Educational Requirements: This position requires a Bachelor's Degree and a minimum of four years of Customer Service experience.

Functional Job Description

1. Provide leadership and direction for the Customer Service Office, Bottle Preparation, Sample Courier, and EDD Specialist.

2. Assist the Vice President Sales and Marketing in developing short and long-term sales and marketing strategies.

3. Communicate staffing needs for all related positions so that the divisions can efficiently handle workloads to better ensure customer satisfaction.

4. Responsible for customer service code of conduct and disciplinary action.

5. Perform all of the functions of a Project Manager/Customer Service Representative.

6. Provide review of contracts.

7. Oversee purchasing for Bottle Preparation.

8. Act as liaison with other departments to ensure interdepartmental communication.

9. Ensure customer complaints are resolved to the customer's satisfaction.

10. Represent Customer Service at weekly Managers' Meetings.

11. Communicate the importance of Customer Service within the company.

12. Review client projects and Lab Project Summary Sheets after projects are logged in, on a daily basis.

13. Other duties and responsibilities as assigned by the Vice President of Operations.

Manager of Technical Services

General Description: This exempt managerial position is responsible for overseeing the use of the laboratory facilities and instrumentation, and implementing new method development. This position reports to the Vice President of Operations.

Educational Requirements: This position requires a Bachelor's Degree in Chemistry or a related field with a minimum of ten years of experience.

Functional Job Description

1. Provide assistance for the technical review of client requests for chemical analysis.

2. Oversee response to daily technical problems and questions on unusual projects and/or samples at the request of the Laboratory Manager.

3. Optimize analytical methods to meet client needs.

4. Implement development of SW-846 methods not currently in use in the laboratory at the direction of the Vice President of Operations.

5. Fully document all aspects of method development including assembling a complete package of all developmental/experimental data in a manner that is organized and ready for validation.

6. Evaluate the technical aspects and justify the cost of newly proposed instrumentation or existing instrument upgrades.

7 Assist in writing and reviewing Standard Operating Procedures as needed.

8. Responsible for maintenance and repair of equipment and apparatus being used at Microseeps.

9. Ensure new MDL studies are run and submitted to the Quality Systems Department when instrument repairs or parts replacement affects method sensitivity.

10. Provide support for equipment and data interfacing to the LIMS.

11. Upon recommendation of the Laboratory Manager, provide support in updating the test packages in the LIMS.

12. Recommend upgrades to laboratory apparatus to meet method requirements and to maintain Microseeps position as a leading-edge technology provider.

13. Reviews and participates in new hardware and software purchases for technical instrumentation.

14. Provide training recommendations for staff and conduct technical in-house training.

15. Provide ongoing facility (plant) recommendations.

16. Any other duties as assigned by the Vice President of Operations.

Manager of Field Operations

General Description: This exempt managerial position is responsible for directing the activities of the Field Services personnel. This position reports to the Vice President of Operations.

Educational Requirements: This position requires a Bachelor's Degree and a minimum of two years of management/field experience.

Functional Job Description

1. Provide leadership and direction for the Field Services personnel.
2. Propose the yearly Field Services Budget to the Executive Committee.
3. Schedule field projects.
4. Provide written specifications for projects to field personnel to ensure they are adequately prepared for each project.
5. Responsible for field personnel code of conduct and disciplinary action.
6. Approve purchases in support of Field Operations.
7. Act as liaison with other departments to ensure interdepartmental communication.
8. Represent Field Services at Manager Meetings.
9. Responsible for field services vehicle fleet.
10. Any other duties as assigned by the Vice President of Operations.

Quality Systems Coordinator

General Description: This exempt position is responsible for assisting the Quality Systems Manager to ensure that the Quality Systems Policies and Procedures are implemented in accordance with the Quality Systems Manual and Microseeps Standard Operating Procedures. This position reports to the Quality Systems Manager.

Educational Requirements: This position requires a Bachelor's Degree and 2 years of laboratory experience.

Functional Job Description

1. Administratively responsible for the Quality Systems Program as directed by the Quality Systems Manager.

2. Prepare an annual schedule of laboratory audits.

3. Prepare audit reports and necessary corrective action to the Laboratory Manager.

4. Follow up on all corrective action reports until appropriate action has been taken.

5. Establish a schedule, order supplies, coordinate, and submit final analytical data for performance evaluation studies.

6. Maintain, control, and update the Quality Systems Manual and all of Microseeps' Standard Operating Procedures.

7. Ensure new Operation's Department employees receive required training and orientation and maintain the documentation of the training.

8. Ensure appropriate studies are conducted when necessary i.e. Initial Demonstrations of Proficiency and MDL's.

9. Calibrate all laboratory thermometers once a year.

10. Annually send out scale weights, radiation screening instrument, and NIST thermometer for calibration.

11. Coordinate annual inspections for certifications of balances and fume hoods.

12. Other duties as assigned by the Quality Systems Manager.

Manager of Information Technology

General Description: This exempt position is responsible for managing IT personnel, computer network, LIMS, servers, and IT-related projects and tasks.

Educational Requirements: This position requires a bachelor's degree in Information Science or the equivalent and two years of LIMS administration experience. This position also requires knowledge and skills for managing network information systems. Windows Server, IIS, and TCP/IP experience required. Experience with a scientific oriented organization is preferred.

Functional Job Description

1. Supervise personnel assigned to the IT Department.

2. Submit and manage IT Department budget.

3. Manage all corporate network, server, and desktop/laptop information systems.

4. Manage and document software and information systems-related service licensing.

5. Create and manage user and email accounts.

6. Provide daily and weekly data backups of critical systems and provide data recovery in case of failure or disaster.

9. Provide desktop support services to end users.

10. Develop, and maintain Standard Operating Procedures and other documentation relating to information systems.

11. Provide LIMS administration and management.

12. Provide network and systems security and virus protection.

13. Provide technology recommendations and project management for enhancement and simplification of corporate operations.

14. Coordinate with vendors for information system purchases and repairs as necessary.

15. Communicate LIMS, network, and server updates and issues to appropriate parties.

16. Other projects as assigned by the Vice President of Operations.

Assistant Laboratory Manager

General Description: This exempt managerial position provides assistance to the Laboratory Manager. This position reports to the Laboratory Manager.

Educational Requirements: Bachelor's Degree in a physical science or a related field and five years of experience.

Functional Job Description

1. Manage work flow through laboratory by scheduling laboratory projects and work assignments, and has approval authority for expedited projects at the discretion of the Laboratory Manager.

2. Conduct weekly Lead Analyst meetings to determine the most efficient allocation of resources within the laboratory to meet the current workload.

3. Evaluate laboratory operating procedures and makes recommendations for improvements.

4. Responsible for evaluation of analytical data after entry into the LIMS.

5. Conduct data review.

6. Maintain Operations Department vacation calendar.

7. Generate draft reports, final reports, and preliminary invoices.

8. Communicates with customer service concerning operational issues that have a direct impact on specific methods and/or turnaround time that may affect in-house or repeat projects.

9. File and maintain files for all raw data and final analytical reports in a manner that is easily retrievable.

10. Obtain subcontract laboratory approval and scheduling.

11. Other duties as assigned by the Laboratory Manager.

Procurement Coordinator

General Description: This exempt position is responsible for all aspects of sample custody from sample receipt to storage until final disposal. This position reports directly to the Laboratory Manager.

Education Requirements: This position requires a high school diploma and two years of laboratory experience. A basic knowledge of chemistry and laboratory analytical procedures is required.

Functional Job Description

1. Receive and inspect samples and sample containers and sign appropriate documents according the Standard Operating Procedure for Sample Receiving and the Quality Systems Manual.

2. Record all necessary information on chain of custody.

3. In the event of any discrepancies or non-conformance issues with the above procedures, immediately complete a non-conformance form and submit it to customer service.

4. Notify Project Manager upon receipt of client samples when requested.

5. Accurately log samples into LIMS ensuring that all required fields are completed for the level of analysis required.

6. Label all sample containers.

7. Initiate transfer of samples as soon after receipt as possible to appropriate storage areas, ensuring that all samples are maintained at the appropriate temperature at all times.

8. Notify Lead Analysts immediately upon receipt of samples with short holding times. Complete non-conformance form for all samples that do not get picked up by analysts within the specified holding time.

9. Control and monitors access to and storage of samples in secure storage.

10. Purchase, track, receive, and distribute supplies for the Operations Department.

11. Performs other duties and responsibilities as assigned by the Laboratory Manager.

Lead Analyst

General Description: This exempt supervisory position assists in the daily supervision and training of staff analysts and technicians. This position reports directly to the Laboratory Manager.

Educational Requirements: This position requires a Bachelor of Science Degree in Chemistry or a related field and five years of related work experience, or a combination of college coursework in chemistry with a minimum of eight years of related work experience.

Functional Job Description

1. Conduct routine day to day technical training and supervision of analysts including proper Quality Assurance and Quality Control implementation and training.

2. Ensure that samples arriving with short holding times are analyzed within the applicable time frame.

3. Schedule analysis to efficiently meet holding times, and expedited and priority project schedules. Follow up with analysts to ensure turnaround times are met.

4. Review analytical data for completeness and accuracy. Institute corrective action when quality is found to be below the standards specified in the Standard Operating Procedures.

5. Properly collect, label, and store hazardous waste streams that are generated from the laboratory under the position's direct supervision. Coordinate with the Waste Coordinator for waste pick-up.

6. Ensure that the following logbooks are completed as required by Standard Operating Procedures: Temperature Logs, Balance Logs, Standards Preparation Logs, Run Logs, Sample Preparation Logs and Instrument Maintenance Logs.

7. Work with the Manager of Technical Systems to ensure preventative maintenance is conducted according to the maintenance schedule. Conduct daily preventative maintenance and minor repairs if able.

8. Keep laboratory stocked with supplies by submitting purchase orders to Procurement Coordinator in a timely manner.

9. Assure that all personnel under direct supervision practice quality control procedures in accordance with the Laboratory Quality Systems Manual and specific Standard Operating Procedures.

10. Enforce Health and Safety Procedures in the laboratory in accordance with the Chemical Hygiene Plan.

11. Work with the Health and Safety Officer to help correct suspected unsafe practices, situations, or working spaces.

12. Responsible for ensuring that all workspaces are maintained in a clean and well-organized fashion.

13. Performs other duties and responsibilities as assigned by the Laboratory Manager.

Staff or Laboratory Analyst

General Description: This exempt position is responsible for the timely analysis and custody of client samples. Job responsibilities cover a broad range of duties and responsibilities depending on employee education and experience. This position reports directly to the Lead Analyst.

Educational Requirements: This position covers a range of educational levels from a high-school education with on-the-job training to a Ph.D. in Chemistry or a related field. Analysts must pass Initial Demonstrations of Proficiency for every analytical method before independent analysis can be conducted.

Physical Requirements: This position requires the ability to lift 20 pounds and the ability to stand for extended periods of time.

Functional Job Description

1. In matters concerning order and priority of analysis, this position is accountable to, and will take direction from the Lead Analyst.

2. Keep all workspaces neat, clean, and organized.

3. Obtain samples from Sample Receiving as directed by the Lead Analyst.

4. Observe internal chain of custody requirements for all samples taken from Sample Receiving.

5. Ensure all aliquots analyzed are representative of the entire sample.

6. Analyze samples according to Microseeps Standard Operating Procedures (SOP) and or the applicable Standard or EPA Methods.

7. Observe and practice applicable quality control procedures in accordance with the Laboratory Quality Systems Manual and specific SOPs. This includes analyzing required quality control samples, ensuring results are within specified acceptance limits, and initiating corrective action when results are outside of acceptable parameters.

8. Analyze samples arriving with short holding times within the applicable time frame.

9. Review peer analytical data for completeness and accuracy where possible.

10. Responsible for making accurate entries into logbooks.

11. Conduct equipment preventative maintenance according to the Equipment Maintenance SOP.

12. Maintain supply inventory and notify lead analyst when laboratory supplies are needed.

13. Comply with all Health and Safety Procedures outlined in the Chemical Hygiene Plan.

14. Work with the Health and Safety Officer to help correct suspected unsafe practices, situations, or working spaces.

15. Identify any problems with samples, equipment, or procedures that will affect the integrity of analysis and report them to the Lead Analyst.

16. Performs other duties and responsibilities as assigned by the Lead Analyst or the Laboratory Manager.

Project Manager/Customer Service Representative

General Description: This exempt position is responsible for marketing, inside sales, and project management. This position reports to the Manager of Customer Service.

Educational Requirements: This position requires a minimum of five years of customer service experience in the laboratory, environmental, or chemistry field.

Functional Job Description

1. Generates quotations utilizing the LIMS.
2. Serves as the liaison between clients and the technical departments.
3. Provides supervision and direction to Bottle Preparation and Courier.
4. Takes bottle orders from clients.
5. Enter projects into the LIMS.
6. Manage projects as required according to client contracts.
7. Signs final data reports acknowledging that the reports are being sent.
8. Contact clients as required by Non-Conformance Forms.
9. Arrange rush and special analytical projects with Assistant Laboratory Manager.
10. Participate in conferences, trade shows, and other marketing activities.
11. Responsible for updating client databases with current information.
12. Provide initial review of contracts.
13. Serve as Microseeps representative to the client and as such, conduct themselves with the highest standard of ethics, is responsive to clients' needs, and portrays a level of professionalism for which Microseeps is known.
14. Create, generate, and disseminate company information to clients as requested.
15. Review invoices for accuracy.
16. Other duties as assigned by the Manager of Customer Service.

Field Services Technician

General Description: Field Services Personnel are non-exempt positions that are responsible for direct push sampling, other on-site field services, and other duties as specified below. Field Services Personnel report directly to the Manager of Field Operations.

Educational Requirements: Field Service Personnel must be certified in 29 CFR 1910.120, OSHA's Basic Health and Safety 40 hr. Hazardous Waste Operations and Emergency Response Course.

Functional Job Description:

1. Operate Geoprobe® direct push sampling equipment to collect soil, soil vapor, and water samples.
2. Hand collect water, soil, and soil vapor samples.
3. Install temporary and permanent monitoring wells (piezometers).
4. Operate grout pump to inject HRC and ORC.
5. Ensure the Geoprobe and trailer are stocked with adequate supplies for each field project.
6. Accurately complete Daily Field Activity Logs and Sample Collection Logs for each Field Project.
7. Inventory and order supplies for field projects according to established guidelines.
8. Represent Microseeps to the client in a professional manner.
9. Travel when necessary for out of town field projects.
10. Clean and maintain field vehicles, equipment, and garage.
11. Accurately complete vehicle log for each field project.
12. Assist Customer Service Department with Courier duties.
13. Assist Customer Service Department with Bottle Preparation duties.
14. Other duties as assigned by the Manager of Field Services.

Bottle Preparation Technician

General Description: This non-exempt position consists of up to forty hours of work per week in completing bottle preparation activities. This position reports to the Customer Service Office.

Educational Requirements: High School Diploma with knowledge in basic chemistry. Computer skills are required, as well as, competencies in basic math, reading, and writing.

Physical Requirements: The position requires a maximum lifting capacity of 40 pounds with the ability to lift and carry large and bulky objects. The job skills also require a full range of motion for bending over and straightening.

Functional Job Description

1. Properly assemble, prepare, preserve, package and ship orders following Company Standard Operating Procedures in a cost effective and timely manner.

2. Inventory, order, track, and stock all necessary bottle preparation supplies.

3. Track, maintain, and fill all standing bottle orders for pick-up, delivery, and shipment.

4. Keep accurate records and files of standing orders, supply orders, and end of month inventories.

5. Track client packages that were lost or late via the telephone or by using the available software. (When given a computer/telephone)

6. Practice open communication in all instances with the customer service department in order to assure clients get the best possible service.

7. Maintain a neat, clean, and organized work and storage area.

8. Wear safety glasses while in bottle preparation room, and other protective clothing such as gloves and smocks when using chemicals, handling samples, and cleaning coolers.

9. Assist courier in sample collection, pick-up, or delivery when necessary.

10. Clean and dry all coolers that arrive prior to placing them in storage.

11. Make stock preservatives.

12. Other duties as designated by the Manager of Customer Service.

Appendix B

References

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U.S. Environmental Protection Agency, 1983, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

Standard Methods for the Examination of Water and Wastewater, 20th ed., 1998.

National Environmental Laboratory Accreditation Conference, Quality Systems National Environmental Laboratory Accreditation Program, Rev. 16, July 12, 2002, Chapter 5.

Appendix C

Microseeps Ethics Program

Microseeps, Incorporated

Ethics Program

Approved by: _____ Date: _____
Thomas W. Hill

Reviewed by: _____ Date: _____

1.0 Introduction

At Microseeps, we are committed to operating with integrity. We value the integrity of the individual and the institution. We value honesty and trust in the way we treat one another and in the way we meet our commitments. Consistent with our commitment to integrity, our Ethics Program is based on values. For the program to succeed, each employee must use his or her own values to make decisions that reflect well on the employee and on the company.

This Program will encompass defining improper, unethical, and illegal actions, outline responsibilities, and ways in which the laboratory can prevent and/or detect these types of actions. This Program shall identify specific examples of improper, unethical, or illegal actions and establish potential punishments and penalties for the same. This Program shall include initial and annual ethics training.

The Statement of Ethical Responsibility (see Exhibit 1) shall be read and signed by every Microseeps' employee. This Program shall designate one individual to whom company personnel can report improper, unethical, or illegal practices.

2.0 Definitions

Improper Actions: Deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional.

Unethical or Illegal Actions: The deliberate falsification of analytical or quality assurance results, where failed method or contractual requirements are made to appear acceptable (also known as laboratory fraud).

3.0 Responsibility

3.1 Laboratory Managers and Supervisors

The Laboratory management and supervisors at all levels are responsible for:

- Implementing the Ethics Program
- Providing clear guidelines on matters of everyday conduct to all employees.
- Stressing to all employees the need for a commitment in word and deed to the ethics policy and practices.
- Demonstrating their own commitment to the ethics policy and practices in the management of their area of responsibility and the activities of all employees under their supervision.

- Maintaining a workplace environment that encourages frank and open communication, free from the fear of reprisal, concerning the upholding of the ethics policy and practices.
- Distributing the Ethics Program to all employees.
- Developing and presenting training to ensure that all employees understand the laboratory's ethics principles and practices.
- Provide continuing counsel on laboratory practices, policies, rules, and regulations to any employee who seeks assistance.
- Maintaining working conditions supportive of employee responsibilities.
- Enforcing compliance with the ethics policy and practices.
- Recognizing employees who make exemplary efforts to implement and uphold the ethics policy and practices.
- Reviewing and investigating all allegations of wrongdoing.
- Ensuring that all current and new employees under their supervision have received a copy of the Ethics Policy and are trained in its meaning and applications.
- Reviewing the level of Ethics Policy knowledge and understanding of the employees under their supervision.

3.2 Employees

All employees are responsible for:

- Reviewing regularly their knowledge and understanding of the ethics policy and practices.
- Upholding the ethics policy and practices as demonstrated in their daily conduct.
- Contributing to a workplace environment that is conducive to the maintenance of the ethics policy and practices in daily activities.
- Seeking help when the proper course of action is unclear or unknown to them.
- Remaining alert and sensitive to situations that could result in actions by any employee that are improper, illegal, unethical, or otherwise in violation of the ethics policy and practices.
- Counseling fellow employees when it appears that they are in danger of violating the ethics policy and practices.
- Reporting violations of the ethics policy and practices to their supervisor, or higher-level management.
- Writing thorough case narratives explaining why analytical data may or may not be useful due to anything in the analytical process that may have been wholly or partially deficient.

4.0 Prevention and Detection

A key element in preventing fraud is the adoption of an ethics policy that is strictly enforced. Having employees that understand the difference between a mistake and improper behavior and

that are trained to make ethical decisions is one of the best prevention strategies. A strongly reinforced ethics-training program is one of the key cornerstones to an effective total ethical process.

4.1 Examples of Unethical Actions

There are a number of laboratory practices, which may constitute fraud. The following are some examples:

- Falsification of results to meet method requirements.
- Reporting of results without analyses to support (i.e. dry-labbing).
- Selective exclusion of data to meet QC criteria (initial calibration points dropped without technical or statistical justification).
- Misrepresentation of laboratory performance by presenting calibration data or QC limits within data reports that are not linked to the data set reported.
- Notation of matrix interference as a basis for exceeding acceptance limits (typically without implementing corrective actions) in interference-free matrices (e.g. method blanks or laboratory control samples).
- Unwarranted manipulation of computer software (e.g. improper background subtraction to meet ion abundance criteria for GC/MS tuning, chromatographic baseline manipulations).
- Improper alteration of analytical conditions (e.g. modifying EM voltage, changing GC temperature program to shorter analytical run time) from standard analysis to sample analysis.
- Misrepresentation of QC samples (e.g. adding surrogates after sample extraction, omitting sample preparation steps for QC samples, over-spiking or under-spiking).
- Reporting results from the analysis on one sample for those of another.

4.2 Prevention and Detection

The Quality Inquiry Form (QIF) can be used as a means to report improper, unethical, or illegal actions. The QIF should be completed and submitted anonymously (if desired) to the Technical Director. An immediate investigation shall be conducted by a member of Microseeps' management to ascertain if a violation did occur.

Other means of prevention and detection include an Internal Audit Program, an ethics-training program, and a requirement for analyst notation and sign-off on manual integration changes to data.

4.3 Disciplinary Action for Unethical Actions

Because the implications of unethical actions are substantial, swift and decisive disciplinary action shall be carried out for violations of the Ethics Policy. If an investigation reveals knowing and willful violation of laboratory ethics by an employee, that employee shall be terminated. Other violations can result in disciplinary actions that range from verbal warnings to written warnings with time off. The severity of the discipline shall match the severity of the offense. All ethics violations shall be documented in employee's personnel files.

5.0 Training

All employees shall receive initial and annual ethics training. The training shall be conducted by Microseeps' management personnel. The training shall include the contents of this policy in its entirety, employee and supervisory responsibilities, examples of unethical behavior, disciplinary action for unethical actions, and a means to report unethical actions.

5.1 Ethics Training Documentation

Training documentation shall be maintained in the Quality Systems Office. All employees are required to sign and date Exhibit 1, the Ethics, and Data Integrity Agreement. This form shall be placed in the employees personnel file.

5.2 Training Frequency

Initial training shall be conducted within two weeks of all initial hires and annually thereafter.

Microseeps, Incorporated

ETHICS AND DATA INTEGRITY AGREEMENT

I, _____, state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at **Microseeps, Incorporated.**

I agree that in the performance of my duties at Microseeps:

I. I shall not intentionally report data values that are not the actual values obtained;

II. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

III. I shall not intentionally represent another individual's work as my own.

I agree to inform Microseeps of any accidental reporting of non-authentic data by myself in a timely manner.

I agree to inform Microseeps of any accidental or intentional reporting of non-authentic data by other employees.

I have read, acknowledge, and understand my personal ethical and legal responsibilities including potential punishments and penalties for improper, unethical, or illegal actions.

_____ _____

Signature Date

Appendix K
Standard Operating Procedures (SOPs) 1 - 16

Appendix K
Standard Operating Procedures (SOPs) 1 - 16

Appendix L
TestAmerica North Canton - Laboratory Quality Manual

(Document on CD-ROM)

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

Laboratory Quality Manual for

TestAmerica North Canton

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Approved by:

7/23/07
Laboratory Director — Date
Opal Davis-Johnson

7/19/07
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Revision 5.0
July 16, 2007
Effective Date 7/23/07
Controlled Copy Number: _____

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Purpose and Scope

The purpose of this Laboratory Quality Manual (LQM) is to describe the implementation the TestAmerica Laboratories Quality System at the TestAmerica North Canton laboratory. The LQM is written within the guidelines of the TestAmerica Quality Management Plan (QMP), which applies to all TestAmerica laboratories. The organization of this LQM is based on the "EPA Requirements for Quality Management Plans" (EPA QA/R-2, August 1994). This LQM outlines specific policies, organization, responsibilities, and activities required to assure high quality laboratory services. The LQM also fulfills the requirements of our clients, government agencies, and NELAC to document the laboratory Quality System.

This LQM contains references to other essential TestAmerica quality documents. The company-wide QMP, TestAmerica North Canton LQM, and referenced policies and SOPs are interrelated. Together they provide an integrated quality foundation that meets the objectives of the TestAmerica Quality Assurance Policy, as stated in Section 1.2.

The requirements set forth in this document are applicable to all employees at the TestAmerica North Canton laboratory. The policies and practices described here are presented as minimum guidelines only. Based on good scientific judgment, more rigorous requirements may be applied by laboratory employees. Specific requirements delineated in project plans may supersede general quality requirements described in this manual. One such project requirement, OhioVAP, is listed below:

- Quality Manual Sections 3.3, 3.4, 3.5, and Table 3.4-1: Per OhioVAP OAC Rule 374-300-4(B) – TestAmerica North Canton will retain project records for all samples performed under the OhioVAP program for a period of ten years. After ten years, TestAmerica North Canton must notify the Director of intent to destroy records.

- Quality Manual Section 8.2.1: Per OhioVAP OAC Rule 3745-300-04(1)(6) – TestAmerica North Canton will not update SOPs and Quality Manuals associated with OhioVAP projects unless they have been reviewed and approved by the agency.

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1.0 Management Commitment and Organization

1.1 TestAmerica Vision and Mission Statement

Vision Statement

TestAmerica will be the industry leader for environmental testing and data deliverables.

Mission Statement

TestAmerica is committed to providing exceptional client service, highest quality legally defensible data, and the most comprehensive range of capabilities in the environmental testing industry.

1.2 TestAmerica Quality Assurance Policy

It is TestAmerica's policy to:

- provide high quality, consistent, and objective environmental testing services that meet all relevant federal, state, and municipal regulatory requirements;
- generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use;
- provide TestAmerica clients with the highest level of professionalism and the best service practices in the industry;
- build continuous improvement mechanisms into all laboratory administration, and managerial activities; and
- maintain a working environment that fosters open communication with both clients and staff.

1.3 TestAmerica Management Statement of Commitment to Quality Assurance

TestAmerica management is committed to providing the highest quality data and the best service in the environmental testing industry. To ensure that the data produced and reported by TestAmerica meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, TestAmerica maintains a Quality System that is clear, effective, well communicated, and supported at all levels in the company.

1.4 Ethics, Waste, Fraud and Abuse

Establishing and maintaining a high ethical standard is an important element of a Quality System. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times, TestAmerica has established an Ethics Agreement (see Figure 1.4-1). Ethics is also a major component of the TestAmerica QA training program (see Section 4 for details). A central tenant is that

management must consistently convey the message to analysts that financial pressures can never be allowed to compromise the quality of work. See the following policies for further details on specific policies related to this section:

LQM Section 6 – Computer Hardware and Software
T-Q-005 – Recording Laboratory Observations and Raw Data
QA-010 – Maintaining Time Integrity
S-Q-004 – Acceptable Manual Integration Practices
P-T-001 – Selection of Calibration Points

Figure 1.4-1 TestAmerica Example Ethics Agreement

TestAmerica
EMPLOYEE ETHICS STATEMENT

I understand that TestAmerica is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:

- I will not intentionally report data values that are inconsistent with the actual values observed or measured;
- I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;
- I will not intentionally misrepresent another individual's work as my own or represent my own work as someone else's;
- I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers; I shall not modify data (either sample or QC data) unless the modification can be technically justified through a measurable analytical process, such as one deemed acceptable to the laboratory's Standard Operating Procedures, Quality Assurance Manual or Technical Director. All such modifications must be clearly and thoroughly documented in the appropriate laboratory notebooks/worksheets and/or raw data and include my initials or signature and date.
- I shall not make false statements to, or seek to otherwise deceive, members of Management or their representatives, agents, or clients/customers. I will not, through acts of commission, omission, erasure, or destruction, improperly report measurement standards, quality control data, test results or conclusions.
- I shall not compare or disclose results for any Performance Testing (PT) sample, or other similar QA or QC requirements, with any employee of any other laboratory, including any other TestAmerica laboratory, prior to the required submission date of the results to the person, organization, or entity supplying the PT sample.
- I shall immediately inform my supervisor or other member of management regarding any intentional or unintentional reporting of my own inauthentic data. Such report shall be given both orally and in writing to the supervisor or other member of management contacted and to the local Quality Assurance Officer/Manager. The Quality Assurance Officer/Manager will initial and date the information and return a copy to me; I shall not condone any accidental or intentional reporting of inauthentic data by other employees and will immediately report its occurrence. If I have actual knowledge of such acts committed by any other employees, and I do not report such information to designated members of management, it shall be considered as serious as if I personally committed the offense. Accordingly, in that event, I understand that I may be subject to immediate termination of employment.
- I understand that if any supervisor, manager, or representative of TestAmerica management instructs, requests, or directs me to perform nay of the aforementioned improper laboratory practices, or if I am in doubt or uncertain as to whether or not such laboratory practices are proper, I will not comply. In fact, I must report such event to all appropriate members of Management including, but not limited to, the Lab Director, all supervisors and managers with direct line reporting relationship between me and the Lab Director, and the local Quality Assurance representative, excluding such individuals who participated in such perceived improper instruction, request, or directive. In addition, I may contact Corporate Quality Assurance / Ethics Compliance Officer(s) for assistance.
- I understand the critical importance of accurately reporting data, measurements, and results, whether initially requested by a client, or retained by TestAmerica and submitted to a client at a later date, or

retained by TestAmerica for subsequent internal use;

- I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the TestAmerica family of companies.
- I shall not accept gifts of a value that would adversely influence judgement.
- I shall avoid conflicts of interest and report any potential conflicts to the management (e.g., employment or consulting with competitors, clients, or vendors);
- I shall not participate in unfair competition practices (e.g., slandering competitors, collusion with other labs to restrict others from bidding on projects);
- I shall not misrepresent certifications and status of certifications to clients or regulators;
- I shall not intentionally discharge wastes illegally down the drain or onto the ground.
- I understand that any attempt by management or an employee to circumvent these policies will be subject to disciplinary action.

As a TestAmerica employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

I understand that if my job includes supervisory responsibilities, I shall not instruct, request, or direct any subordinate to perform any laboratory practice which is unethical or improper. Also, I shall not discourage, intimidate, or inhibit an employee who may choose to appropriately appeal my supervisory instruction, request, or directive which the employee perceives to be improper, nor retaliate against those who do.

The Ethics Policy has been explained to me by my Supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE _____ Date _____

Supervisor/Trainer: _____ Date _____

1.5 Organizational Structure and Relationships

TestAmerica North Canton is a local operating unit of TestAmerica Laboratories, Inc.

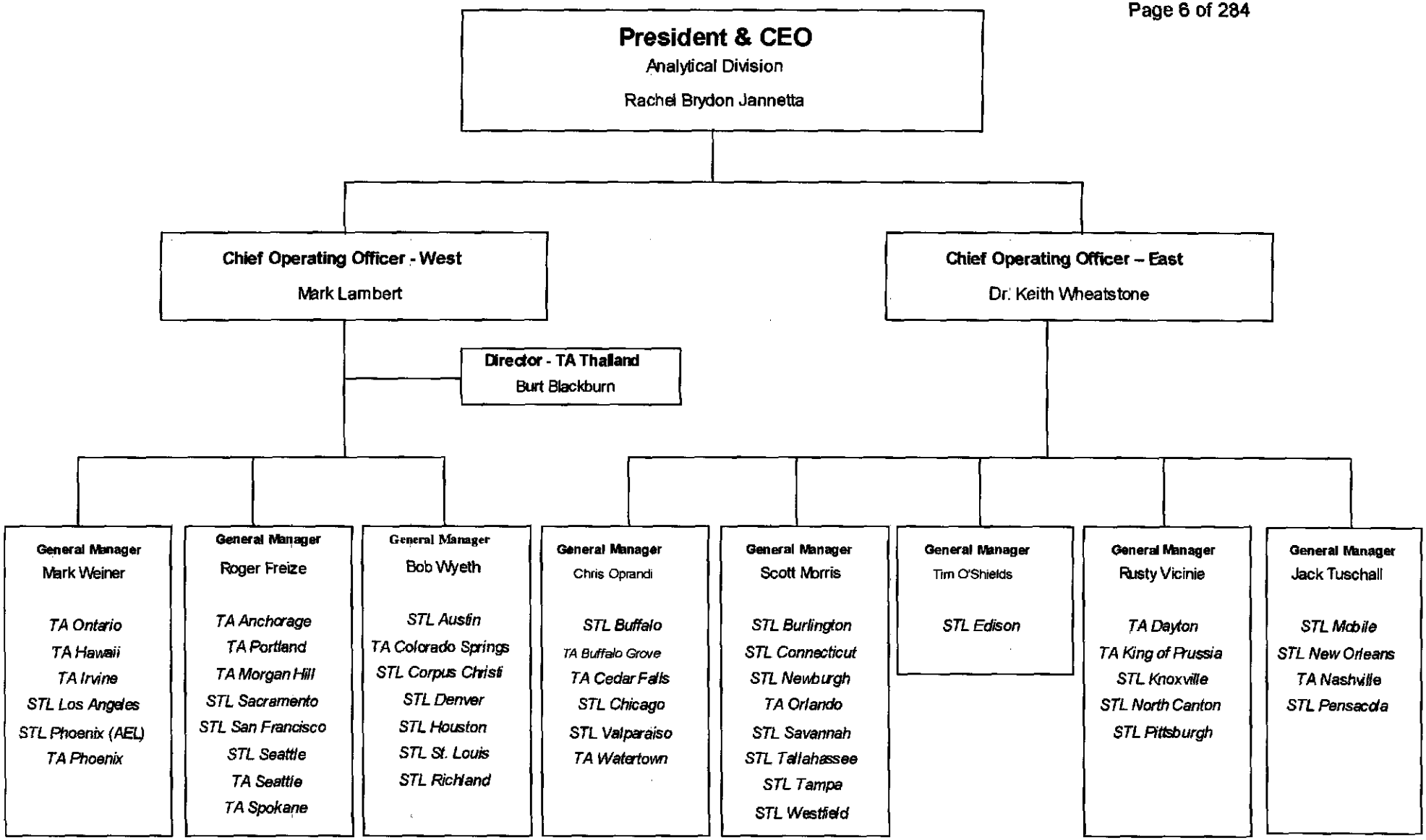
The organizational structure for Severn Trent Laboratories, Inc. is presented in Figure 1.5-1. The responsibilities and authorities of the members of the TestAmerica corporate staff employees are described in the TestAmerica QMP

TestAmerica North Canton has day-to-day independent operational authority that is overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality Assurance, etc.). The TestAmerica North Canton laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for TestAmerica North Canton is presented in Figure 1.5-2. A list of key TestAmerica North Canton personnel is provided in Figure 1.5-3. The lab maintains Job Descriptions that contain general job responsibilities for all laboratory employees. The following section outlines responsibilities and authorities for all employees of the TestAmerica North Canton laboratory, as they relate to quality management.

The TestAmerica North Canton QA Manager (QAM) is independent from day-to-day laboratory operations, has no direct analytical testing responsibilities, and is free from financial and other undue pressures that might adversely affect the quality of work. The QAM, a key member of the laboratory's management team, has direct access to the Corporate Quality Assurance Director on all matters involving quality. The QAM is available to any lab employee to resolve quality or ethical issues. The QAM, if required, has the authority to cease operations adversely affecting the validity or integrity of the analytical data.

TestAmerica

Figure 1.5-1 TestAmerica Organizational Structure



TestAmerica North Canton Laboratory Organization

Figure 1.5-2 TestAmerica North Canton Organizational Structure

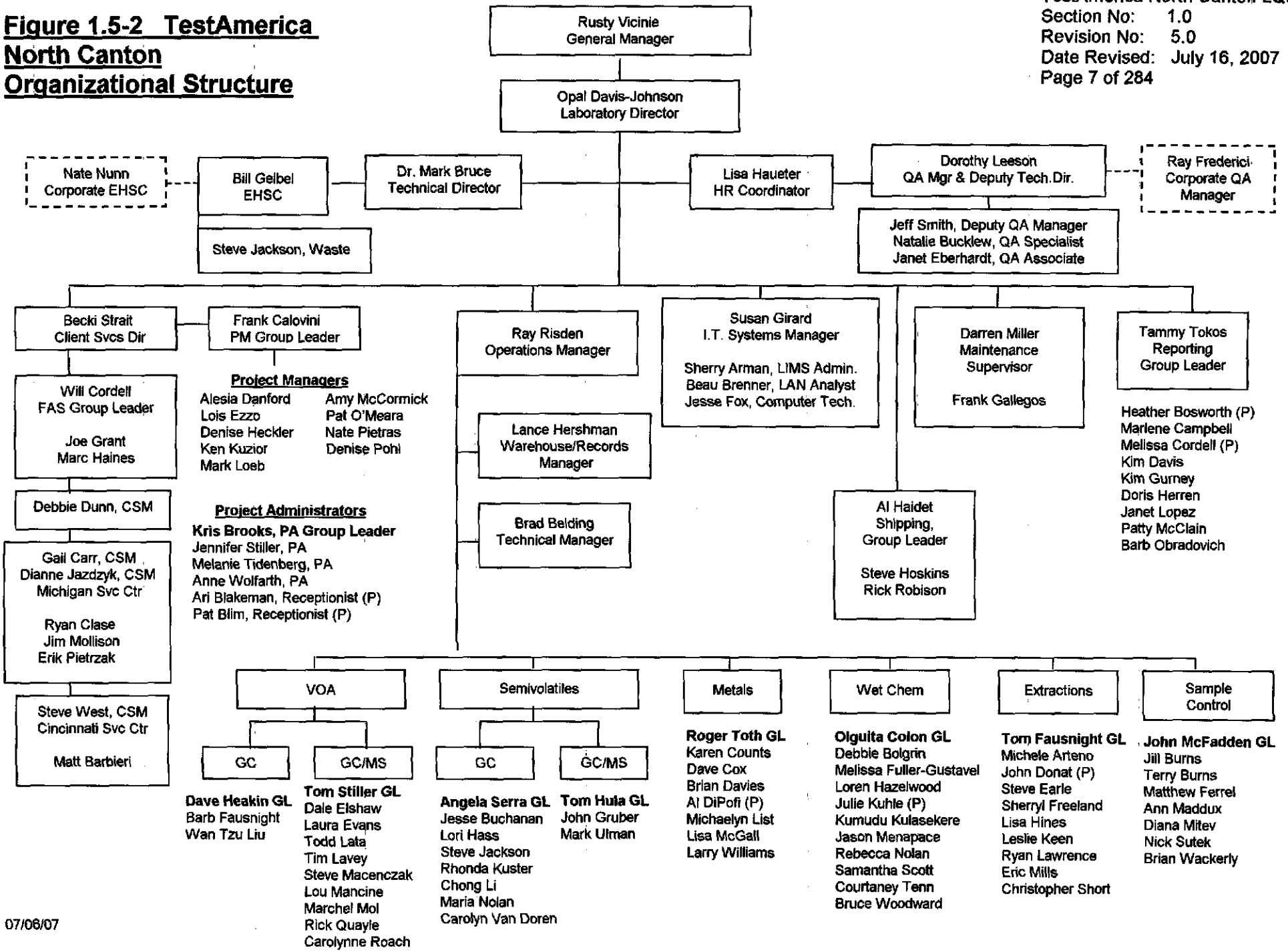


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Figure 1.5-3 TestAmerica North Canton Key Personnel

Name	Position	Degree/Discipline	Years Experience
Rusty Vicinie	General Manager, NE Region	BS Chemistry and Biology	24
Opal Davis-Johnson	Laboratory Director	BA Chemistry	21
Raymond Risden	Operations Manager	BS Biology (Chemistry Minor)	13
Dorothy Leeson	Quality Assurance Manager	BS Chemistry	17
Mark Bruce	Technical Director	PhD Analytical Chemistry	22
Rebecca Strait	Client Services Director	MBA International Business	19
Deborah Dunn	Customer Service Manager	BA Biology	18
Gail Carr	Customer Service Manager	BS Biology	22
Dianne Jazdzyk	Customer Service Manager	MBA Business Administration	25
Steve West	Customer Service Manager	Work Experience	12
Bill Geibel	Envir. Health & Safety Coordinator	MS Env. Science & Management BS Pre-Medicine	3
Tammy Tokos	Reporting Group Leader	AS Computer Programming	22
Olguita Colon	General Chemistry Supervisor	BS Natural Resources Policy	8
Will Cordell	Field Analytical Supervisor	BS Env Sci & Natural Resources	15
Tom Fausnight	Extractions Supervisor	Work Experience	18
Al Haidet	Shipping Supervisor	Work Experience	29
Dave Heakin	GC Volatiles Supervisor	BS Chemistry	19
Tom Hula	GC/MS Semivolatiles Supervisor	BA Chemistry	20
Steve Jackson	Waste Management Supervisor	AA Environmental Science	16
John McFadden	Sample Control Supervisor	BS Biology	3
Darren Miller	Maintenance Supervisor	Work Experience	27
Roger Toth	Metals Supervisor	BS Environmental Sciences	21
Angela Serra	GC Semivolatiles Supervisor	BS Chemical Engineering	7
Tom Stiller	GC/MS Volatiles Supervisor	BS Biology	19
Frank Calovini	Project Management Group Leader	BS Microbiology	21
Alesia Danford	Project Management	Nursing – LPN	23
Lois Ezzo	Project Management	BA Biology	18
Denise Heckler	Project Management	BS Chemistry	19
Ken Kuzior	Project Management	BS Chemistry	21
Mark Loeb	Project Management	BS Chemistry	20
Amy McCormick	Project Management	Work Experience	17
Pat O'Meara	Project Management	BA Biology	17
Nate Pietras	Project Management	AS Environmental Technology	9
Denise Pohl	Project Management	BA Biology	28

1.6 Quality Organization

All personnel are responsible for quality, which includes complying with all QA/QC requirements that pertain to their organizational/technical function.

1.6.1 Quality Assurance Manager

- Reports directly to the Laboratory Director and, for all QA matters, to the Corporate QA Director to maintain independence of QA oversight
- Responsible for the implementing and communicating the QMP
- Maintains, approves, and implements the LQM
- Has joint signature authority, with the Laboratory Director and Technical Manager for approval of quality documents, e.g., LQM, policies, and SOPs
- Directs controlled distribution of laboratory quality documents
- Provides Quality System training to all new personnel
- Reviews and approves documentation of analyst training records
- Serves as a focal point for QA and QC issues, reviews corrective actions and recommends resolution for recurring nonconformances within the laboratory
- Assists in maintaining regulatory analytical compliance, including maintaining certifications, and in this regard has signature authority for laboratory quality documents
- Monitors data quality measures via statistical methods to verify that the laboratory routinely meets stated quality goals
- Performs systems, data, contract compliance, and surveillance audits.
- Hosts external audits conducted by outside agencies
- Responsible for approving quality control reference data changes in the LIMS
- Oversees the selection, review, and approval of analytical subcontractors
- Prepares monthly QA Reports to management describing significant quality events
- Has the final authority to accept or reject data and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data
- Responsible for implementing internal/external audit findings corrective actions

1.6.2 Laboratory Director

- Reports directly to the Regional General Manager
- Responsible for implementation and adherence by lab staff to the TestAmerica QMP, TestAmerica North Canton LQM and all policies and procedures within the laboratory
- Has signature authority for LQM, policies, SOPs, and contracts (as detailed in TestAmerica policy)
- Assesses the effectiveness of the QMP and LQM within the operation
- Maintains adequate trained staff documented on organization charts
- Responsible for implementing internal/external audit findings corrective actions

1.6.3 Operations Manager/Laboratory Supervisor

- Reports directly to the Laboratory Director
- Supervises daily activities of the Operational Groups
- Schedules analytical operations
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Supervises maintenance of instruments and scheduling of repairs
- Works with the Project Managers and Group Leaders to assure the requirements of projects are met in a timely manner
- Supervises daily activities of the Sample Control Group.

1.6.4 Laboratory Technical Director

- Reports directly to Laboratory Director
- Responsible for the technical operation of the laboratory
- Has joint signature authority for LQM, policies, SOPs, and training records
- Performs technical training in area(s) of expertise
- Interfaces with management on technical needs and solving day-to-day technical issues
- Investigates technical issues related to projects as directed by QA
- Evaluates new methods, technical proposals, and statements of work
- Certifies technical laboratory personnel based on education and background to ensure that staff have demonstrated capability in the activities for which they are responsible by reviewing and signing analyst demonstrations.
- Performs other tasks as required by NELAC.

The Technical Director meets the requirements specified in the Section 4.1.1.1 of the NELAC standards.

1.6.5 Project Management Group Leader

- Reports directly to the Manager of Client Services
- Supervises daily activities of the Project Management and Administrative Groups
- Works with the Operations Manager and/or Group/Team Leaders to ensure the requirements of projects are met in a timely manner

1.6.6 Manager of Client Services

- Reports directly to Laboratory Director
- Supervises Field Analytical Services Group
- Supervises Service Centers
- Supervises daily activities of the customer
- Supervises Service Managers

1.6.7 *Customer Service Managers (CSMs)*

- Reports directly to the Manager of Client Services
- Defines customer requirements through project definition
- Assesses and assures customer satisfaction
- Provides feedback to management on changing customer needs
- Brings together resources necessary to ensure customer satisfaction.

1.6.8 *Project Manager*

- Reports directly to the Project Manager Group Leader
- Monitors analytical and QA project requirements for a specified project
- Acts as a liaison between the client and the laboratory staff
- Communicates project-specific requirements to all parties involved
- Assists the laboratory staff with interpretation of work plans, contracts, and QAPP requirements
- Oversees project data packages for completeness and compliance to client needs
- Has signature authority for final reports
- Keeps the laboratory and client informed of project status
- Together with the QA Manager, approves customer requested variances to methods and to standard laboratory protocols
- Monitors, reviews, and evaluates the progress and performance of projects
- Reports client inquiries involving data quality issues or data acceptability to the facility QA Manager and to the operations staff
- Conducts project reviews to assess the laboratory's performance in meeting customer requirements
- Prepares reissue requests for project data
- Responsible for meeting quality requirements.

1.6.9 *Group Leader*

- Reports directly to the Operations Manager
- Supervises daily activities of analyses within the group
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Evaluates instrument performance and supervises the calibration, preventive maintenance, and scheduling of repairs
- Oversees or performs review and approval of all analytical data
- Reports nonconformances to the appropriate managers
- Responsible for generation of SOPs for their section
- Responsible for meeting quality requirements.

1.6.10 Analyst

- Performs analytical methods and data recording in accordance with documented procedures
- Performs and documents calibration and preventive maintenance
- Performs data processing and data review procedures
- Reports nonconformances to the Supervisor/Manager and QA Manager
- Ensures sample and data integrity by adhering to internal chain-of-custody procedures
- Responsible for meeting quality requirements defined in this LQM and other supporting QA procedures.

1.6.11 Sample Custodian

- Ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody
- Reports nonconformances associated with condition-upon-receipt of samples
- Logs samples into the LIMS
- Ensures that all samples are stored in the proper environment
- Assists Environmental Health and Safety staff with sample disposal
- Responsible for meeting quality requirements.

1.6.12 Report Production Staff

- Accurately generates and compiles analytical reports and associated deliverables for delivery to the client
- Responsible for meeting quality requirements
- Produce as needed reports that meet the NELAC requirements.

2.0 Quality System and Description

2.1 Objectives of the TestAmerica Quality System

The Quality System is a set of management principles, objectives, policies, responsibilities, and implementation plans at the organizational and project-specific levels. The goal of the TestAmerica Quality System is to ensure that business operations are conducted with the highest level of professionalism in the industry. To achieve this goal, it is necessary to provide TestAmerica clients with not only scientifically sound, well documented, and regulatory compliant data, but also to ensure that TestAmerica provides the highest quality service available in the industry. A well-structured and well-communicated Quality System is essential in meeting this goal. The TestAmerica Quality System is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

2.2 Structure of the TestAmerica Quality System

At the highest level, the TestAmerica Quality Management Plan (QMP) is the basis for the TestAmerica Quality System. The QMP provides the guidance under which all TestAmerica facilities conduct their operations. This Laboratory Quality Manual (LQM) describes the implementation of the Quality System at the TestAmerica North Canton laboratory. This LQM and the series of associated quality documents define the organization, project-specific principles, goals, controls, and tools of the Quality System as it is applied at this laboratory. The Quality System as described in this LQM demonstrates the commitment to accepted laboratory practices by TestAmerica North Canton.

2.3 Quality Assurance and Quality Controls

Quality Assurance (QA) is defined as the system of activities that ensures the quality of a process, product, or service. Quality controls (QC) are the tools used to monitor and regulate the desired type and quality of product. The QA activities and QC controls employed in TestAmerica North Canton are defined in the following quality documents.

2.4 Quality Documents

The TestAmerica Quality System is developed from the reference documents shown in Table 2.4-1. The review and control of the TestAmerica North Canton documents described in the following subsections is described in Section 3 of this LQM. A cross-reference of the LQM to NELAC requirements quality manuals is presented in Table 2.4-2.

2.4.1 TestAmerica Quality Management Plan (QMP)

The requirements set forth in the QMP are applicable to all TestAmerica facilities. The policies and practices outlined in the QMP are minimum guidelines only. Requirements that are more rigorous may be applied for specific client or regulatory programs.

2.4.2 TestAmerica Company-Wide Policies

TestAmerica Laboratories has certain policies that apply company-wide. These policies are consistent with the QMP, and set forth requirements that all TestAmerica facilities are to follow.

2.4.3 Laboratory Quality Manual (LQM)

This TestAmerica North Canton LQM along with the associated policies and SOPs, provides the criteria and specifications for the generation of environmental analytical data. The LQM provides QC criteria for standard procedures, facility-specific instrumentation, and reporting.

2.4.4 TestAmerica North Canton Quality Policy Documents

Quality policies are referenced throughout the LQM that provide further detail to specific requirements of the QMP and LQM. These policies describe general quality objectives and guidelines in effect for this facility, rather than the details of specific practices. Refer to Table 2.4-3 for a list of the TestAmerica North Canton signature-required approvals. Table 2.4-4 lists the frequency of review for the documents.

2.4.5 Standard Operating Procedures

Standard Operating Procedures (SOPs) describe step-by-step instructions for performing a method or activity. In addition, there are SOPs that relate to other support services performed in the laboratory. Details of SOP format and document control are described in SOP NC-QA-0027, Preparation and Management of Standard Operating Procedures, and Policy S-Q-001, Official Document Control and Archive. SOPs that are actively used in this laboratory are listed in Table 8.2-2. SOPs are living documents and may supersede some requirements in this document.

2.4.6 Quality Assurance Project or Program Plans (QAPPs)

Regulations and contracts may contain QA requirements that are different from those described in this LQM. To address unique project requirements, Quality Assurance Project Plans (QAPPs) may be prepared and implemented. The requirements documented in a QAPP, as agreed to by TestAmerica North Canton, take precedence over the LQM for that project. Typical specifications contained in a QAPP or similar documentation include:

- New or modified testing methods
- Unique QC logic
- Special requirements for equipment use and maintenance
- Special handling due to safety considerations
- Project-specific detection and reporting limits

- Project-specific accuracy and precision limits or the statistical treatment of data
- Additional or unique documentation or records management requirements

2.4.6.1 Quality Assurance Summary

Quality Assurance Summaries (QAS), or equivalent as outlined in the quote, are used to distill client-specific requirements typically documented in project QA plans onto a concise format, highlighting the requirements that are different than the laboratory standard practice. The summary describes for each project the required quality control samples, batching schemes, flagging conventions, deliverables, or other special client requests that may differ from routine laboratory operations. The QAS or equivalent is disseminated to laboratory operations by the Project Manager or Quality Assurance Manager to document client or program specific requirements.

2.4.7 Other Documents

Other documents that can affect the quality program may include the Corporate Safety Manual, memos, guidance documents, work instructions, and periodic management assessment reports. These documents may further define or guide the implementation of quality standards at TestAmerica, but shall not conflict with the LQM or diminish the effectiveness of the Quality System.

3.0 Document Control and Records Management

3.1 Objectives for Control of Quality Documents and Records

Quality Documents - The quality documents discussed in Section 2 of the LQM define the framework of the TestAmerica Quality System. Control and security of these documents are necessary to ensure that all staff have access to current policies and procedures at all times, to ensure that all changes to the policies and procedures are properly reviewed, to ensure that the history of use of documents can be reconstructed, and to ensure that confidential information is not improperly distributed. The system described in this section is designed to accomplish these objectives.

Vital Records – Vital records are the documents that provide objective evidence of the performance of a process or observations of an item. Records management ensures that results produced by the laboratory are scientifically and legally defensible, and ensures that project events can be reconstructed. Confidentially of the records and records retention requirements are discussed in this section.

3.2 Document Control Procedures

Unambiguous identification of a controlled document is maintained by identification of the following items in the document header:

- document title,
- unique document number,
- revision number,
- revision date,
- effective or implementation date, and number of pages

Controlled documents are marked as such, and the QA department keeps records of document distribution. Controlled distribution may be achieved by either electronic or hardcopy means. The effective date is the date when controlled copies are distributed. Controlled documents must be available in the immediate areas where the related work is performed. Details of the numbering system, required format, and restrictions for uncontrolled distribution of documents are in SOP NC-QA-0027, Preparation and Management of Standard Operating Procedures, and TestAmerica Corporate SOP S-Q-001, Official Document Control and Archive.

3.3 Document Review, Approval, and Revision

Controlled quality documents are authorized by the Laboratory Director, the Technical Manager, and the QA Manager. They indicate their authorization by signing the cover page of the document. TestAmerica North Canton quality documents and the individuals responsible for reviewing the documents are listed in Tables 2.4.3 and 2.4.4. In addition to periodic review and revision, quality documents must be revised when a procedure or activity changes in a significant manner. Amendments to documents must be reviewed and approved by the same parties approving the original document, distributed in a controlled manner, and clearly indicated in the document. Obsolete versions of documents are removed from service when new revisions are issued. The QA Department maintains a

record of history of use of all documents based on the effective date. For further details see SOP NC-QA-0027, Preparation and Management of Standard Operating Procedures, and SOP S-Q-001, Official Document Control and Archive.

3.4 Records Management

Records may be either hardcopy or electronic copies. It is not required to maintain both if they are properly secured and are complete and true copies. The record keeping system allows for reconstruction of all laboratory activities that produced the analytical results. The history of the sample is readily understood through the documentation. This includes:

- chain-of-custody records, including intra-laboratory and inter-laboratory transfers of samples;
- records identifying the personnel involved in sampling, preparation, calibration, and testing;
- observations, calculations, and derived data;
- information relating to laboratory facilities, equipment, analytical test methods, and related laboratory activities (e.g., sample preparation, standards preparation, and data verification);
- original records clearly identifying all subcontracted test data, and
- a copy of the final test report

Requirements for data recording are described in Policy T-Q-005, Recording Laboratory Observations and Raw Data. Details concerning control of electronic records are given inSOP NC-QA-0019, Records Information Management, and P-L-001, Record Retention. The types of vital records maintained are listed in Table 3.4-1.

TestAmerica North Canton utilizes a controlled access public drive to post controlled electronic QA records such as MDLs, SOPs, Laboratory or QA forms, Certifications, etc. Only QA personnel have write access to the QA public drives labeled, "QAQC" and "QA". The records are maintained and backed-up according to the record retention policy. The processes for producing these electronic (or hardcopy) records are described in their specific QA Policy, SOP, and/or the TestAmerica QMP.

3.5 Document and Record Storage, Retention, and Disposal

It is the policy of TestAmerica North Canton that company records will be available to meet business needs and comply with all applicable legal record retention and disposition requirements. TestAmerica North Canton retains copies of records in a manner that allows prompt retrieval of documents and records for inspection purposes. In accordance with NELAC, all quality documents and records are stored for at least five years. Other types of records have different retention requirement, refer to Table 3.4-1 for details.

Specific projects and regulatory programs have longer record retention requirements than the standard TestAmerica record retention time. Refer to QMP Table 6 for a listing of examples of special program requirements. The inventory sheet accompanying the stored records must include disposal instructions, which take into account any special requirements, and who to contact for authorization prior to destroying the data.

When records, as contained in files, are transferred to a records storage area or off-site storage area, they shall be placed in suitable containers and include an inventory sheet (hard copy or electronic) prepared by the person submitting the records. The contents of each container shall be compared to the inventory sheet and labeled. If there are any discrepancies, the container and inventory sheet shall be returned to the person who prepared the box for correction. Archives are indexed such that records are accessible on a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Backup copies of electronic media are stored in off-site archive facilities or controlled access electronic safes, and are protected against deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented. Further details of the laboratory's document and records archiving process are described in SOP NC-QA-0019, Records Information Management.

If the laboratory transfers ownership, vital records will be transferred to the new owner. If the laboratory goes out of business, vital records will be transferred to another operating TestAmerica laboratory or to our clients.

3.6 Data Confidentiality

Data and sample materials provided by the client or at the client's request, and the results obtained by TestAmerica, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay TestAmerica for all services rendered or is otherwise in breach of the terms and conditions set forth in the TestAmerica and client contract) subject to any disclosure required by law or legal process. TestAmerica reports, and the data and information provided therein, are for the exclusive use and benefit of client, and are not released to a third party without written consent from the client.

In some cases the client may identify projects requiring confidentiality due to national security. Information concerning these projects will be limited only to those TestAmerica North Canton associates with a need to know.

The audit reports supplied by federal, state, and local regulatory agencies are public information and can be released without written consent of those agencies. However, specific client audits are confidential and must be approved by the client before releasing them to a third party.

4.0 Staff Qualification, Orientation and Training

All activities performed by TestAmerica North Canton shall be accomplished by qualified personnel. Each staff member must have the combination of experience and education needed to demonstrate the required knowledge for his or her position. Each must also have an appropriate general knowledge of laboratory operations, test methods, quality assurance and quality control procedures, and records management. Minimum training requirements are shown in Figure 4-1. SOP CORP-QA-0013 describes details of the training process and documentation. The Corporate Safety Manual describes details for health and safety training.

4.1 Qualifications

TestAmerica maintains job descriptions for all positions. These job descriptions specify the minimum qualifications for education and experience, knowledge and skills, which are necessary to perform at a satisfactory level. Qualifications of professional staff are documented by resumes that include academic credentials, employment history, experience, and professional registrations. A copy of each person's resume is maintained in an electronic file, and is readily available for inspection.

4.2 Orientation and Technical Training

Each new staff member shall receive orientation in quality, ethics, and in health and safety. Each new staff member shall be supervised in their assigned duties by their supervisor or a knowledgeable individual designated by the supervisor. The ability and authorization to perform independently shall be documented in the training files, as described below, with technical duties approved by the Group Leader, Technical Director, or designee.

4.2.1 Quality Assurance (QA) Orientation

Each new staff member will receive a QA orientation. The QA Manager or designee will conduct this orientation within one month of the new employee's first day on the job. The orientation will, at a minimum, include the following topics:

- TestAmerica Quality System and hierarchy of quality documents (QMP, LQM, policies, and SOPs)
- Key elements of the LQM and the Quality Control Policy (QA-003)
- Introduction to the nonconformance memo (NCM) system and corrective action procedures
- Proper data recording practices
- TestAmerica Ethics Agreement, including the potential consequences of unethical behavior
- Role of the QA department.

The QA orientation will be documented on a training record, which is signed by the trainee. The documentation will be placed in the employee's training file.

4.2.2 Quality Training

Continued training in the mission and goals of the QMP and LQM shall be provided annually. These may be done in a single session or divided into separate sessions conducted at different times throughout the year. Formal training sessions are conducted and documented by the QA Manager or designee. In addition, each lab staff member shall read and document their awareness of the quality documents related to his or her position.

4.2.3 Health and Safety, Orientation and Training

Each new employee, contract worker, or working visitor is required to go through health and safety orientation and training as described in the Corporate Safety Manual. The Health and Safety Coordinator must conduct the orientation as soon as possible after the individual reports to work and before chemicals are handled. More comprehensive health and training, both initial and on going, must be completed at the frequency given in the Corporate Safety Manual.

4.3 Training Files

Each active TestAmerica North Canton staff member has an individual training file maintained by the QA Manager or designee. This file can be documented on paper forms or in a database. The following sections shall be included in the training files at a minimum:

- Resume - containing hardcopy or a reference to the electronic file
- Quality Assurance - containing documentation of QA/QC orientation and training completed
- Health and Safety - orientation and training documents
- Technical Proficiency - initial and on-going demonstrations of proficiency, one-on-one training, training courses or workshops on specific equipment or analytical methods is documented in this file.

Other types of records to be included in the training file include work place regulatory compliance training, and professional development courses. Signed Ethic Agreements are located in the employee's training files under the control of the QA Department. The exact contents will vary depending upon a person's job function and tenure with the company. Details of requirements for training records and the approval process are given in SOP CORP-QA-0013.

4.4 Technical Proficiency Training

All new personnel are required to demonstrate competency in performing a particular method by successfully completing a Demonstration of Capability (DOC) before conducting analysis independently on client samples. On-going proficiency must be demonstrated annually.

DOCs are most commonly performed by analysis of four replicate QC check samples. Results of successive LCS analyses can be used to fulfill the DOC requirement. As required by the referenced method, the accuracy and precision, measured as average recovery and standard deviation (using n-1 population), of the four replicates are calculated and compared to the method limits or against current laboratory limits if multi-laboratory method acceptance limits are not specified. Single-blind proficiency samples,

other NELAC acceptable proficiency samples, and Method Detection Limit (MDL) studies can be utilized to document proficiency. The DOC Certification documentation must be signed by the Technical Director and the Quality Assurance Manager and filed in the employee's training file (see example in CORP-QA-0013). The DOC Certification documentation must include a statement that the individual has read, understood, and agreed to perform the most recent version of the test procedure. In procedures such as %Solids, Color, Dissolved oxygen, Ignitability etc., where spiking is not an option and for which quality control samples are not readily available, the proficiency can be demonstrated by analyzing a duplicate sample provided the RPD≤10%.

Figure 4-1 Employee Minimum Training Requirements

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Initial training before start of production work.	All
	Additional training as specified in the CSM	As required
Quality Assurance	Orientation within one month of hire date	All
	Annual QA program training	All
Technical Proficiency	Initial demonstration prior to unsupervised method performance	Technical staff
	Annual on-going demonstration	Technical staff

5.0 Procurement of Supplies and Services

Controlling the quality of supplies and services is necessary to ensure that TestAmerica North Canton provides high quality analytical services to our clients. The TestAmerica procurement program requires:

- assurance that purchased items and services meet requirements set by TestAmerica North Canton and perform as expected
- definitions and descriptions of the levels of documentation required for applicable technical and administrative procurement functions
- maintenance of records of all suppliers from whom we obtain services or supplies required for our analytical testing

Additional information is contained in TestAmerica Policy, P-Pu-007, "Vendor Selection Procedure".

5.1 Selection of Vendors

Materials and supplies are purchased from approved vendors. Prospective vendors are selected based upon criteria appropriate to the materials or supplies provided. Policy TestAmerica /PG-001 "Procurement and Contracts" details the process used. For national vendors and contracts, the vendor is selected by the TestAmerica Procurement Director through a competitive bidding process, strategic business alliance or negotiated vendor partnership. Potential vendors are required to complete a vendor acceptance application and are evaluated on the following criteria, as appropriate:

- the vendor's history of providing identical or similar products that perform satisfactorily in actual use
- the vendor's service record and ability to provide a complete product line and commensurate service
- the vendor's ability to administer inventory at the TestAmerica North Canton facility through an inventory management system that will ensure correct stocking levels as well as shelf-life tracking
- objective evaluation of the vendor's current quality records, supported by documentation
- results of audits by TestAmerica of the vendor's technical and quality capabilities

Vendors that provide measuring equipment, solvents, chemical standards, instrument service contracts, or subcontracted laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items.

5.2 Controlling Quality of Purchased Items

The quality of equipment, reagents, solvents, chemical standards, gases, and laboratory containers used in analyses must be of known quality so that their effect upon analytical results can be defined. These quality specifications are derived from analytical method requirements, project-specific requirements, and defined national standards for analytical testing. Quality specifications of materials are described in analytical SOPs. These quality

specifications shall be included or referenced in the purchasing documents for the items being purchased. This includes specifications for the purity of standards, reagents, or chemicals, and technical specifications for accuracy and precision (e.g., Class A volumetric glassware). Reference to a catalogue number, model, lot number, or chemical grade is sufficient.

The Laboratory Director or designee has the responsibility for approving purchase orders. The section supervisors or designees are responsible for ensuring that the requested quality of materials ordered matches those received, for verifying that material storage is properly maintained and for removing materials from use when shelf life has expired.

5.2.1 Evaluation of Off-the-Shelf Items

For items that are used regularly by TestAmerica North Canton where no unique requirements or specifications exist, the items may be purchased off-the-shelf. These items are ordered from the supplier on the basis of specifications set forth in the supplier's published product description. These may include items such as glassware, filter paper, pipettes, and chromatography columns. The items are evaluated as a function of the standard analytical process.

5.2.2 Evaluation of Instruments

Evaluation of instruments purchased shall be conducted according to an acceptance-testing plan. The acceptance testing plan may be defined by the vendor or the method demonstration requirements specified in the laboratory analytical SOPs. Acceptance criteria may include instrument reliability, sensitivity, stability, selectivity, accuracy, precision, and ability to interface with existing computer systems.

5.2.3 Evaluation of Critical Solvents and Acids

TestAmerica conducts additional evaluations for certain solvents and chemical reagents where our criteria for purity are more stringent than the vendor's. Key solvents and reagents are subject to chemical analysis on a lot-by-lot basis before they are put into use. They are tested at one of the TestAmerica laboratories, and the chemical test results are evaluated by a designated quality representative. Solvents or reagents must meet the specifications given in SOP S-T-001, Quality Testing of Solvents and Acids, prior to release and use by TestAmerica Laboratories. These are listed in Table 5.2-3.

5.2.4 Evaluation of Chemical Standards or Standard Reference Materials

Where available chemical standards will be traceable to the National Institute of Standards Technology (NIST) or an equivalent source. This is largely limited to physical and inorganic chemical standards. If NIST traceability is not commercially available, commercially certified materials shall be used, which are then tested for accuracy before reporting data. Details of the testing procedures and documentation are described in the laboratory SOP. Standards must be received with a certification report from the vendor with information such as purity/concentration, traceability, lot number, expiration date, preparation date,

unique identification number, formula weight, density, mass and/or volume of standards, and suggested storage requirements. Further details about labeling and handling of standards are described in Section 8.5.4.2 of this LQM.

5.2.5 Corrective Action for Failure to Meet Required Specifications

Corrective actions for failure of an item to meet required specifications are as follows:

- review of current supplies to eliminate the problem item
- notification to the TestAmerica Procurement Director to avoid additional problems at other TestAmerica labs
- return of the problem item to the vendor
- evaluate the impact on product or process

The QA Manager shall be notified of any significant or systematic quality problems. The TestAmerica Procurement Director and the TestAmerica Quality Assurance Director shall be notified of any quality problems with national vendors.

5.3 Procurement of Subcontract Laboratory Services

Whether external to TestAmerica or not, all subcontracting from the TestAmerica North Canton laboratory to another laboratory is arranged with the documented consent of the client, in a timely response that shall not be unreasonably refused. All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Where applicable, specific QC guidelines, QAPjPs, and similar project documents are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC).

Subcontract laboratories may receive an on-site audit by a representative of TestAmerica QA staff if it is deemed appropriate by the QA Manager. The audit involves an assessment of compliance with the required test method, QC requirements, documentation, as well as any special client requirements. The QA manager can request documentation for a desk audit as well.

Project reports received from external laboratories are not altered and are included in original form in the final report provided by TestAmerica. Intra-company subcontracting may also occur between TestAmerica facilities. The originating laboratory is responsible for communicating QA/QC, reporting, and other project requirements.

The final report from TestAmerica North Canton clearly identifies what testing was performed by other laboratories.

6.0 Computer Hardware and Software

The primary purpose of quality assurance systems for computer hardware and software is to protect the integrity of computer-resident data. Procedures are in place at TestAmerica North Canton to assure that computer-resident data are accurate, traceable to a known source, protected against loss, and secure.

TestAmerica computer and hardware controls are based on the guidance in EPA's "Good Automated Laboratory Practices" (GALP), August, 1995. This includes both corporate level Information Technology (IT) functions and TestAmerica North Canton IT functions. Some GALP requirements, such as management responsibilities and the training program, are addressed in other sections of the LQM. Some corporate level IT functions, such as the system change management procedures, is described in more detail in corporate IT documents. Table 6-1 provides a cross reference of practices outlined in Section 8 of the GALP manual to corresponding sections of TestAmerica QA and IT documents. TestAmerica North Canton list of hardware and software are available upon request, and maintained by the local IT Dept.

6.1 Computer Hardware

Computer hardware used in the generation, measurement, or assessment of client data shall be of appropriate design and adequate capacity to function according to specifications. Computer equipment must be installed in accordance with the manufacturer's recommendations, and undergo documented acceptance testing.

6.1.1 Wide-Area Systems

TestAmerica North Canton LIMs and the Office Network run on a wide-area network (WAN) serving multiple laboratories. The central node for the network is located at the Denver facility. The central processor is an IBM AS-400 with multiple servers and Cisco routers. Records for the system architecture, testing and maintenance, such as Initial Program Loads (IPLs), are documented in the AS-400 System Log, which is also in Denver. The central System Administrator maintains records for installation of the network hardware.

6.1.2 Local Systems

The local systems consist of computer equipment for analytical instruments, data evaluation, and upload to the LIMS. A local-area network (LAN) supports the local office software. Testing, maintenance, and repair of the local computer hardware are the responsibility of the TestAmerica North Canton LAN Analyst. The LAN Analyst maintains documentation for the local systems.

6.2 Facilities and Security

6.2.1 Central Computer Facilities

The environmental conditions of the facility housing the LIMS are controlled to protect against data loss. Access to the central computer facility in Denver is

restricted by keypad entry used by IT staff. The central computer room is temperature controlled, and has an Uninterrupted Power Supply (UPS) plus a power generator to ensure that the WAN functions are not disrupted by power failures. Backup media, such as tapes and disks, are maintained daily. In addition, full volume backup copies of the raw data are shipped offsite to a commercial facility specially designed to store electronic data.

6.2.2 Local Computer Facilities

Facilities for housing local computer hardware must meet manufacturer's recommendations. Electronic data must be protected against environmental hazards such as fire, water damage, and strong electromagnetic fields. Data files will have backup copies made at regular intervals to protect against accidental loss through hardware or software failure.

6.2.3 Controlled Software Access

The integrity of data is also assured by maintaining limited access to administrative functions through a hierarchy of operating system shells controlled by passwords. Access is granted by the LAN Administrator depending on a person's experience, training, and assigned duties.

Protection against unauthorized Internet access is provided by firewalls.

6.2.4 Virus Protection

Commercial virus protection programs are installed on all computers to detect and remove computer viruses. LAN Analysts are to be notified whenever a virus is detected so that they can isolate any portions of the systems that may be at risk.

6.3 LIMS Raw Data

LIMs raw data and instrument raw data from instrument data systems such as Target, IDB, and Chemstation are stored on the Office Automation servers (e.g., QARVCO01). The Systems Administrator and the LAN Analyst are responsible for maintaining the servers.

The individuals responsible for entering and recording raw data must be uniquely identified in the data, together with the date and time the data were entered. The instrument transmitting raw data must be uniquely identified, together with the date and time of the transmission. Further data recording requirements exist to document manual integrations (see Policy S-Q-004 for details).

Procedures for verifying raw data are discussed in LQM Sections 8.8-8.8.3.

6.4 Software

If computer software is used to acquire, process, or report client data, that software is tested to ensure that it correctly performs its intended function. Software is validated or verified, depending upon its complexity, size, and whether it was purchased or developed by TestAmerica. The following definitions are used by TestAmerica:

- Validation - the process of establishing documented evidence, which provides a high degree of assurance that a specific process will consistently produce a product meeting, predetermined specifications and quality attributes. This process demonstrates and documents that the software performs correctly and meets all specified requirements.

- Verification - the process of checking the accuracy of automatically (electronically) calculated information.

6.4.1 Industry Standard Software

Industry standard software programs are defined as those, which are purchased and widely used without modification to the program itself. The program is initially verified for use by using test problems with known solutions to demonstrate that the program is operational for the desired application.

All purchased software must be used in accordance with the terms of its software license. Any use of software contrary to its license terms is expressly prohibited by TestAmerica.

6.4.2 Testing of TestAmerica-Developed Software

For programs used to process client data and developed within TestAmerica, and externally prepared programs, which are modified by TestAmerica, validation or verification must be performed. The process used is dependent upon the function of the software as follows:

- Large complex systems consisting of several programs operating in unison to produce an intended result must be validated.

- For smaller software that only performs numerical manipulation, sample sets of numbers for which results are known should be processed and the results verified. In this case, known results are usually generated by performing hand calculations using the same equations and procedures as the software to verify that the software produces identical results.

- Software that performs as part of instrument operation should be verified as previously described and by processing reference materials through the instrument system. Processed instrument response should be evaluated against expected instrument response and performance.

6.4.3 Control of Software Changes

TestAmerica has a well established process for prioritizing and managing changes to LIMS and LIMS-related software. Proposals to modify software are written in a Software Enhancement Request, which includes a description of the task to be accomplished, the software to be modified, its functional requirements, and necessary algorithms. The Software Enhancement Request is submitted to the Change Management Committee for approval. The Committee establishes a development schedule and approves the resources needed. Documentation of changes, version control, and historical records of changes is the responsibility of

the IT Manager. Because these are modern networked systems, the documentation is kept on the network, rather than keeping redundant records at each facility as GALP suggests. All system software changes are developed in a test area and must pass the designed tests before it is installed in the working area.

The same principles of documenting software changes apply to spreadsheets, small databases, or other small programs that are used solely at the TestAmerica North Canton lab. The verification/validation records must explain the functional requirements, the algorithms and formulas used, the testing performed, and is maintained by the lab QA Manager or designee.

6.4.4 Software Maintenance

Software problems are presented to the local TestAmerica LIMS Help Desk. The problem is discussed to make sure it is understood, and then a solution is determined and prioritized.

6.4.5 Software Revalidation

Whenever a program is changed, the change is evaluated to determine if it is significant enough to make revalidation necessary. If features have been added, previous test problems are rerun to demonstrate that their function has not been affected. New test problems are processed, as previously discussed, to verify added performance. If software revision changes the basic operation of the program, complete revalidation of the program may be required.

Spreadsheets and software used to acquire, process, or report client data must be documented and re-verified when changes are made. The test problems used to provide initial verification is reprocessed and the results compared to demonstrate that performance of the software is unchanged.

Laboratory operations are responsible for the generation of the validation and verification data for instrument level software. QA will maintain the necessary documentation, including checking the target concentration computations to make sure they are correct. TestAmerica Information Technology is responsible for generation and maintenance of documentation relating to verification and validation of the TestAmerica LIMs system.

6.5 Comprehensive System Testing

Comprehensive system testing is performed periodically. Independent auditors, such as Price Waterhouse, include computer systems in their audits, which are commissioned by the laboratory executive management.

6.6 Records Retention

As required by NELAC, electronic raw data and computer documentation are stored for a minimum of five years. See LQM Section 3.0 for further records retention details.

7.0 Contract Review and Project Planning

The generation of environmental analytical data is an intricate process. Success is dependent upon the timely execution of interrelated steps. For many environmental sampling and analysis programs, testing design is site or project specific and is not necessarily the same as the laboratory's standard service. It is TestAmerica's intent to provide both standard and customized laboratory services to our clients, provided that any special requirements are documented in writing, and provided performing the work in this manner does not cause the laboratory to violate relevant regulatory requirements. TestAmerica North Canton has an organizational system in place to ensure that projects are properly planned prior to project initiation. This means that laboratory personnel understand project requirements, that the client clearly understands the lab's capabilities, that the laboratory has the facilities and resources needed to perform the required tests, that samples will be properly handled, that contingency plans are in place, and that analytical data will be reported in accordance with project needs.

7.1 Contract Review

The process of client request for proposal (RFP) and the laboratory's tender of a written response is a process of communication between both parties to understand project requirements and the laboratory's capabilities. All contracts for new work entered into by TestAmerica North Canton are reviewed by the Customer Service Manager (CSM) or designee. Agreements for continuing work are the responsibility of laboratory Project Managers (PMs) or the CSM. Depending on the size and scope of the proposed project, the Laboratory Director and other TestAmerica management staff can also be involved. Technical staff (Operations Manager, QA Manager, and IT staff) can be called upon to perform a review of the technical and QA/QC requirements. The CSM or PM, with this internal support, will work with clients to align project requirements with laboratory capabilities. Any contract requirement or contract modification communicated to TestAmerica verbally is documented and communicated to the client in writing. Any discrepancy between client requirements and TestAmerica's capability to meet those requirements is resolved in writing before acceptance of the contract

All contracts, Quality Assurance Project Plans (QAPP), Sampling and Analysis Plans (SAPs) contract amendments and documented communications become part of the project record as detailed in Section 3.5.

7.2 Certifications and Approvals

A necessary part of the review and work acceptance procedure is the evaluation of project needs for laboratory certification. The persons reviewing the prospective project must determine if project work plans or regulatory permits are tied to specific laboratory certifications or approvals. Where such requirements exist, the laboratory must have the certifications or approvals in place before the work begins. QA personnel coordinate with the state certification agencies to maintain or add additional parameters. Copies of current laboratory certifications are maintained by the QA office, and are available upon request.

7.3 Data Collection Process

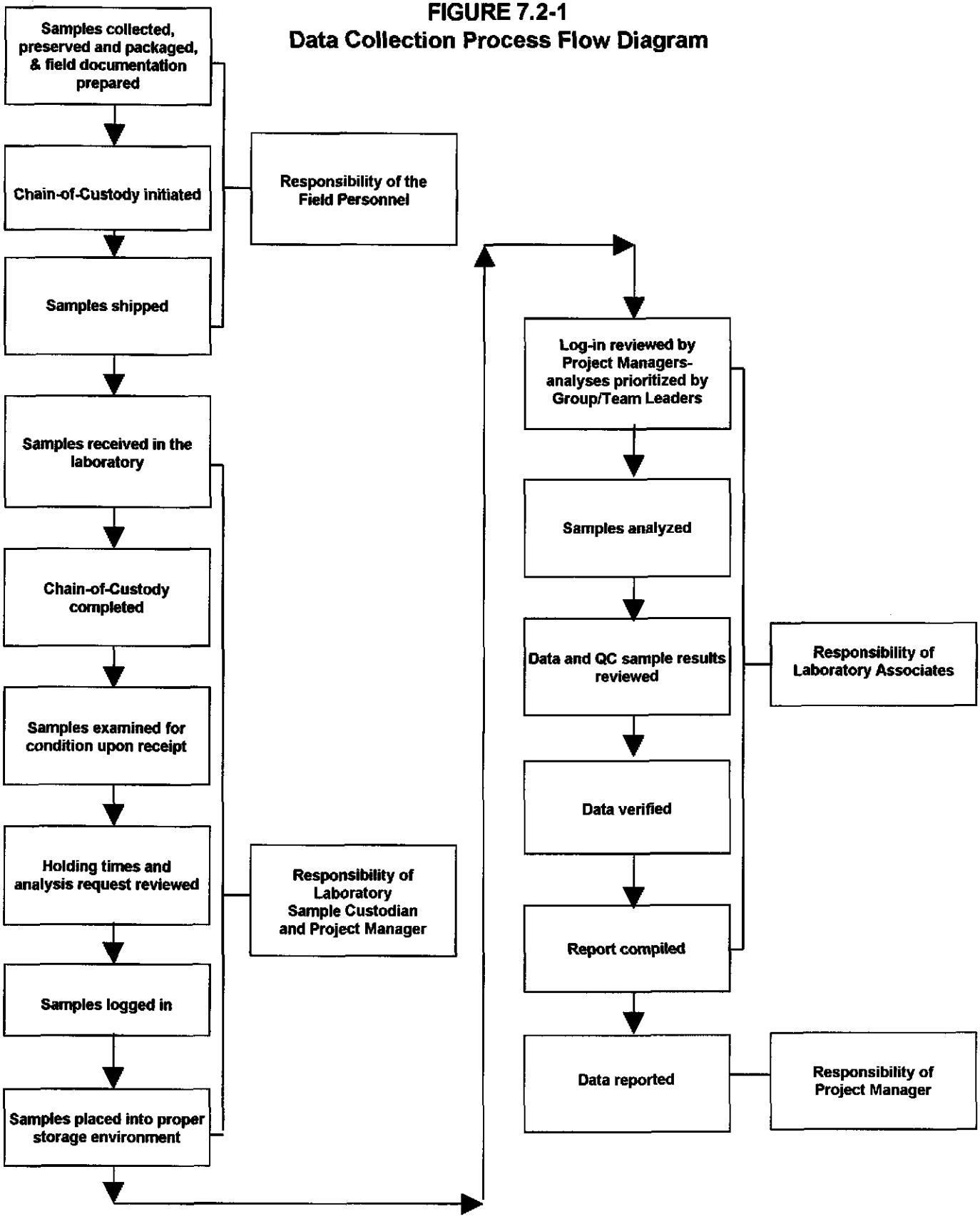
The sample collection and data generation processes are shown in Figure 7.2-1. These processes are designed to produce analytical data that accurately reflect the nature of the site or sampling point.

7.4 Project Organizational Responsibilities

Each laboratory client is assigned a single point of contact, usually a PM, to ensure that there is a strong line of communication between the client and TestAmerica North Canton. As a matter of policy, CSMs or designee, PMs, and Operations Managers work together to accomplish the following prior to receipt of samples at the laboratory:

- Samples are scheduled for arrival at the laboratory
- All unique project requirements have been identified and communicated to all appropriate personnel
- Standardized client, state, federal, or TestAmerica programs are appropriately selected
- Fully-qualified and client approved subcontract laboratories have been selected if needed
- A review has been performed on all pre-project documents such as proposals, contracts, and/or QAPPs to identify the type of tests required and to ensure project requirements are within the scope of the laboratory being used
- All appropriate and required preparations have been made at the laboratory to accommodate or meet project requirements as described in proposals, contracts, and/or QAPPs
- It has been determined that the laboratory has the capability and the capacity to analyze the samples including equipment, staff, space and workload
- The laboratory is capable of meeting the required sample holding times and is able to report the resulting data within the time line specified by the client
- All known safety hazards associated with the samples have been communicated to all appropriate personnel.

FIGURE 7.2-1
Data Collection Process Flow Diagram



For particularly large or involved projects, TestAmerica North Canton encourages clients to visit the laboratory and/or participate in kickoff meetings with the laboratory staff.

7.5 Communicating Project Requirements Internally

TestAmerica North Canton PMs shall document all project-specific requirements prior to receipt of samples. The LIMS system requires the PM to enter a "quote" before any samples can be logged in. In addition to price information, the "quote" is a detailed technical specification of the work to be performed. The quote includes identification of project personnel, numbers and types of samples, tests to be performed, reporting limits, QC to be performed, control limits, data qualifier flags to be used, significant figures to be used, and the types of deliverables required. This is the primary means of communicating routine project requirements to laboratory personnel.

Brief non-routine project requirements are entered into the comments section of LIMS. The comments are reviewed by analysts as analyses are being scheduled and before testing has started. If the special requirements are too lengthy for the LIMS Comment Section, the PM must conduct a project kickoff meeting with each of the operational groups involved, including Central Reporting and Quality Assurance.

7.6 Contingency Planning

An effective QA program must emphasize contingency planning, actions to prevent problems from reoccurring, and to ensure timely and effective completion of a measurement effort. The following are considered relative to contingency planning.

7.6.1 Staffing

A primary objective is to ensure that qualified staff is available to perform the necessary analytical work, regardless of employee turnover, vacation, illness, or other absences. TestAmerica North Canton is a relatively large laboratory with multiple staff capabilities for the majority of tests performed. However, other sources of trained personnel are potentially available to assist in the event of unforeseen absences. Given sufficient time for necessary orientation, temporary agency staff can be used. More significantly, TestAmerica is a large laboratory network and a large pool of qualified staff can be made available from other TestAmerica laboratories.

7.6.2 Backup Instrumentation

Within TestAmerica North Canton, duplicate instrumentation is available for most methods to allow uninterrupted work flow if one piece of equipment fails. The laboratory may also choose to lease equipment. However, in circumstances where a catastrophic instrument failure occurs, alternative, but equivalent, methods may be recommended to the client for approval.

- Preventive Maintenance - TestAmerica's preventive maintenance program is designed to minimize analytical instrument malfunctions, permit simple adjustments, and to ensure fewer and shorter breakdowns of critical analytical equipment. (See Section 8.11, "Preventive Maintenance and Service".)

- TestAmerica Laboratories & Subcontractor Laboratories - To support the laboratory during peak periods or in the event of a critical instrument malfunction, TestAmerica has the capability to arrange for the use of other TestAmerica *laboratories or other qualified analytical* laboratories as subcontractors for short-term backup analytical support. **However, use of a subcontractor laboratory must be approved by the client in writing.** For projects requiring NELAC approval, the subcontractor must also be NELAC approved. See Section 5.3 for other procedures related to the control of subcontract laboratory services.

- Uninterruptible Power Supply – TestAmerica North Canton has an uninterruptible Power Supply (UPS) system that provides line conditioning and backup power to the *LIMS computer system/server and laboratory instruments*. This contingency allows sufficient time for the main computer system to be shut down and for data archival. All electronically generated data that are stored on the main or instrument computer systems and on individual personal computer (PC) hard drives are backed up at regular intervals. In the event that the main or instrument laboratory computer systems fail, the analytical data can be retrieved.

8.0 Work Processes and Operations

Many activities related to environmental projects activities are planned and designed externally to the laboratory or field operation, and are presented to the laboratory in the form of a contract, work plan, sampling and analysis plan (SAP) or QA Project Plan (QAPP). Laboratory and field activities are in turn planned, implemented, and assessed by TestAmerica to meet client requirements according to approved procedures and methodologies. The LQM provides the systems to document and implement these activities. The execution and assessment of the implemented operational systems are detailed in TestAmerica SOPs. The entire process is assessed on a regular basis for conformance to prescribed requirements.

Standard practices for TestAmerica North Canton operations are detailed in this section. Specific project or program requirements that differ from those described here can be met, but they must be explicitly stated in approved contracts, work plans, QAPPs or other project documents. Special project requirements can generally be accommodated provided that they are properly documented, communicated, and they do not cause the laboratory to violate relevant regulatory requirements.

Table 8.2-3 lists the test methods performed by TestAmerica North Canton. Table 8.2-2 lists the SOPs associated with those methods. Table 8.0 provides a list of the major equipment in place at the laboratory, and Figure 8.11 (at the end of this Section) shows the laboratory floor plan. All of these documents are living documents, and the most recent version is available upon request.

8.1 Traceability of Measurements

TestAmerica documents all laboratory activities in sufficient detail to allow their reconstruction. To this end, documentation is generated to trace a sample from its point of origin, through receipt in the laboratory, analysis, reporting and disposal.

The required documentation includes, but is not limited, to:

- Chain of custody documenting movement and possession of samples
- Sample preparation
- Sample analysis
- Calibration and QC data associated with the samples
- Instrument maintenance
- Control of ancillary equipment and materials (e.g., DI water and glassware)
- Sample disposal
- Final reports

These topics are described in this section. Traceability of chemical standards is also discussed in Section 5.2.4.

8.2 Analytical Methods

Whenever possible, TestAmerica operations use industry- and regulatory agency-recognized analytical methods from source documents published by agencies such as the Environmental Protection Agency (EPA), and the American Society for Testing and Materials (ASTM) as described in TestAmerica SOPs. Analytical methods performed by TestAmerica Laboratories are given in Table 8.2-3 through 8.2-9 lists the methods routinely performed at the laboratory.

Method performance data, as described in Section 8.2.2 below, are developed by the laboratory operations staff to demonstrate method proficiency. The operations staff and the QA staff evaluate and approve the performance data before a methodology is performed routinely. The method must also be described and documented in an SOP.

8.2.1 Standard Operating Procedures (SOPs)

SOPs are required for all repetitive analytical and administrative activities ranging from the receipt of samples in the laboratory through their analysis, reporting, and subsequent disposal. Training, health and safety procedures, QC, method procedures, and instrument and equipment calibrations are included in SOPs. SOP requirements are discussed in the Policy QA-001, Standard Operating Procedures, and SOP NC-QA-0027, Preparation and Management of Standard Operating Procedures. The specifications in the policy meet NELAC requirements. Table 8.2-2 lists laboratory standard operating procedures.

New SOPs and proposed SOP revisions are reviewed by technically qualified lab personnel. SOPs are controlled documents and are distributed and maintained as described in Policy QA-001, Standard Operating Procedures, and SOP NC-QA-0027, Preparation and Management of Standard Operating Procedures. Requirements for SOP approval and frequency of review are listed in Tables 2.4-3 and 2.4-4. All significant modifications to the published method are described in a section of the SOP. All operations must be performed as described in these SOPs.

Planned changes in procedure, which may occur due to expected sample matrix effects or project requirements are documented in the project files. These planned changes may be documented using nonconformance memos, NCMs (see discussion of NCMs in Section 9.1), project-specific case narratives, or as modifications or additions to associated QAPPs. Every effort is made to obtain client written approval prior to implementing the change.

Unplanned deviations in the SOPs, which may occur due to sample matrix or other events, are documented in NCMs and/or in the project-specific case narratives.

8.2.2 Method Validation and Verification

Before analyzing samples by a new method or method modification, the method must be verified or validated. After which, analyst capability must be demonstrated (see Section 4.4).

8.2.2.1 Method Verification

Method verification is required for methods developed by authoritative agencies, such as EPA or ASTM. The level of verification can vary depending on the type of method or level of modification, but generally should include:

- Determination of method sensitivity,
- Determination of working range,
- An initial demonstration of capability (as specified by NELAC), and
- A written SOP or project-specific written protocol.

Each of these is described in the next section.

8.2.2.2 Method Validation

A complete validation is required for methods developed by TestAmerica North Canton. While method validation can take a variety of courses, the following are the key concerns:

Determination of Method Selectivity
Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method

Determination of Method Sensitivity
Method sensitivity is normally demonstrated using the 40CFR 136B method detection limit protocol (see MDLs, section 8.2.3, below), but can also be based on variance of blank results, and signal-to-noise ratios.

Determination of Interferences
This is demonstrated by analyzing samples of the matrix of interest that is known to be free of the analyte(s) of interest.

Determination of New Matrix
Prior to the initial analysis of a new or unknown sample matrix, a minimum of 3 MS/MSD samples in said matrix must be analyzed. The percent recoveries must be compared to in-house or program control limits. This action requires consultation with the client prior to implementation.

Determination of Range
In most cases, analytical range is determined and demonstrated by comparison of the response of an analyte at different concentrations to targeted criteria. Often, the targeted criteria are represented by the goodness of fit or linearity of the experimental data to a continuous mathematical function or curve. The curve is used to establish the range of quantitation, with the lower and upper values representing the upper and lower quantitation limits. Curves are not limited to linear relationships.

Determination of Accuracy and Precision
Accuracy and precision studies are generally performed using replicate analysis of samples of known concentration. The resulting percent recovery and relative standard deviation, or other precision measure, is calculated and compared to a set of target criteria.

Documentation of Method
The method is formally documented in an SOP (see Policy QA-001 and SOP NC-QA-0027for details). If a method modification is being performed for a specific short-term project, the modification should be described in a written protocol that is approved by the lab's client, in addition to the in-house approvals required by Policy QA-001and SOP NC-QA-0027.

Continued Demonstration of Method Performance
Continued ability of the lab to perform the method is addressed in the SOP. Generally this is accomplished with the specified calibration and batch QC requirements.

8.2.3 Method Detection Limits

It is TestAmerica North Canton's policy to follow the specification in the U.S. EPA 40 CFR Part 136 Appendix B in determining MDLs for chemical tests. The TestAmerica requirement for this procedure is further detailed in SOP S-Q-003 entitled "Method Detection Limit Studies". This SOP requires that the MDLs be determined for each analyte of interest representing the matrices within the capability of the primary analytical methods. TestAmerica North Canton performs MDLs per instrument. Ongoing MDL verification is performed via MDL checks or MDL studies. For DoD projects, MDL requirements are specified in SOP NC-QA-0016, Supplemental Practices for DoD Project Work. The laboratory's MDLs are available upon request.

8.2.4 Instrument Detection Limits

IDLs are not required by the SW-846 methods, with the exception of Method 6020 and DOD QSM current revision for Method 6010B. When required, IDLs will be performed in accordance with Policy QA-014, Determination of Instrument Detection Limits.

Prior to acceptance and use for reporting purposes, all data from detection limit studies and reporting limits must undergo technical review and approval by the laboratory management and QA staff.

8.2.5 Reporting Limits

Reporting limits are established and modified within TestAmerica according to TestAmerica SOP S-T-002, Reporting Limits for QuantIMS R1. Two reporting limit conventions are discussed in the policy: the standard Reporting Limit (RL) and the Project-Specific Reporting Limit (PSRL). The standard TestAmerica Reporting Limit (RL) is the lowest level at which measurements become quantitatively meaningful. The RL is always greater than the statistically determined MDL. PSRLs are used when project data quality objectives (DQO) require a reporting limit other than the RL. PSRLs tailor TestAmerica's product to meet customer requirements. Higher PSRLs may be established based on maximum contaminant level (MCLs), applicable or relevant and appropriate requirements (ARARs), or project-specific data quality objectives (DQOs). PSRLs below the lab's standard RL may be used, but they must be supported by the MDL. The TestAmerica RLs and PSRLs are maintained by the LIMS. Tables 8.2-10-1 through 8.2-10-7 list the standard reporting limits for TAL/TCL analytes.

8.3 Data Quality Objectives

Data quality objectives (DQOs) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application (EPA 1994)[1]. Typically, DQOs are identified during project scope and the development of sampling and analysis plans. In this LQM, however, we refer to only the analytical DQOs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the

[1] "Guidance for the Data Quality Objectives Process", EPA 600/R-96/005, September 1994.

environmental sample before the sample is received in the laboratory. The EPA has established six primary analytical DQOs for environmental studies. These DQOs are precision, accuracy, representativeness, completeness, comparability, and detectability.

The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. TestAmerica incorporates numerous QA and QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA/QC samples and their applications, described in Section 8.4, are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples are received from the client as unknown samples. Analytical laboratory QC samples for inorganic and organic analyses may include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), and surrogate spikes.

8.3.1 Precision And Accuracy

Precision is an estimate of variability, that is, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. The precision of a measurement system is affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent difference (RPD) for duplicate measurements. Table 8.6-1 illustrates the formulae used to calculate units of precision (i.e., RSD and RPD).

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100).

The precision and accuracy measures that are to be used in evaluating inorganic and organic constituents at TestAmerica are provided in Tables 8.4-5 through 8.4-7, in method-specific SOPs, and in the documentation for the analytical method of interest.

Precision and accuracy are determined, in part, by analyzing data from matrix spike and matrix spike duplicates, unspiked duplicates, LCSs, and single blind audit samples. A description of these QC samples is provided in Section 8.4.

8.3.2 Completeness

Completeness is a measure of the percentage of measurements that are judged to be valid measurements. At a minimum, the objective for completeness of data is 90% for each constituent analyzed.

8.3.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that,

through sample homogeneity, the sample analysis result (concentration) is representative of the constituent concentration in the sample matrix. Representativeness is also affected by sample handling and analysis. Samples with improper preservation, expired holding time, blank contamination, etc., are not representative. An aliquot that is representative of the original sample is analyzed.

8.3.4 Comparability

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data. Comparability is also affected by data quality. Only data of known quality (i.e., precision and bias) are comparable.

8.4 Quality Control Samples

Two types of Quality Control (QC) samples are field QC samples and laboratory QC samples. Field QC samples are collected during the sampling event and are useful in determining sampling precision and accuracy and monitoring for contamination that may occur during collection, transport or storage of environmental samples. Laboratory QC samples are routinely added at the laboratory to the normal sample stream. Successful analysis of these samples demonstrates that the laboratory is operating within prescribed requirements for accuracy and precision. In addition, utilizing matrix-specific laboratory QC samples, information regarding the effect of the matrix or field conditions on the analytical results can be obtained. The following sections describe common field and laboratory QC samples.

8.4.1 Field QC Samples

When field QC sample collection and analysis are required for a project, it is the responsibility of the project sampling supervisor to ensure that this sampling is performed correctly and at the project-required frequencies. Field QC samples may or may not be identified as such to the laboratory and are considered by the laboratory as field samples for the purpose of QC batching, sample preparation and analysis. Field QC sample results are reported in the same manner as actual field samples. No correction of the analytical data is done in the laboratory based on the analysis of field QC samples. Field QC sample types, applicability to organic and inorganic analyses, precision and accuracy applications and by whom they are introduced are summarized in Table 8.4-1.

8.4.2 Laboratory QC Samples

Laboratory performance QC is required to ensure the laboratory systems (instrumentation, sample preparation, analysis, data reduction, etc.) are operating within acceptable QC guidelines during data generation as required to meet the client's objectives. Laboratory QC samples consist of method blanks (MB), instrument blanks, laboratory control samples (LCS) and calibration verification samples. In addition to laboratory performance QC, matrix-specific QC is utilized to determine the effect of the sample matrix on the data being generated. Typically, this includes matrix spikes (MS), matrix spike duplicates (MSD), sample duplicates, and the use of surrogate compounds.

Laboratory and matrix-spike QC sample types are summarized in Tables 8.4-2 through 8.4-4. In addition, Tables 8.4-5 through 8.4-6 list laboratory QC samples, acceptance criteria and corrective actions by reference method. The following sections provide descriptions of laboratory QC samples and their frequency of use. Policy QA-003, "Quality Control Program", describes in detail the QC data evaluation process.

8.4.2.1 Quality Control (QC) Batch

The QC batch consists of a set of up to 20 field samples that behave similarly (i.e., same matrix) and are processed using the same procedures, reagents, and standards within the same time period. This definition of a QC batch is utilized by TestAmerica unless there is clear regulatory guidance, contract specifications, or differing client requirements that are explicitly documented. Environmental samples are associated to QC samples in the LIMs via the analytical batch, which is clearly indicated on each analytical report as a cross-reference between the samples and associated QC. Further details and requirements for the application of the definition of QC batch are described in Policy QA-003.

8.4.2.2 Method Blank

The method blank (MB) is a QC sample that consists of all reagents specific to the method and is carried through every aspect of the procedure, including preparation, cleanup, and analysis. The method blank is used to identify any interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Potential sources of contamination include solvent, reagents, glassware, other sample processing hardware, or the laboratory environment. In general, the method blank is a volume of deionized laboratory water for water samples, or a purified solid matrix for soil/sediment samples, that is processed as a sample. In the event that no appropriate solid matrix exists, deionized water may be used. The volume or weight of the method blank must be approximately equal to the sample volume or sample weight processed. A method blank shall be prepared with each group of samples processed.

8.4.2.3 Instrument/ Calibration Blank

The instrument blank is an unprocessed aliquot of reagent used to monitor the contamination of the analytical system at the instrument. System contamination may lead to the reporting of elevated analyte concentrations or false positive data. The instrument blank does not undergo the entire analytical process and generally consists of an aliquot of the same reagent(s) used for a sample dilution. Instrument blanks are also referred to as continuing calibration blanks (CCBs).

8.4.2.4 Laboratory Control Sample

A laboratory control sample (LCS) is a laboratory-prepared clean matrix sample that is fortified with target analytes or a solid reference material purchased from an approved vendor. The LCS contains target analytes specified in the method or specified by the client, and must contain the same analytes as the matrix spike and matrix spike duplicate. For certain regulatory or client programs, an LCS may contain a long list of analytes. However, in these cases, a subset of analytes, as defined by the program, is used to determine the acceptability of a batch of sample data. The LCS recovery data are used to monitor the analytical method performance in terms of analytical accuracy. Ongoing evaluation of the LCS recoveries demonstrates that the laboratory is performing the method within statistical control (i.e., accuracy and precision) in the absence of matrix interference. The LCS results, coupled with MS data, help determine whether the laboratory performed the method correctly or the sample matrix affected the analytical results. When a laboratory control sample duplicate (LCSD) is required, a percent recovery for each target analyte is calculated, as well as a relative percent difference (RPD) between the LCS and the LCSD.

8.4.2.5 Matrix Spike

A matrix spike (MS) is an environmental sample to which known concentrations of target analytes have been added. MS samples are analyzed to evaluate the effect of the sample matrix on the analytical methodology. MS samples are generated by taking a separate aliquot of an actual field sample and spiking it with the selected target analyte(s) prior to sample extraction. The MS sample then undergoes the same extraction and analytical procedures as the unfortified client sample. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked and not on samples collected at other locations that are included in the QC batch.

8.4.2.6 Matrix Spike Duplicate

A matrix spike duplicate (MSD) is a second aliquot of a sample that is spiked with the selected target analyte(s) and analyzed with the associated sample and MS sample. The results of the MS and MSD are used together to determine the effect of a matrix on the accuracy and precision of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results may have immediate bearing only on the specific sample spiked and not all samples in the QC batch.

8.4.2.7 Sample Duplicate

A sample duplicate is a second aliquot of an environmental sample taken from the same sample container that is processed identically with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The results are compared to determine the sample homogeneity and the precision of the analytical process.

8.4.2.8 Surrogates

Surrogates are organic compounds that are similar in chemical composition and behavior to the target analytes but that are not normally found in environmental samples. Surrogates are added to all appropriate samples and QC samples being tested for organic analytes to monitor the effect of the sample matrix and the procedure on the accuracy of the process.

8.4.2.9 Analytical Spike

An analytical spike is created by spiking target analytes into a prepared portion (i.e., post digestion) of a sample just prior to analysis. It provides information on matrix effects encountered during analysis such as suppression or enhancement of instrument signal levels. It is most often used in elemental analysis involving various forms of atomic emission or atomic absorption spectroscopy. A single analytical spike serves as a single point application of the "method of standard additions" or MSA.

8.4.2.10 Interference Check Sample

An interference check sample (ICS) is a solution containing known concentrations of both interfering and analyte elements. Analysis of this sample can be used to verify background and interelement correction factors.

8.4.2.11 Internal Standards

An internal standard (IS) is a compound or element with similar chemical characteristics and behavior in the analysis process to the target analytes, but is not normally found in environmental samples. The internal standard is usually added after sample preparation. The primary function of the internal standard is quantitation; however, it also provides a short-term indication of instrument performance.

8.5 Data Collection Operations

Laboratory analyses are designed to produce data that are representative of existing conditions present at the time the sample was obtained. The data collection design includes field sampling events, sample handling and custody, analytical operations, data recording procedures, data assessments, data verification, and data reporting requirements and techniques to assess limitations of data use.

8.5.1 Field Collection and Shipment

In order to provide a sample that most accurately represents the test matrix, field sample collection personnel must abide by the sample collection guidelines and procedures established by involved regulatory agencies. A significant part of the efforts of regulatory agencies include the use of "approved" sample containers, chemical and physical preservation techniques, and observance of specified holding times. It is imperative that all samples be collected and preserved according to the appropriate analytical method specified in the QAPP (if one exists). Although the sampling may be performed by non-TestAmerica personnel, the importance of sampling and transportation of the sample to the laboratory is understood and must be considered during data validation.

Sampling requirements must be communicated to the sampling team prior to field collection.

Field personnel are responsible for labeling each individual sample collected with the following information:

- Project name
- Unique client sample number
- Sample location (including as appropriate: borehole and depth or grid coordinates)
- Sampling date and time
- Sample preservation
- Analysis required.

An overriding consideration for the resulting analytical data is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, laboratory custody, and disposal must be documented to accomplish this. Figure 8.5-1 shows an example Chain-of-Custody (COC) form that is used by the TestAmerica laboratory to document this evidence. Field personnel are responsible for initiating the COC form.

The prompt shipment of samples to the laboratory is necessary to ensure that required holding times are met. Samples should be shipped by an overnight carrier, be hand-delivered, or transported in a manner that assures prompt delivery to the laboratory.

8.5.2 Sample Containers, Shipping Containers, Preservatives, and Holding Times

8.5.2.1 Sample Containers

A sample container is defined as the sealed enclosure, usually made of plastic or borosilicate glass or other device, such as Encore, that the sample is collected in and stored in until analysis. All sample containers provided by TestAmerica operations for environmental sampling are new. All documentation certifying sample container cleanliness must be maintained by the laboratory or the vendor and can be provided to the client upon request.

Figure 8.5-1 Example TestAmerica Chain-of-Custody Form

TestAmerica North Canton
4101 Shuffle Drive N.W.
North Canton, OH 44720
phone 330-497-9396 fax 330-497-0772

Chain of Custody Record

Client Contact	**Project Manager:**	**Site Contact:**	**Date:**
Your Company Name here	**Tel/Fax:**	**Lab Contact:**	**Carrier:**
Address	**Analysis Turnaround Time**		
City/State/Zip	Calendar (C) or Work Days (W)		
(xxx) xxx-xxxx Phone	TAT if different from Below _____		
(xxx) xxx-xxxx FAX	☐ 2 weeks		
Project Name:	☐ 1 week		
Site:	☐ 2 days		
P O #	☐ 1 day		

Sample Identification	Sample Date	Sample Time	Sample Type	Matrix	# of Cont.

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other

Possible Hazard Identification

☐ *Non-Hazard* ☐ *Flammable* ☐ *Skin Irritant* ☐ *Poison B* ☐ *Unknown* ☐

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)

☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For* ☐ *Months*

Special Instructions/QC Requirements & Comments:

Relinquished by:	Company:	Date/Time:	Received by:	Company:
Relinquished by:	Company:	Date/Time:	Received by:	Company:
Relinquished by:	Company:	Date/Time:	Received by:	Company:

The sample containers to be supplied are listed in Tables 8.5. Container volumes listed in these tables may be decreased with the approval of the laboratory QA Manager or Technical Director to accommodate reduced sample volumes required by the facility SOP or increased as applicable to accommodate duplicates and matrix spikes.

8.5.2.2 Shipping Containers

Shipping containers are defined as the sealed enclosure in which the sample containers are stored during shipment from the sample collection site to the analytical laboratory. Shipping containers must be of sufficient number and size to accommodate the samples in an upright condition. Shipping containers must also meet all requirements for the shipment of environmental samples.

Packaged samples must be shipped to the analytical laboratory in a safe manner that preserves the integrity of the samples. The most common method of sample shipment employs coolers or ice chests that are sealed with custody tape and shipping tape. These coolers must be durable and resistant to crushing during shipment. All coolers must be well maintained and cleaned to prevent cross-contamination of the samples. All coolers contain a plastic liner to limit contamination. It is the ultimate responsibility of the person collecting and packaging the sample for shipment to ensure that the shipping containers are clean and functional.

To help prevent sample breakage during shipment, additional consideration must be given to providing shock absorbency to all samples packaged inside the shipping container. Use of bubble-wrap around each sample container is the best way to provide this protection. Foam packing materials are also successfully used.

8.5.2.3 Sample Preservatives

Most analytes have a finite holding time in a given sample matrix. Sample preservation is the chemical or physical means by which samples are treated during and/or following sample collection to aid in the stability of the analytes of interest in that matrix. Sample holding times are also adversely affected when samples are improperly preserved, or shipped unpreserved. The preservation of samples at the time of sample collection will follow the requirements of the analytical methods used. This preservation includes the addition of reagents to deter chemical and biochemical degradation and the maintenance of refrigeration during transit and ultimate storage in the laboratory. The required preservatives for the analysis to be performed on each matrix are included in Tables 8.5-1 through 8.5-3.

8.5.2.4 Sample Holding Times

Holding time is defined as the maximum allowable time a sample can be stored after sample collection and preservation until appropriate processing occurs (preparation or analysis). The holding time may vary according to method or client requirements and is tracked and controlled via the LIMs. Tests designated with holding times as "analyze immediately or ASAP" are considered parameters that should be tested by field personnel or on-site. Each operation has a system in place to ensure that holding times are monitored by each group within the operating unit. It is the responsibility of each TestAmerica associate processing the sample to assure that holding times are met. TestAmerica is responsible for meeting all holding times for properly preserved samples received within 48 hours of collection or if less than half the holding time has passed. If these conditions are not met, TestAmerica will attempt to expedite sample analysis as soon as possible. When holding times are exceeded, the laboratory uses a Nonconformance Memo (NCM) to identify and document the root cause of the holding time violation.

Sample holding times are listed in Tables 8.5-1 through 8.5-3.

8.5.3 Sample Handling

TestAmerica North Canton SOP, Sample Receiving NC-SC-0005, describes the sample receipt and log-in process in detail. The following sections describe the general policies followed by TestAmerica.

8.5.3.1 Sample Receipt

Samples shall be received and logged in at TestAmerica by a designated sample custodian or other properly trained associate. Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment. At a minimum, this consists of cut-resistant gloves, a lab coat, and safety glasses
- Examine the shipping containers to verify that the custody tape is intact
- Examine all sample containers for damage
- Open shipping containers in adequately ventilated areas to assure worker safety
- Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the Cooler Receipt Form
- Compare samples received against those listed on the COC
- Verify that sample holding times have not been exceeded
- Examine all shipping records for accuracy and completeness

- Determine sample pH (if required for the scheduled analysis) (except VOA and TOX samples) and record on the Cooler Receipt Form (CRF)
- Sign and date the COC immediately (only after shipment is accepted) and attach the waybill
- Note any problems associated with the coolers and samples on the cooler receipt form and notify the PM who in turn notifies the client
- Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test
- Place the samples in proper laboratory storage.

A Cooler Receipt Form (CRF) or an equivalent form/system is generated by sample control during the sample log-in process to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The affected client shall be notified by the PM or designee of all CRFs generated for their samples. The PM is responsible for resolving with the client how to proceed with the samples and documenting the decision to proceed with the analysis of compromised samples. CRFs must be resolved prior to sample preparation and analysis. The completed CRF shall be stored in the project file. An example CRF is shown in Figure 8.5-2. The report narrative will include an explanation of sample receiving related anomalies.

8.5.3.2 Exceptions or Discrepancies

TestAmerica reserves the right to reject samples for any of the following reasons:

- No custody seals as required by project
- No chain of custody documentation provided
- Preservation inappropriate for analysis requested
- Sample container inappropriate for analysis requested
- Sample received out of holding time for analysis requested
- Incomplete sample information provided
- Discrepancies between COC and sample labels
- Samples have high levels of polychlorinated dibenzo-p-dioxins/ dibenzo furans (PCDD/PCDFs)
- Samples have a high level gross alpha or beta radiation
- Samples are from a site known to contain chemical warfare agents (CWAs) and the samples have not been screened for them.
- Samples containing high levels of PCBs, Cyanides, Sulfides, and Hydrofluoric Acid.
- Tissue samples that may contain viruses harmful to humans.
- Samples with percent (%) levels of Target analytes must be discussed in advance before receipt.

These or any other project exceptions or discrepancies are discussed with the client and agreed upon action taken.

Figure 8.5-2 Example TestAmerica Cooler Receipt Form/Narrative

TestAmerica Cooler Receipt Form/Narrative **Lot Number:_____**
North Canton Facility

Client:_____ Project:_____ Quote#:_____

Cooler Received on:_____ Opened on:_____ By: _____

Fedx ☐ Client Drop Off ☐ UPS ☐ DHL ☐ FAS ☐ TestAmerica Courier ☐ (Signature)

Stetson ☐ US Cargo ☐ Other:_____

TestAmerica Cooler No#_____ Foam Box ☐ Client Cooler ☐ Other_____

1. Were custody seals on the outside of the cooler? Yes ☐ No ☐ Intact? Yes ☐ No ☐ NA ☐
   If YES, Quantity_____
   Were the custody seals signed and dated? Yes ☐ No ☐ NA ☐
2. Shipper's packing slip attached to this form? Yes ☐ No ☐ NA ☐
3. Did custody papers accompany the samples? Yes ☐ No ☐ Relinquished by client? Yes ☐ No ☐
4. Did you sign the custody papers in the appropriate place? Yes ☐ No ☐
5. Packing material used: Bubble Wrap ☐ Foam ☐ None ☐ Other :_____
6. Cooler temperature upon receipt _____ °C (see back of form for multiple coolers/temp)

METHOD: Temp Vial ☐ Coolant & Sample ☐ Against Bottles ☐ IR ☐ ICE/H_20 Slurry ☐

COOLANT: Wet Ice ☐ Blue Ice ☐ Dry Ice ☐ Water ☐ None ☐

7. Did all bottles arrive in good condition (Unbroken)? Yes ☐ No ☐
8. Could all bottle labels and/or tags be reconciled with the COC? Yes ☐ No ☐
9. Were samples at the correct pH upon receipt? Yes ☐ No ☐ NA ☐
10. Were correct bottles used for the tests indicated? Yes ☐ No ☐
11. Were air bubbles >6 mm in any VOA vials? Yes ☐ No ☐ NA ☐
12. Sufficient quantity received to perform indicated analyses? Yes ☐ No ☐
13. Was a Trip Blank present in the cooler? Yes ☐ No ☐ Were VOAs on the COC? Yes ☐ No ☐

Contacted PM _____ Date:_____ by:_____ via Voice Mail ☐ Verbal ☐ Other ☐

Concerning:_____

1. CHAIN OF CUSTODY

The following discrepancies occurred:

_____

_____

_____

_____

_____

2. SAMPLE CONDITION

Sample(s)_____ were received after the recommended holding time had expired.

Sample(s)_____ were received in a broken container.

3. SAMPLE PRESERVATION

Sample(s) _____ were further preserved in sample receiving to meet recommended pH level(s). *Nitric Acid Lot #042607-HNO3 - Sulfuric Acid Lot # 092006-H2SO4; Sodium Hydroxide Lot # 122805 -NaOH; Hydrochloric Acid Lot # 100504-HCl; Sodium Hydroxide and Zinc Acetate Lot # 050205-CH3COO2ZN/NaOH*

Sample(s)_____ were received with bubble > 6 mm in diameter (cc: PM)

4. Other (see below or back)

Client ID	pH	Date	Initials

TestAmerica Cooler Receipt Form/Narrative
North Canton Facility

Client ID	pH	Date	Initials

Cooler	Temp	Method	Coolant

Discrepancies Cont'd

8.5.3.3 Sample Log-in

Sample log-in activities at TestAmerica are fully documented in Sample Receiving SOP, NC-SC-0005. The following is a general description of the log-in process:

- Enter the samples in the laboratory LIMS, which contains the following information at a minimum:
- Project name or identification number
- Unique sample numbers (both client and internal laboratory)
- Type of samples
- Required tests
- Date and time of laboratory receipt of samples
- Field ID supplied by field personnel
- Notify the PM and appropriate Group/Team Leader(s) of sample arrival
- Place the completed COCs, waybills, and any additional documentation in the project file.

8.5.3.4 Sample Storage

The primary considerations for sample storage are:

- Maintenance at the method prescribed temperature, if required
- Maintenance of sample integrity through adequate protection from contamination from outside sources or from cross-contamination of samples. Low-level and high-level samples, when known, must be stored separately. Samples and standards must be stored in separate refrigerators or freezers. Storage areas for volatile organic test requests should be monitored weekly by the analysis of a holding (refrigerator) blank (an aliquot of contaminant-free water stored in a VOA vial)
- Security of samples within the laboratory.

The requirements listed in Tables 8.5-1 through 8.5-3 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of sample control personnel. TestAmerica will assign individuals the responsibility of notifying the Group/Team Leaders or their designees if there are any samples which must be analyzed immediately because of holding time requirements.

8.5.3.5 Internal Sample Chain-of-Custody and Interlaboratory Transfers

Sample custody within TestAmerica laboratories is described in Sample Receiving SOP, NC-SC-0005. Internal COC may be required for programs defined by state or federal agency. The sample custody documentation shall include the following minimum requirements:

- Name of associate taking custody of the sample from the sample storage area for preparation or analysis
- Dates sample removed from and returned to the sample storage area
- Identification of tests to be performed on the sample aliquot(s) selected by the associate
- Sample matrix
- Laboratory sample numbers
- Sample storage location.

Additional custody records can be provided by the laboratory at the specific request of the client. Access to TestAmerica is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

Samples transferred to a different laboratory than the original receiving facility are transferred under chain-of-custody (COC). The COC is maintained whether the laboratory is another TestAmerica facility or a subcontracted laboratory. If the entire sample volume is transmitted, the original copy of the client's COC form will be used to document the relinquishing of the sample and will accompany the sample to its destination. A copy of the completed COC form shall be retained in the laboratory project file. In the case where an aliquot of a sample is shipped from the laboratory, a new COC will be generated by the laboratory and shipped with the sample aliquot. The original COC will be retained in the project file at the site holding the original sample container.

Samples are not transferred to other TestAmerica facilities or to subcontractor laboratories without prior approval of the client.

8.5.3.6 Subsampling

Sample preparation procedures are referenced and defined in the method SOPs.

8.5.3.7 Sample Disposal and Return Chain-of-Custody

After the requested analyses on the samples have been completed, any remaining portions of the samples will be maintained by the sample custodian until the samples are disposed of or returned to the client. The disposal of each sample is recorded. Sample disposal procedures and documentation are described in operation-specific SOPs. TestAmerica's routine sample retention period is at least 30 days after the analytical report is issued to the client, unless otherwise specified by the client.

If samples are returned to the client rather than disposed of by the laboratory, the original COC or a new COC is used to document custody

transfer back to the client from the laboratory. A copy of the completed COC is retained in the laboratory project file.

8.5.4 Calibration Procedures and Criteria

All equipment and instruments used at TestAmerica operations for quantitative measurements are controlled by a formal calibration program. Table 8.0 lists the lab's major analytical instrumentation, and Tables 8.5-4 through 8.5-6 outline calibration requirements. Calibrations may be periodic or operational. These are described in the lab's method SOPs. The Policy P-T-001, "Selection of Data Points Required for an Initial Calibration Curve," is applicable when the number of data points is not described in the method. At a minimum, these calibration procedures shall include:

- Instrument to be calibrated
- Reference standards used for calibration
- Calibration technique (e.g., linear, quadratic)
- Acceptable performance tolerances and corrective actions required if specifications are not met
- Frequency of calibration
- Calibration documentation requirements.

Whenever possible, recognized procedures such as those published by ASTM or the USEPA or procedures provided by manufacturers shall be adopted. If established procedures are not available, a procedure shall be developed considering the type of equipment, stability characteristics of the equipment, required accuracy, and the effect of operation error on the quantities measured.

8.5.4.1 Physical Reference Standards

Physical reference standards associated with periodic calibrations include weights for calibrating balances and certified thermometers for calibrating working thermometers. Whenever possible, physical reference standards shall be calibrated by a body that can provide traceability to nationally or internationally recognized standards. If these standards are not available, the basis for the reference standard shall be documented.

Physical reference standards shall be used only for calibration procedures and shall be stored separately from equipment used for analysis.

8.5.4.2 Chemical Reference Standards and Reagents

Chemical reference standards are generally associated with operational calibration. These standards include reference materials traceable to recognized standards suppliers. This may include vendor-certified materials traceable to national or international standard reference materials (e.g., NIST). This topic is also discussed in Section 5.2.4.

All chemical reference standards maintained in the laboratory for use in calibrations (or as QC spiking solutions) and reagents prepared in the laboratory shall be labeled or referenced to appropriate documentation (hard copy or electronic) with the following information at a minimum:

- A unique identification including concentration (solutions containing several analytes can be identified such that the solution constituents and concentrations can be referenced to a logbook)
- Medium prepared in
- Preparation date
- Expiration date
- Initials of preparer.

Vials containing standard solutions that are not large enough to accommodate labels listing the above information may be referenced to a laboratory logbook/ notebook entry or standards software. The expiration date of the working standard and reagent must not exceed the expiration date of the original material. These records should provide sufficient detail to allow one to reproduce the standard or reagent.

Records for all purchased standards and reagents shall include the date of receipt, the date opened, and, where applicable, the expiration date.

8.5.4.3 Standard Verification

When possible, reference standards are purchased from a TestAmerica preapproved vendor. Standards are verified by quantitation against a second known standard before reporting data. The standard for verification must meet the laboratory's criteria for the independent/second source ICV verification. Therefore, the verification of a new standard initial calibration with a second source ICV meets this verification requirement, some "bad acting" analytes may not meet these criteria and must be approved by the QAM before use. Standard spiking solutions and surrogates shall be verified by analyzing an LCS with the new standards and verifying against historical criteria limits. Special standards that are obtained from another source must also be independently verified at the laboratory. Verification by the laboratory of a reference standard from neat materials is also necessary.

To extend the use of an expired standard, which may not be allowed by all programs, reverification is necessary provided that new analysis produces acceptable data. The verification of an expired standard is performed against a current, independent standard reference material by analyzing within a valid calibration and QC.

Stock and working standards and reagents are checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. Care is exercised in the proper storage and handling of standard and reagent solutions. Standards and reagents are always stored separately from samples.

An independent or second source standard is used to verify initial calibrations. An independent/second source standard is defined as a standard composed of the same target constituents as, but from a different source than those used in the standards for the initial calibration. An independent standard may be a laboratory-prepared or a certified independent standard solution(s). Independence of reference material can be achieved by:

(1) purchasing reference materials from two separate vendors,
(2) using a different lot from the same vendor that is certified by the vendor as an independent standard, or
(3) having two separate individuals prepare the calibration and verification standard solutions if independent sources are not available.

8.5.4.4 Periodic Calibration

Periodic calibration is performed at prescribed intervals. In general, equipment that can be calibrated periodically is a distinct, singular purpose unit and is relatively stable in performance. These include balances, micropipettors, thermometers, refrigerators, freezers, and ovens. Equipment employed at TestAmerica North Canton requiring periodic calibration are listed along with their respective (NELAC) calibration requirements in Tables 8.5-3.

8.5.4.5 Operational and Continuing Calibration

Operational calibration is routinely performed as part of instrument usage, such as the development of a standard calibration curve (see Tables 8.5-4 through 8.5-6). The accuracy of initial calibrations are to be verified prior to sample analysis through the use of an independent standard in situations where the source method requires calibration verification.

Detailed requirements for operational and continuing calibration are contained in method-specific SOPs.

When an initial calibration is not performed on the day of analysis, the validity of the initial calibration verification must be verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch.

- A continuing instrument calibration verification must be performed as outlined in Tables 8.5-5 (inorganics) and 8.5-6 (organics).

- Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, and calibration curve or response factor.

- When the acceptance criteria for the continuing calibration verification is exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the continuing calibration verification is exceeded low, i.e., low bias, those sample results shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

8.5.4.6 Calibration Failure

Equipment or instruments that fail calibration or become inoperable during use shall be tagged out (NCM created) to indicate they are out of calibration. Such instruments or equipment shall be repaired and successfully recalibrated before reuse. Following recalibration or verification, back to control will be documented in the injection/run log and/or maintenance logbook through the routine identification of the required calibration runs specified by the standard operating procedure.

8.5.4.7 Calibration Records

Calibration shall be documented for each piece of equipment subject to calibration. All calibration records (periodic and operational) directly affect data and may not be limited to one project. These records shall be stored in either the quality records or the associated project files. Project files that include sample data shall either include the calibration records or include reference to them.

8.6 Quality Assessment

The effectiveness of the QA practices is measured by the quality of data generated by the laboratory. Procedures are in place to detect, prevent, and correct quality problems and to ensure quality improvement. Items and processes that do not meet established requirements must be investigated to determine their cause. Improvements must be implemented in the operations that will prevent a recurrence of these quality problems and provide overall quality performance. All phases of laboratory work should be designed with the objective of preventing problems and improving quality on a continuous basis.

8.6.1 Data Quality Assessment

Data quality is judged in terms of precision, accuracy, representativeness, completeness and comparability. The areas of representativeness, comparability, and completeness for an overall project, inclusive of sampling issues, may be beyond the control of the laboratory.

Precision and accuracy assessments are made as part of the evaluation of laboratory QC data generated during sample preparation and analysis. The QC samples employed at TestAmerica North Canton as part of routine sample analysis are summarized in Section 8.4 of this document. Table 8.6-1 shows the precision and accuracy measurements employed. Analytical method SOPs and TestAmerica Policy Number QA-003 include information on requirements for the type of QC samples, frequencies, and acceptance criteria. Additionally, the SOPs and Policy describe the appropriate actions to be taken when a QC sample result does not meet acceptance criteria.

8.6.2 Statistical Evaluation of Data

In-house limits for all QC data must be evaluated and compared to the limits published in the methods for applicable matrices. Method limits will be employed until sufficient QC data are acquired. A minimum of 20 to 30 data points are recommended to establish the in-house QC limits. Calculated results of the QC (LCS) samples are evaluated by comparing against control limits (3-sigma).

Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the non-conformance system, to monitor for trends. Program-specific data analysis requirements for control charts are followed as required for data generated under those programs. These additional requirements shall be documented in a QAPP or QAS.

Precision and accuracy measurements employed by TestAmerica North Canton are available upon request. Calculated results of these QC samples are evaluated using statistical tables or control charts.

8.7 Data Recording Procedures

To ensure data integrity, all documentation of data and records generated or used during the process of data generation must be performed in compliance with Section 3 of this document and Policy T-Q-005, Recording Laboratory Observations and Raw Data.

8.8 Data Reduction and Verification Procedures

Data review procedures comprise a set of computerized and manual checks applied at appropriate levels of the measurement process. Data review begins with the reduction or processing of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to the final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst who performs a 100 percent review of the data to ensure the work was done correctly the first time. Data verification continues with review by a second reviewer who verifies that data reduction has been correctly performed and that the analytical results correspond to the data acquired and processed. This procedure is outlined in Figure 8.8-1.

8.8.1 Data Reduction and Initial Verification

Data reduction and initial verification may be performed by more than one analyst depending upon the analytical method employed. The preparation and analytical data may be reviewed independently by different analysts. In these instances, each item may not be applicable to the subset of the data verified or an item may be applicable in both instances. It is the responsibility of the analyst to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:

- Sample preparation information is correct and complete including documentation of standard identification, solvent lot numbers, sample amounts, etc.
- Analysis information is correct and complete including proper identification of analysis output (charts, chromatograms, mass spectra, etc.)
- Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers
- The appropriate SOPs have been followed and are identified in the project and/or laboratory records
- Proper documentation procedures have been followed
- All non-conformances have been documented
- Special sample preparation and analytical requirements have been met.
- The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or otherwise specified by the client.

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.

Manual integrations are sometimes necessary to correct misintegrations by an automatic data system software program, but must only be performed when necessary. Further discussion of manual integrations and the required documentation is given in Policy No. S-Q-004, "Acceptable Manual Integration Practices".

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.

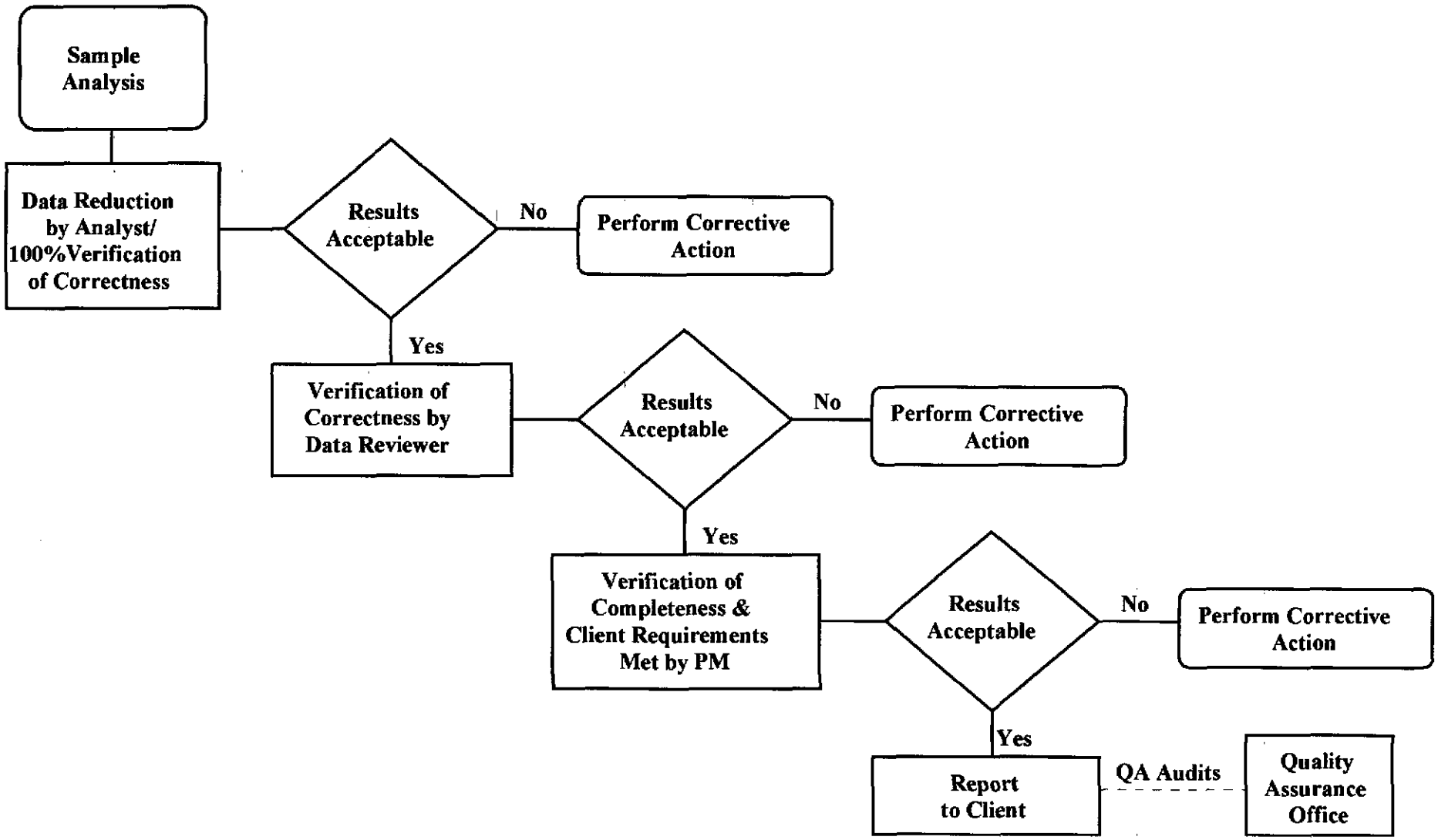
If data are directly acquired from instrumentation or a test procedure and processed, or immediately entered into LIMS, the analyst must verify that the following are correct:

- Project and sample numbers
- Calibration constants and RFs
- Units
- Numerical values used for reporting limits.

Analysis-specific calculations for methods are provided in SOPs. In cases where computers perform the calculations, software must be validated or verified, as described in Section 6.0 of this document, before it is used to process data.

The data reduction is documented, signed and dated by the analyst completing the process. Initial verification of the data reduction by the same analyst is documented on a data review checklist, signed and dated by the analyst. Data review requirements are described in Section 5.3.6 of the QMP.

FIGURE 8.8-1
DATA REDUCTION, VERIFICATION, AND REPORTING



8.8.2 Data Verification

Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through Level 1 review is performed by an experienced peer, supervisor, or designee. This Level 2 check is performed to ensure that Level 1 review has been completed correctly and thoroughly. The second level reviewer examines the data signed by the analyst. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:

- Adherence of the procedure used to the requested analytical method SOP
- Correct interpretation of chromatograms, mass spectra, etc.
- Correctness of numerical input when computer programs are used (checked randomly)
- Correct identification and quantitation of constituents with appropriate qualifiers
- Numerical correctness of calculations and formulas (checked randomly)
- Acceptability of QC data (100% review)
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.)
- Documentation of dilution factors, standard concentrations, etc.
- Sample holding time assessment.

This review also serves as verification that the process the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and client-specific instructions.
- Nonconforming events have been addressed by corrective action as defined on a nonconformance memo
- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct
- The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.

The specific items covered in the second stage of data verification may vary according to the analytical method, but this review of the data must be documented by signing the same checklist. Data review requirements are described in Section 5.3.6 of the QMP.

8.8.3 Completeness Verification

A third-level review is performed by the reporting and project management staff. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG)
- Every parameter or target compound requested is reported with either a value or reporting limit
- All nonconformances, including holding time violations, and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory
- The final report contains all the supporting documentation required by the project, and is in either the standard TestAmerica format or in the client-required format.
- Implement checks to monitor the quality of laboratory results using correlation of results for different parameters of a sample (for example, does the TOC results justify the concentration of organic compounds found by GC/MS.)
- A narrative to accompany the final report will be finalized by the PM. This narrative will include relevant comments collected during the earlier reviews.

8.9 Data Reporting

8.9.1 Data Reports

TestAmerica North Canton is capable of developing a variety of data deliverable reports. Standard reports will contain:

- Cover Letter/Narrative - Information on sample types, tests performed, any problems encountered, and general comments are provided.
- Analytical Data - Data are reported by sample or by test with the appropriate significant figures and reporting limits, and have been adjusted for dilution, if appropriate. Pertinent information including dates sampled, received, prepared, extracted, and analyzed are provided.
- Laboratory Performance QC Information - The results of LCSs and method blanks analyzed with the project are listed. Any data or QC anomalies are discussed in the narrative.
- Matrix-Specific QC Information - Results of any sample duplicates and MS/MSDs analyzed with the samples as batch QC are reported. Other project-specific QC requested by the client are also reported. The results include supporting information such as amount spiked, percent recovery, or percent difference/RPD.
- Methodology - Reference for analytical methodology used is cited.
- Other Deliverables - Other deliverables available include disk deliverables,

CDROM, sample raw data packages, complete deliverable packages, and custom report formats. Requirements for electronic reporting are defined in SOP NC-QA-0019, Records Information Management, and P-L-001, Record Retention.

8.9.2 Final Report Details

TestAmerica North Canton will provide paginated reports or a uniquely defined, identifiable certificate/report (i.e. electronic file, CD). The report will include:

a) Report title, name, address and phone number of the laboratory.
b) Name and address of client/project name/client identification number.
c) Description (lab ID of sample).
d) Dates and Time of sample collections (if known), receipt, preparation and analysis.
e) If the required holding time is 72 or less, time of sample preparation and analysis.
f) Method identifiers traceable to all procedures used.
g) Reporting limit.
h) Test result with appropriate units and how reported (wet weight/dry weight). Also identify any results outside of quantitation limits. When required, a statement of the estimated uncertainty of the test result should be added.
i) If appropriate, description of any QC failures or deviations from SOPs.
j) Signature and title of the individual responsible for the report. Electronic signature is acceptable.
k) Date of issue.
l) All subcontract work must be clearly identified, and name and address of outside subcontractor noted.
m) Where relevant, a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory
n) Where relevant, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory.

After final report any correction, addition, or deletion must clearly identify its purpose and meet the above reporting requirements as appropriate.

All applicable elements from above should be available for review.

8.9.3 Verbal Results

TestAmerica North Canton, as a policy, discourages the release of data verbally or without full data review. If however, the client requests analytical results to be communicated verbally or by facsimile prior to final review, they must be clearly identified as "Preliminary" results. The client must understand that the data have not undergone the required levels of review and may potentially change.

8.9.4 Reporting Analytical Results

Sample results are reported according to analytical method SOPs or client specifications. Normally, the laboratory uses the TestAmerica North Canton Reporting Limit (RL) at which any analyte of interest detected at or above that level is reported as a positive value and any analyte of interest not detectable or detected below that level is reported as "not detected" at the RL. The laboratory will normally report results within the calibration, however, any reported results outside of the calibration range will be documented in the final report.

If a QC measurement is out of control and the data is to be reported, data qualifiers are reported with samples associated with failed QC measurements.

The laboratory must certify that the test results meet all NELAC requirements or provide reasons and/or justification if they do not.

In some cases a contract, QAPP, or documented client request may require the laboratory to report sample results in a specified manner. Some examples are given below:

- The laboratory may be requested to report all analytes of interest that are less than the laboratory's RL but are greater than the MDL. This data will be flagged with an appropriate qualifier or noted in the report case narrative. (See precautions in SOP S-T-002, Reporting Limits for QuantIMS R1).
- The laboratory may be requested to report any tentatively identified compounds (TICs). These data will be flagged with an appropriate qualifier.
- The laboratory may be requested to report sample results using an RL that is higher than their normal level. In this case, only the analytes of interest found at or above that level would be reported as positive values. In this case, the laboratory will state the PSRL rather than the RL. All analytes of interest not detected or detectable below that level would be reported as "not detected" at the PSRL.

  In this situation, the laboratory must include documentation in the project file that supports the reporting procedure employed. It is the responsibility of the laboratory to provide for a reporting system that assures that any problems associated with an analysis are properly documented on a nonconformance memo, communicated to the appropriate TestAmerica North Canton staff, and addressed appropriately in the data report.

8.9.5 Reissued Deliverables

If, after issuance of a report, TestAmerica North Canton observes any mistake that affects the results reported or the QC interpretation of those results, the client will be notified. After issuance of the report, the laboratory report remains unchanged. Any material amendments to a report after issue made only in the form of a further document, or data transfer must include the statement "Supplement to Test Report" or otherwise identified.

8.9.6 Client Confidentiality

Data and sample materials provided by the client or at the client's request, and the results obtained by TestAmerica, shall be held in confidence, unless such information is generally available to the public or is in the public domain. TestAmerica reports, and the data and information provided therein, are for the exclusive use and benefit of our clients, and are not released to a third party without written consent from the client. Data confidentiality is also discussed *Section* 3.6.

8.10 Data Validation

Data validation for TestAmerica refers to data reviews conducted in accordance with the USEPA CLP "Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses" and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses", or modifications thereof, for non-CLP type analyses. For DoD projects, EM-200-1-10 may be used for validation purposes.

This form of data validation provides an impartial evaluation of the laboratory's results. Data validation may be requested by the client for a percentage of data and is usually performed by a third party--one which was not involved with the sample analysis. Qualifiers are assigned to data, when required, according to the requirements of the data validation protocol being used.

8.11 Preventive Maintenance and Service

Facilities, instruments, equipment, and parts are subject to wear, deterioration, or change in operational characteristics. Within TestAmerica, preventive maintenance, coupled with vendor service agreements, is an organized program of actions taken to maintain facilities and equipment in control.

8.11.1 Analytical Instrumentation and Equipment

The primary purpose of the maintenance program is to prevent instrument and equipment failure and to minimize down time. A properly implemented maintenance program increases the reliability of a measurement system.

Each instrument or piece of equipment shall be uniquely identified. The laboratory maintains the following:

- Instrument/equipment inventory list
- Instrument/equipment major spare parts list or inventory
- External service agreement documents (if applicable)
- Instrument-specific preventive maintenance logbook or file for each functional unit.

Logbooks must be kept for each major piece of equipment used in the laboratory. The records of routine maintenance and non-routine maintenance shall include at a minimum:

- Name and serial number and/or unique ID of the item or equipment
- Date placed into service
- Detailed description of the problem
- Detailed description of the corrective action taken to resolve the problem.
- Dates and results of re-calibrations/re-verifications indicating return to control, including documentation, i.e., run number(s) from a successful calibration or batch number of successful analysis. **NOTE:** This documentation is required in the event of a catastrophic failure, instrument tag-out, etc., to demonstrate the instrument was successfully returned to analytical control. All instrument tag-outs must be documented as a non-conformance with Non-Conformance Memo.
- Analyst initials and the date maintenance was performed whether by the analyst or a contracted service representative.

Any item or equipment that does not perform to specifications or defective shall be taken out of service, and tagged as out of service until it has been repaired and shown by calibration/ verification to perform satisfactorily.

8.11.2 Frequency of Equipment Maintenance

The frequency of maintenance must consider manufacturer's recommendations and previous experience. Frequency of preventive maintenance along with the recommended preventive maintenance schedules are given in Tables 8.11-1 through 8.11-24 for analytical instrumentation and equipment or defined in operation specific routine maintenance SOPs. Frequency of maintenance for the facility systems is documented in the Corporate Safety Manual (CSM).

8.11.3 Facilities

Another important aspect of the laboratory operation is the existence and maintenance of adequate, safe, and clean facilities including appropriate engineering controls such as proper ventilation, lighting, dust control, hoods, air flow, protection from extreme temperatures, waste disposal, and a source of stable power. The facility floor plan is provided in Figure 8.11-1.

The maintenance and use of these facilities and proper operations are described in the CSM. The Laboratory Director has responsibility for ensuring a properly maintained facility. The Laboratory Director also has the responsibility for ensuring that facilities are available to store samples properly without contamination, work areas are equipped with adequate bench, hood and operational space, and that procedures are in place to ensure the areas are free from chemical contamination that may affect analytical results. The Volatile laboratories are in a separate

laboratory area from the Semivolatile Laboratories. The Volatile laboratories utilize a separate air handling system with positive pressure to reduce possible contamination (refer to Floor Plan 8.11-1).

8.11.4 Facility Security

The laboratory building is a limited access, secure facility. To ensure that only authorized personnel are able to enter the building from an entrance that is not monitored, entry into each building is limited by use of electronic locks activated by magnetic keys which are issued only to authorized personnel.

During business hours, guest entry is possible only through the main entrance. This entrance is monitored at all times by a receptionist. All guests are required to sign in by using a visitor logbook and wear a visitor's badge. The visitor must be escorted by a TestAmerica employee at all times. Routine vendors are authorized for select sections of the laboratory without an escort. A Visitor Safety Form must be completed by all visitors. All vendors must sign a Vendor Form annually. Clients may drop off samples through a Sample Receiving door after calling for entry.

8.12 Requirements for Ancillary Equipment and Materials

8.12.1 Water

High purity water (e.g., ASTM reagent grade or equivalent water) will be used in all metals, wet chemistry, and organic analyses. Demonstration of contaminant-free water is shown through the analysis of method blanks consisting of the reagent water on a each working day for the analyte of interest.

An Ion-exchange deionizing system or reverse osmosis unit plus an appropriate polishing unit (0.45 µm member filter) compromises the commercial water system.

The reagent water produced by the commercial water treatment system is checked and documented daily for the following:

The commercial water system is checked daily for any possible service needs. The commercial system has an indicator light (light on= ok, and light off = contact service) to relay when a service call is necessary. A daily logsheet is filled out, indicating the light has been checked.

The laboratory is also notified (via laboratory phone paging system) not to use the reagent water system when routine water tank exchanges occur by service personnel.

For volatile analyses the laboratory purchases bottled water that is further purified by purging with an inert gas before use to remove potential traces of organic solvents.

8.12.2 Compressed Air and Gases

Ultra high-purity compressed gases from pre-approved vendors or in-house gas generators will be used when required for instrumentation. These air and gases must meet the requirements and specifications of the analytical methods performed. In-line filters will be used when appropriate to minimize contamination and moisture from the gases.

8.12.3 Glassware Preparation

Glassware preparation procedures implemented at operating units are designed to ensure that contaminants are not introduced during sample analysis. Procedures describing glassware preparation are detailed in operation-specific Glassware Washing SOP, NC-QA-0014.

8.12.4 Chemical Storage

Storage of chemicals shall be conducted in a manner to minimize the potential for fire or release of hazardous material resulting from an unplanned chemical reaction. Refrigerators used for storing flammable liquids must have spark-free interior construction. Flammable solvents shall be stored in appropriate cabinets meeting all necessary codes. All chemicals are stored according to chemical compatibility. Further details regarding chemical storage are provided in the CSM.

8.12.5 Waste Management

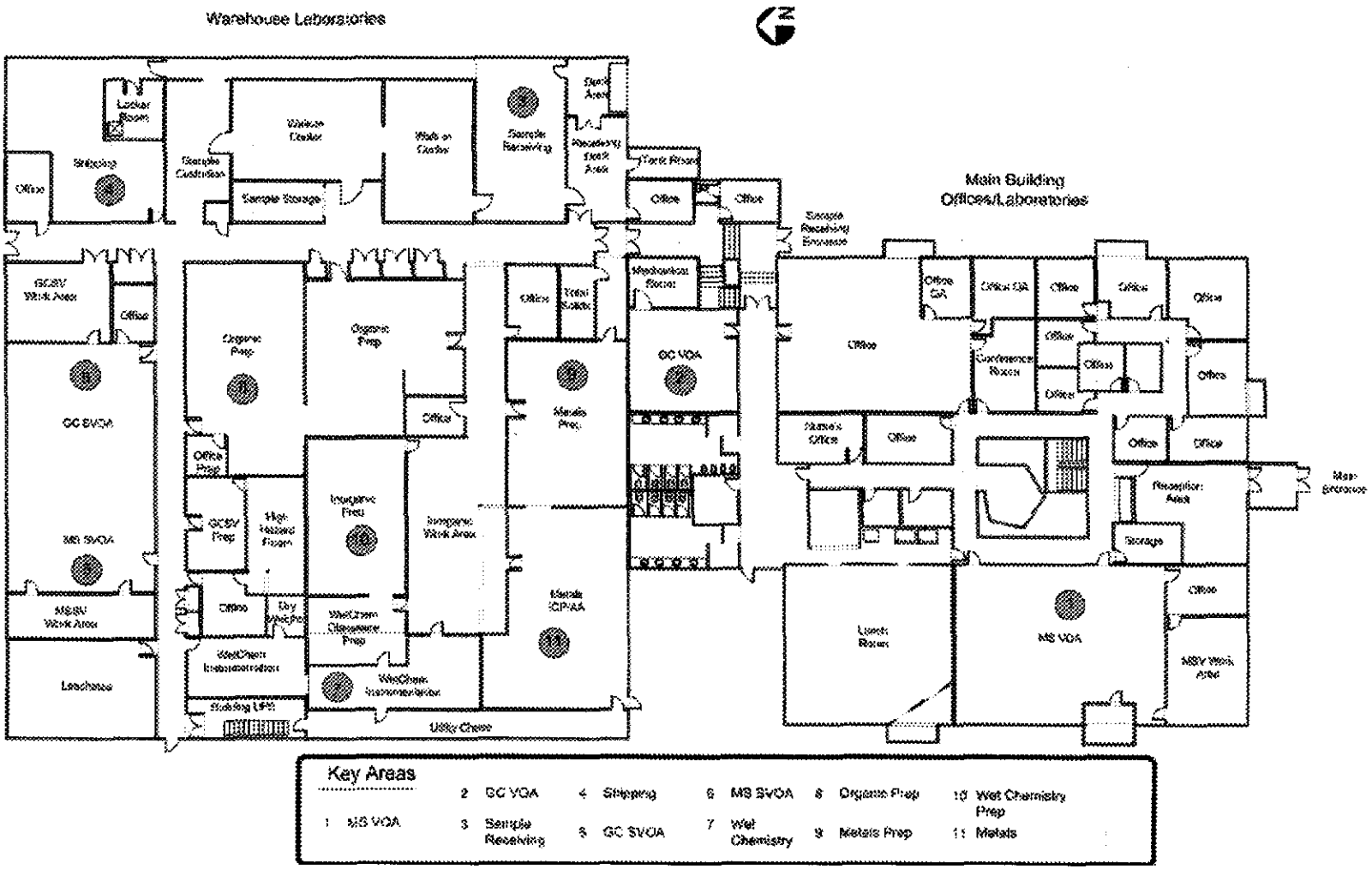
The goal of the TestAmerica policy for waste management is to ensure that laboratory wastes are disposed of safely and in a manner consistent with applicable federal, state and local regulations. The waste disposal program is designed to assure that minimal harm to people and the environment shall result from the disposal of laboratory chemicals and samples. This goal is accomplished by requiring that the laboratory comply with the procedures presented in the CSM.

FIGURE 8.11-1 FLOOR PLAN

TestAmerica – North Canton

4101 Shuffel Dr NW
North Canton, OH 44720

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING



9.0 Quality Assessment and Response

9.1 Nonconformances

A nonconformance is an unplanned deviation from an established protocol or plan and in some cases may be exceptionally permitted departures from the documented policies and procedures or from standard specifications. The deviation may be the result of TestAmerica actions as a systematic error, then termed a deficiency. A single isolated event or event beyond the control of TestAmerica is termed an anomaly.

Nonconformances can be identified on the basis of internal or external systems or performance audits, sample processing, routine calibration and monitoring of analytical and support equipment, or QC sample analyses. The Technical Director, Operations Manager, Project Manager, QA Manager, Group Leader, and Analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected, the issue is immediately brought to the attention of QA and Laboratory Management.

9.1.1 Nonconformance Memo (NCM)

All nonconformances, deficiencies and anomalies, are documented via an electronic process or on a Cooler Receipt Form that meets NCM requirements as approved by QA. An allowed exception is log-in conformance problems, which are documented on a Cooler Receipt Form or equivalent (see Section 8.5 and Figure 8.5-2). A detailed description of the procedure and responsibilities associated with nonconformance documentation, communication, and resolution is described in SOP CORP-QA-0010, "Nonconformance and Corrective Action".

The Clouseau NCM program, available on the local-area network throughout the laboratory, is the main vehicle for documenting and communicating NCMs. The program allows anyone in the laboratory to document a nonconformance, explain the cause of the problem, and link to the LIMS system to identify the samples and clients involved. The program uses the local email to automatically notify the person's Supervisor, the Project Managers associated with the samples, and the QA Department. The program is used to document approval and completion of the immediate corrective actions for the samples involved, and can be used to document long-term corrective actions. It provides a place to document resolution of problems with the clients and to query the associated database to examine trends and prepare management reports. A copy (paper or electronic) of the nonconformance memo will be kept in the project files along with the data it refers to. A copy (paper or electronic) shall also be kept in the quality files.

9.2 Client Complaints

Client inquiries and complaints are generally received through the PM or Customer Services Manager (CSM). Typically, the PM or CSM communicates with the client to determine the details of the inquiries, including technical data problems, deliverable issues, turn-around-time problems, etc. Technical and deliverable issues are coordinated by the PM and usually involve input from operations, QA, and management staff. A formal written response to the client is coordinated by the PM, but may on occasion be delivered by the CSM or the Quality Manager. The procedure for documenting complaints is detailed in SOP S-C-002, Complaint Handling and Service Recovery. Client complaints are recorded and summarized in the monthly QA Reports to Management (see Section 9.6 for more about the monthly QA reports).

9.3 Corrective Actions

Corrective actions are measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence. Investigations of potential problems and corrective actions should be timely, determine the root cause, and evaluate any propagation of the error or problem. Whenever a systematic error is discovered that affects the accuracy or defensibility of results reported to TestAmerica clients, Corporate QA involvement followed by client notification would be part of the corrective action.

Corrective actions should be implemented with an understanding of the technology and work activities associated with the quality element, with appropriate training of TestAmerica associates and vendors, and should be monitored for progress and success. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be documented properly. On-the-spot actions are used to correct minor problems, such as recalibration, retuning, or a minor repair (e.g., replacement of a minor part) of a malfunctioning instrument or the correction of poor analytical technique being used by an analyst. These occurrences are documented in the appropriate injection, run, or analysis logbooks. Similarly, routine instrument maintenance, malfunctions, and power failures are also documented in the appropriate instrument maintenance logbooks. These events do not require a formal NCM process, provided reported analytical results are not affected. Corrective actions specific to quality controls for analytical methods are discussed in the operational-specific SOPs.

9.3.1 Monitoring Corrective Actions

The QA department, either in the Clouseau database or in paper files maintains all formal corrective action documentation. The QA department reviews all corrective actions and confirms they are closed. The QA department may also implement a spot assessment audit. The purpose of these audits is to monitor the implementation of the corrective action and to determine whether the action taken has been effective in overcoming the issue identified.

9.4 Internal Audits

Internal audits are performed to assess the degree of adherence to established policies, procedures and standards. TestAmerica personnel who are independent of the area being evaluated conduct these assessments. Audits can identify areas for improvement with regard to compliance with policies, procedures and standards. Audits also provide a means for correction prior to system failure.

Audits and assessments are generally conducted through the use of checklists and relevant reference documents. The findings of all audits and assessments are documented as is the laboratory response and any corrective actions. Follow-up checks are performed and the status of implementation of corrective actions is documented for all categories of audits and assessments. This cycle continues until all issues are closed.

9.4.1 Audit Types and Frequency

The following types of audits are performed at TestAmerica North Canton:

Figure 9.4.1-1 AUDIT TYPES AND FREQUENCY

Audit Type	Performed By	Frequency
Systems Audits	QA Department or designee	Annual per lab section
Data Authenticity Audits	QA Department or designee	Annually for all analysts
Internal Audit	QA Department or designee	As needed to monitor specific issues
Proficiency Testing	QA Department or designee	Two samples per year per program as required by NELAC

9.4.2 Systems Audits

Facility systems audits are comprehensive technical and systems evaluations covering each operational and support area at least once per year, and may be performed by a TestAmerica auditor, the Laboratory QAM, or designee. Generally, a rotating schedule is established throughout the year to ensure adequate coverage of all areas. This schedule can change as situations in the laboratory warrant. The objectives and schedule of the audit are communicated to the laboratory groups being assessed in advance of the audit. At the completion of the audit, a debriefing is held to outline the findings, including identification of positive performance, to discuss areas of deficiencies, and to

answer questions. The audit report issued by the QA Manager. The audit report is addressed to the area supervisor and/or manager, and copied to the General Manager and Laboratory Director. Written audit responses are required. The audit response from the lab areas must follow the format of the original audit report, and is sent from the respondents to all individuals copied on the audit report. Where a corrective action requires longer than 30 days to complete, the target date for the corrective action is stated and evidence of corrective action is submitted to the QA department in the agreed upon time frame.

9.4.3 Data Authenticity Audits

Data audits are routinely performed and documented to ensure that project records meet project requirements as described in method SOPs, project plans, or other documented requirements. The data audit is used to identify any lab errors that may have occurred. Significant issues found in the course of the audit are brought to the attention of appropriate personnel for clarification, and overseeing correction of final reports if necessary. Data audits include spot-checking manual integrations to determine if they are appropriate via a data checker program (Mint Miner). Errors found in client project reports are revised and the revision sent to the client (also see Section 8.9.5).

9.4.4 Spot Assessments

Spot assessments, equivalent to special audits in the TestAmerica QMP, are conducted on as needed basis, generally as a follow up to specific issues such as client complaints, validator concerns, corrective actions, control chart or NCM trends, proficiency testing results, data audits, or external audit issues. Spot assessments are focused on a specific issue. The frequency, report format, distribution, and timeframes are tailored to address the nature of the issue.

9.4.5 Proficiency Testing

Proficiency testing samples (PTs) are analyzed to verify the ability of the laboratory to correctly identify and quantitate compounds in PT samples. PT samples may be supplied internally or externally as single-blind or double-blind samples. They can be used to assess if a deficiency has been corrected, they can be used to document the proficiency of the analyst performing the analysis, or they can be used to assess the overall performance of an analytical method.

PT samples are handled and tested in the same manner as environmental samples - it is not acceptable to run multiple replicates that would not otherwise be performed, it is not acceptable to average multiple results, and PT results cannot be shared among labs in advance of the close of the study. PT test sample data is archived using the same requirements as for project and raw data record retention.

9.4.5.1 External PT Samples

TestAmerica North Canton participates in a number of PT studies, as shown in Table 9.4-1. The primary one being the NELAC PT program, which involves a minimum of two PT rounds each year for NELAC field of testing for which the lab is maintaining certification.

9.4.5.2 Internal PT Samples

Each TestAmerica facility performing chemical analyses also participates in a double-blind performance evaluation. An external vendor is contracted to submit double blind samples to the TestAmerica labs. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor. The PT contractor provides a detailed report to the Corporate QA Manager and to each of the TestAmerica facilities.

9.5 External Audits

Clients and external regulatory authorities regularly audit TestAmerica North Canton. TestAmerica is available for these audits, and makes every effort to provide the auditors with the personnel, documentation and assistance they require. TestAmerica recommends that all audits be scheduled with the QA department so that all necessary personnel are available on the day of the audit. All deficiencies reported to the laboratory must be responded to within the time frame specified by the auditors. It is the responsibility of the QA Manager to coordinate the response to the audit report. The development and implementation of the corrective actions is the responsibility of the operations management of the affected areas and must be approved by the laboratory operations and management prior to submitting the final response. It is the responsibility of the QA Manager or designee to verify implementation of the corrective actions and inform the responsible manager of the closure of all deficiencies from the audit.

9.6 Management Reviews

9.6.1 Quality Reports to Management

A monthly QA report is prepared by the QA Manager and forwarded to the Laboratory Director, and the Corporate QA Manager. The reports include metrics (i.e., frequency and number of revised reports, frequency and number of client complaints) to assess the effectiveness of the Quality System. The contents of the monthly report include:

- Audits
  Results of internal systems audits performed
  Results of external systems audits hosted
  Data audits performed, percent of total packages per month plus any issues

- Revised Reports / Client Complaints
  Frequency of revised reports
  Total number of client complaints, issues, and resolution

- Certification / Parameter Changes

- Proficiency Testing
  Score for each PT as a percentage of maximum score
  Note repeat failures and/or significant problems
- Miscellaneous QA and Operational Issues
  Narrative outlying improvements, regulatory compliance issues, general concerns, and assistance required from management

This information is compiled by the Corporate QA Manager together with similar information from and about other TestAmerica laboratories, which is then presented in a report to the TestAmerica Chief Operating Officer.

9.6.2 Management Systems Review

Annually at a minimum, the laboratory management will evaluate the status of the quality systems in the laboratory to ensure the procedures and policies are in place and they are adhered to. The Laboratory Director, Operations Manager, Customer Service Manager, and Quality Assurance Manager regularly meet to evaluate quality system(s). Management systems reviews are accomplished by the QA Manager based on monthly quality assurance reporting, goal setting and LQM review. The evaluation(s) shall consider:

- The suitability of policies and procedures
- Reports from managerial and supervisory personnel
- The outcome of recent internal audits
- Corrective and preventative actions
- Assessments by external bodies
- The results of interlaboratory comparisons and proficiency tests
- Status of QA documents
- Reviews of QA related requirements in RFPs, SOWs, SAPs, and QAPPs
- Changes in the volume and type of work and the effects on QA systems
- Client feedback
- Complaints
- Quality control activities
- Resources and staff training

TestAmerica North Canton
Laboratory Quality Manual

Table Section

TABLE 2.4-1
CROSS REFERENCE OF LQM TO NELAC REQUIREMENTS FOR QUALITY MANUALS

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
a) A quality policy statement, including objectives and commitments, by top management	LQM Chapter 1
b) The organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts	LQM Chapter 1
c) The relationship between management, technical operations, support services and the quality system	LQM Chapter 1 LQM Chapter 7
d) Procedures to ensure that all records required under this chapter are retained as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force	LQM Chapter 2 LQM Chapter 3
e) Job descriptions of key staff and reference to the job descriptions of other staff	LQM Chapter 1 and Chapter 4 Separate document (hardcopy and/or electronic) provides job descriptions
f) Identification of the laboratory's approved signatories; at a minimum, the title page of the Quality Manual must have the signed concurrence, (with appropriate titles) of all responsible parties including the QA officer, technical director, and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager	LQM Title/Approval Page
g) The laboratory's procedures for achieving traceability of measurements	LQM Chapter 8

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems

TABLE 2.4-1
CROSS REFERENCE OF LQM TO NELAC REQUIREMENTS FOR QUALITY MANUALS (Cont'd)

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
h) A list of all test methods under which the laboratory performs its accredited testing	LQM Table 8.2-1
i) Mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	LQM Chapter 7
j) Reference to the calibration and/or verification test procedures used	LQM Chapter 8 Tables 8.5-6 through 8.5-7
k) Procedures for handling submitted samples	LQM Sections 8.5.2 and 8.5.3
l) Reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests	Equipment list is Table 8.0-1 LQM Sections 5.2.4, 8.1, 8.5.4, 8.11 to 8.12
m) Reference to procedures for calibration, verification and maintenance of equipment	Calibrations in Tables 8.5-6 through 8.11-30 LQM Sections 8.5.4, 8.11 to 8.12
n) Reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes	LQM Section 9.4.5
o) Procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur	LQM Chapter 9 SOP CORP-QA-0010, "Nonconformance and Corrective Action"

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems

TABLE 2.4-1
CROSS REFERENCE OF LQM TO NELAC REQUIREMENTS FOR QUALITY MANUALS (Cont'd)

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
p) The laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications	LQM Section 7.4 and 9.1
q) Procedures for dealing with complaints	LQM Section 9.2
r) Procedures for protecting confidentiality and proprietary rights (including national security concerns)	LQM Section 8.9.6
s) Procedures for audits and data reviews	LQM Section 5.3.6, 8.8 and 9.4 SOP CORP-QA-0004 "Independent QA Data Review"
t) Processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and/or receive any needed training	LQM Chapter 4 SOP CORP-QA-0013, "Employee Orientation and Training"
u) Process/procedures for educating and training personnel in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions	LQM Section 1.4 Policy QA-008 – Data Recording Requirements QA-010 – Maintaining Time Integrity S-Q-004 – Acceptable Manual Integration Practices P-T-001 – Selection of Calibration Points
v) Reference to procedures for reporting analytical results	LQM Section 8.9 Policy QA-004 "Rounding and Significant Figures" Policy S-T-002 "Reporting Limits for Quantims"

TABLE 2.4-1
CROSS REFERENCE OF LQM TO NELAC REQUIREMENTS FOR QUALITY MANUALS (Cont'd)

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
w) A table of contents and applicable list of references, glossaries, and appendices	LQM Table of Contents List of Policies and SOPs Table 2.3-2

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems

TABLE 2.4-2
TESTAMERICA NORTH CANTON QUALITY DOCUMENTS AND REQUIRED APPROVAL

Quality Document	Required Approvals
Laboratory Quality Manual (LQM)	• Laboratory Director • Technical Director • Quality Assurance Manager
TestAmerica North Canton Policies	• Laboratory Director • Quality Assurance Manager
TestAmerica North Canton Standard Operating Procedures (SOPs)	• Laboratory Director • Technical Specialist • Laboratory Health and Safety Coordinator [1] • Quality Assurance Manager

[1] Required only if procedure encompasses more than standard office safety requirements.

TABLE 2.4-3
TESTAMERICA QUALITY DOCUMENT REVIEW FREQUENCY [1]

Document Type	Frequency of Review	Responsible Party
Laboratory Quality Manual (LQM)	Every Two Years	Quality Assurance Manager
Standard Operating Procedures (SOP)	Every Two Years[2]	Quality Assurance Manger & Technical Specialist
Policies	Every Two Years	Quality Assurance Manager

[1] Review Sheets can be utilized in lieu of changing an entire document to indicate that an SOP or policy has been reviewed, and no changes were necessary (QA-001).

[2] DOD requires an annual review of all SOPs pertaining to analysis of methods listed in SOP NC-QA-0016.

TABLE 3.4-1[1]

TESTAMERICA NORTH CANTON RECORDS AND RETENTION SCHEDULE

Type of Record	Retention	Disposition
General Laboratory Documents		
Instrument output	5 yrs from project completion	Shred or burn
Quality control data	5 yrs from project completion	Shred or burn
Field sample data	5 yrs from project completion	Shred or burn
Final analytical reports	5 yrs from project completion	Shred or burn
Instrument logbooks	5 yrs from last entry	Shred or burn
Equipment monitoring & maintenance records	5 yrs from last entry	Shred or burn
Instrument calibration records	5 yrs from last entry	Shred or burn
Standard preparation logs	5 yrs from last entry	Shred or burn
Standards certificates	5 yrs from last entry	Shred or burn
Measurement & test equipment logs (e.g., refrig., balances, etc.)	5 yrs from last entry	Shred or burn
Method & instrument validation records	5 yrs from last entry	Shred or burn
Instrument manuals	Retain until superseded	Trash
Project management files	5 yrs from date of archival	Shred or burn
Quotes & proposals	2 yrs from date of expiration	Shred or burn
LQM, policies, & SOPs	5 yrs from date of archiving	Shred or burn
Analyst demonstrations of proficiency	5 yrs from date of archival	Shred or burn
Quality assurance audits	5 yrs from last entry	Shred or burn
Certifications & approvals	5 yrs from last entry	Shred or burn
Employee signature list	5 yrs from date of archival	Shred or burn
MDL Studies	5 yrs from last entry	Shred or burn
Performance testing studies	5 yrs from last entry	Shred or burn
QA reports to management	5 yrs from last entry	Shred or burn
Quality control charts	5 yrs from last entry	Shred or burn

TABLE 3.4-1[1]

TESTAMERICA NORTH CANTON RECORDS AND RETENTION SCHEDULE

Type of Record	Retention	Disposition
Environment, Health and Safety Records		
Medical records	Retain while active & 30 yrs from last entry	Shred or burn
Employee exposure & monitoring records	Retain while active & 30 yrs from last entry	Shred or burn
Workers compensation files & first report of injury	Retain while active & 30 yrs from last entry	Shred or burn
Accident logs (OSHA Form 200)	5 yrs from last entry	Shred or burn
Accident reports	5 yrs from last entry	Shred or burn
Environmental permits	5 yrs from last entry	Shred or burn
Environmental management, e.g., discharge reports	5 yrs from last entry	Shred or burn
Health & safety audits	5 yrs from last entry	Shred or burn
Chemical Hygiene Plan	5yrs from archival	
Safety Inspections	5 yrs from last entry	Shred or burn
TLD exposure records	5 yrs from last entry	Shred or burn
EH&S training	5 yrs from last entry	Shred or burn
Accounting	See Accounting and Controls Procedures Manual	
Administrative		
Personnel records (not including medical or disability records)	7 years from last entry	Shred or burn

[1] Client record files can be kept longer than the retention period upon negotiation with the client (i.e., storage fees, notification timelines, etc.), and/or regulatory or program requirements.

TABLE 5.2-1 [1]

LIST OF TESTAMERICA QUALITY-RELATED ITEMS WHICH REQUIRE EVALUATION PRIOR TO USE

Quality-Related Item
Acetone
Dichloromethane
Hexane
Hydrochloric acid
Methanol
Nitric acid
Hydrogen Peroxide
Sulfuric acid
Toluene

[1] SOP S-T-001, Testing of Solvents and Acids

TABLE 6-1
GALP Cross Reference to LQM

GALP Section	GALP Guidance	TestAmerica Document
8.1 Laboratory Management	8.1.1 ensure that personnel clearly understand the functions they are to perform	LQM 1.6 and 4.0
	8.1.2 ensure that QAU monitors computer activities	LQM 6.0
	8.1.3 ensure that personnel, resources, and facilities are adequate and available as scheduled	LQM 1.6
	8.1.4 receive reports of QAU inspection and audit reports, and ensure corrective actions are promptly taken in response to any deficiencie	LQM 9.4 and 9.5
	8.1.5 approve SOPs related to the computer activities, and ensure that deviations to the SOPs are documented	LQM 3.3 and 6.0
	8.1.6 assure that GALP provisions are followed	LQM 6.0
8.2 Personnel	8.2.1 must have adequate education, training, and experience to perform assigned IT functions	CORP-IT-013; LQM 4.0
	8.2.2 a summary of training, experience, and job description must be maintained	LQM 4.1
	8.2.3 personnel must be of sufficient number for timely and proper operation of the computer systems	CORP-IT-0002; LQM 6.0
8.3 Quality Assurance Personnel	8.3.1 shall be separate and independent of IT personnel, and shall report directly to laboratory management	LQM 1.6.1
	8.3.2 shall have immediate access to the computer data, SOPs, and other records	LQM 6.0
	8.3.3 inspect the LIMS at intervals to ensure the integrity of LIMS raw data, and shall present inspection reports to management	LQM 6.3 and 9.4
	8.3.4 determine that no deviations from approved SOPs were made without proper authorization and documentation	LQM 9.1.1
	8.3.5 periodically audit raw data to ensure their integrity	LQM 9.4
	8.3.6 maintain adequate records of the QAU operations	LQM 9.0
8.4 LIMS Raw Data	8.4.1 LIMS raw data and the storage media on which they reside must be identified and documented. The documentation shall be included in the lab's SOPs.	System map is with IT Director
	8.4.2 the individual(s) responsible for entering and recording LIMS raw data must be uniquely identified, together with the date and time the data were entered	QA-008
	8.4.3 the instrument transmitting raw data must be uniquely identified in the record, together with the date and time of transmission	QA-008

TABLE 6-1
GALP Cross Reference To LQM (cont'd)

GALP Section	GALP Guidance	TestAmerica Document
	8.4.4 procedures and practices used to verify LIMS raw data must be documented in controlled SOPs	CORP-IT-0007; LQM 8.8-8.8.3
8.5 Software	8.5.1 SOPs shall be established for: a. software development b. software testing c. change control d. version control e. maintaining historical file	CORP-IT-013; CORP-IT-0001; CORP-IT-0007
	8.5.2 documentation shall be maintained for: a. software description & functional requirements b. algorithms and formulas c. testing and quality assurance	CORP-IT-013
	8.5.3 all documentation is readily available in the facility where the software is used and SOPs are readily available where procedures are performed	CORP-IT-013
	8.5.4 a historical file of software and documentaiton shall be retained	CORP-IT-0001, Section 4.14.1
8.6 Security	Laboratory management shall ensure that security practices are adequate to assure the integrity of data	LQM 6.2 CORP-IT-0005; CORP-IT-013
8.7 Hardware	8.7.1 must be of adequate design and capacity, and a documented description maintained	LQM 6.1; CORP-IT-013
	8.7.2 must be installed in accordance with manufacturer's recommendations, and undergo documented acceptance testing as described in a laboratory SOP	CORP-IT-0001; CORP-IT-013; LQM 6.1
	8.7.3 testing, maintenance, and repair must be described in a laboratory SOP	CORP-IT-013
8.8 Comprehensive Testing	Management shall ensure that comprehensive testing shall be documented at least every 24 months or more frequently as a result of software changes.	CORP-IT-0001; LQM 6.4
8.9 Records Retention	Procedures must be in place for the retention of LIMS raw data and documentation and records pertaining to LIMS	QMP; LQM 3.4-3.5

TABLE 6-1
GALP Cross Reference To LQM (cont'd)

GALP Section	GALP Guidance	TestAmerica Document
8.10 Facilities	8.10.1 the environmental conditions of the facility housing the LIMS must be controlled to protect against data loss	LQM 6.2
	8.10.2 environmental conditions for storing LIMS raw data and records must be adequate	LQM 6.2
8.11 SOPs	8.11.1 SOPs, as described above, must be maintained and readily available where the procedure is performed	LQM 3.1-3.2; SOP Index
	8.11.2 SOPs must be reviewed periodically to ensure that they are accurate	LQM 3.3
	8.11.3 SOPs must be authorized and controlled, with all changes subject to the same approvals and control	LQM 3.3
	8.11.4 an historical file of SOPs must be maintained	LQM 3.5

TABLE 8.0
NORTH CANTON INSTRUMENT LIST

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
Metals ICP	Thermo Jarrell Ash	Trace Analyzer 61E	1994	Yes	6010A & B, 200.7
	Thermo Jarrell Ash	Trace Analyzer 61E	1994	Yes	6010A & B, 200.7
Metals ICP/MS	Perkin Elmer (I-7)	ELAN 6100	2001	Yes	6020, 200.8
	Thermo (I-8)	Series 2	2007	Yes	6020, 200.8
Metals Mercury Analyzer	Leeman (CVAA)	PS200 II	1999	Yes	7470A, 7471, 245.1
	Leeman (CVAF) Low Level	Hydra AF Gold+, Model #112-00067-1	2001	Yes	1631E
	Leeman (CVAA)	Hydra AA	2006	Yes	7470A, 7471, 245.1
GC/MS Semivolatiles	Agilent A4AG2	5975-C	Purchased	NA	8270
	Hewlett-Packard HP7	5973-6890	Purchased	NA	8270
	Hewlett-Packard HP8	5973-6890	Purchased	NA	8270
	Hewlett-Packard HP9	5973-6890	Purchased	NA	8270
	Hewlett-Packard HP10	5973-6890	Purchased	NA	8270
GC/MS Volatiles	Hewlett-Packard (UX2)	5971A-5890	1996	Yes	Screening
	Hewlett-Packard (HP6)	5973-6890	Purchased	Yes	Screening
	Hewlett-Packard (UX7)	5973-6890	Purchased	Yes	Screening
	Hewlett-Packard (UX8)	5973-6890	Purchased	Yes	8260, 624
	Hewlett-Packard (UX9)	5973-6890	Purchased	Yes	8260, 624
	Hewlett-Packard (UX10)	5973-6890	Purchased	Yes	8260
	Agilent (UX11) (former HP)	5973-6890	2000	Yes	8260
	Agilent (UX12)	5973-6890	2002	Yes	8260
	Agilent (UX14)	5973-6890	2003	Yes	8260, 624
	Agilent (UX15)	5973-6890	2005	Yes	8260
	Agilent (UX16)	5975-6890	2005	Yes	8260
GC/MS Volatiles Autosampler	OI Analytical (UX2)	4552	Purchased		Screening
	OI Analytical (HP6)	4552	Purchased		Screening
	OI Analytical (UX7)	4552	Purchased		8260
	OI Analytical (UX8)	4552	Purchased		8260, 624
	OI Analytical (UX9)	4552	Purchased		8260, 6214
	OI Analytical (UX10)	4552	Purchased		8260
	OI Analytical (UX11)	4552	2000		8260
	OI Analytical (UX12)	4552	2002		8260
	OI Analytical (UX14)	4552	2003		8260, 624
	OI Analytical (UX15)	4552	2005		8260
	OI Analytical (UX16)	4552	2005		8260

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
GC/MS Volatiles Purge and Trap	OI Analytical (UX2)	4560	Purchased		Screening
	OI Analytical (HP6)	4560	Purchased		Screening
	OI Analytical (UX7)	4660	2004		Screening
	OI Analytical (UX8)	4560	Purchased		8260, 624
	OI Analytical (UX9)	4560	Purchased		8260, 624
	OI Analytical (UX10)	4560	Purchased		8260
	OI Analytical (UX11)	4560	2000		8260
	OI Analytical (UX12)	4560	2002		8260
	OI Analytical (UX14)	4560	2003		8260, 624
	OI Analytical (UX15)	4660	2005		8260
	OI Analytical (UX16)	4660	2005		8260
	EST (spare)	Encon	2002		
WC Ion Chromatograph	Dionex	DX-320	2001	NA	
	Dionex	DX-120	1999	NA	
WC TOC	OI Analytical	1010 TOC Analyzer	2005		
WC TOX	Thermo Electron	1200-S/N 973515	1998		
	Thermo Electron	1200-S/N 2001.0174	2005		
	Thermo Electron	1200-S/N 2005.0234	2005		
WC UV/VIS	Milton Roy	Spectronic 401	1993	No	
	Genesys	Spectronic 20	1998	No	
WC Autotitrator	Man-Tech	PC – Titrate	2001		
WC pH Meter	Orion pH Meter	250A	1985		
	Orion (Ammonia ISE)	520A	1996		
WC Dissolved Oxygen Meter	YSI	52C E	1993	No	
WC Turbidimeter	HF Scientific	Micro 100	2001	No	
WC BOD	Labtronics, Inc.	BOD	1999		
WC Block Digester	Andrews	2210 Phenol	1999		
	Andrews	2205 Ammonia	1999		
	Lachat	BD46 TKN	1992		

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
WC Cyanide	Midi Serial #1000-99-01	PRG-2520-BL	1999		
WC Conductivity	Man-Tech	4310	1989		
WC Flashpoint	Petrolab (199443)	Petrotest	1999		
	Herzog	HFP 339	2007		
WC Discrete Analyzer	Kone	Konelab 200	2001		
	Kone	Konelab 250	2005		
WCResidual Chlorine Meter	Hanna	HI 93701	2000		
	Hanna	HI 93701	2006		
WC TRAACS	Bran & Luebbe	800	1993		
Ext. 6-Position Accelerated Soxhlet Extractor	Gerhardt Soxtherm	8 units	2003		PCB's
Extractions Sonicators	Misonix	3000	2005	NA	8015, 8051, 8080, 8082, 8270
	Fisher	550	2001	NA	
GC Semivolatiles	Hewlett-Packard (P1)	6890 EPC & Dual ECD Y-Splitter	1998	Yes	8151, 8082, 8081, 608
	Hewlett-Packard (P2)	6890 EPC & Dual ECD Y-Splitter	1998	Yes	8151, 8082, 8081, 608
	Hewlett-Packard (P3)	6890 EPC & Dual ECD Y-Splitter	1998	Yes	8151, 8082, 8081, 608
	Hewlett-Packard (P4)	6890 EPC & Dual ECD Y-Splitter	1999	Yes	8151, 8082, 8081, 608
	Hewlett-Packard (P5)	6890 EPC & Dual ECD Y-Splitter	1999	Yes	8151, 8082, 8081, 608
	Hewlett-Packard (P6)	6890 EPC & Dual FID	2000	Yes	8015B, RSK, VSO
	Hewlett-Packard (P7)	6890 EPC & Dual FID	2000	Yes	8015B, RSK, VSO
	Hewlett-Packard (P9)	6890 EPC & Dual ECD Y-Splitter	1999	Yes	8151, 8082, 8081, 608
	Hewlett-Packard (P10)	6890 EPC & Dual ECD Y-Splitter	1999	Yes	8151, 8082, 8081, 608

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
GC Semivolatiles (Cont'd)	Hewlett-Packard (P11)	6890 EPC & Dual ECD Y-Splitter	2005	Yes	8151, 8082, 8081, 608
GC Semivolatiles HPLC	Hewlett-Packard (L2)	HPLC 1100	1998	Yes	8310, 610, 8315, Organic Acid
	Hewlett-Packard (L3)	HPLC 1100	1998	Yes	8310, 610, 8315, Organic Acid
GC Volatiles	Agilent (A)	6890 PID/FID	2004	Yes	
	Hewlett-Packard (O)	6890 Dual PID/Hall	1997	Yes	
	Hewlett-Packard (P)	6890 PID/HALL		Yes	
	Hewlett-Packard (Y)	6890N PID/FID	2003	Yes	
	Hewlett-Packard (P8)	6890 EPC & Dual FID	2000	Yes	8015B, RSK, VSO
GC Volatiles Auto Sampler	OI Analytical (O)	Archon			
	OI Analytical (Y)	4552			
	Varian (A)	Archon			
	Varian (P)	4552			
GC Volatiles Purge & Trap	Tekmar (O)	4560			
	Tekmar (P)	3000			
	OI Analytical (A)	4560			
	OI Analytical (Y)	3000			
GM Bedford	Agilent	6890N	2004	Yes	8082 PCB's
	Agilent	6890N	2004	Yes	8082 PCB's
	Agilent	6890N	2005	Yes	8082 PCB's
	Misonix Sonicator	3000	2006	NA	8082 PCB's

Table 8.2-2
Standard Operating Procedures Listing [1, 2, 3]

Dept	SOP No.	Revision	Active Date	SOP Title
GC	CORP-GC-0001NC	5.8	02/06/06	Gas Chromatographic Analysis Based on Methods 8000B, 8021B, 8081A, 8082, 8151A, 8310, 8141A, 8015B, and 615
Inorganic Prep	CORP-IP-0002NC	2.5	12/02/04	Acid Digestion for Soil Samples by SW846 Method 3050B
Inorganic Prep	CORP-IP-0003NC	1.6	02/07/07	Acid Digestion for Aqueous Samples by SW846 and MCAWW 200 Series Methods
Organic Prep	CORP-IP-0004NC	1.2	11/11/04	Toxicity Characteristic Leaching Procedure and Synthetic Precipitation Leaching Procedure SW846 Method 1311, SW-846 Method 1312
GC/MS	CORP-MS-0001NC	2.12	03/01/07	GC/MS Analysis Based on Method 8270C - **UPDATE III**
GC/MS	CORP-MS-0002NC	2.5	04/03/07	Determination of Volatile Organics by GC/MS 8260B, 8260A and 624 (Note: Update II and Update III are in one SOP)
Metals	CORP-MT-0001NC	3.4	01/08/04	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis **Update III** 6010B, 200.7
Metals	CORP-MT-0005NC	2.5	01/08/04	Mercury in Aqueous Samples by Cold Vapor Atomic Absorption (Prep and Analysis) SW846 7470A, MCAWW 245.1
Metals	CORP-MT-0007NC	2.5	11/22/04	Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy (Prep and Analysis) SW846 7471A, MCAWW 245.5
Organic Prep	CORP-OP-0001NC	4.2	01/30/06	Extraction and Cleanup of Organic Compounds From Waters and Soils **UPDATE III** SW846 3500-Ser., 3600-Ser., 8151A, and 600-Ser.
Health and Safety	Corporate Safety Manual	6	03/31/06	Corporate Safety Manual
Quality Assurance	CORP-QA-0010	2	6/15/1999 (reviewed 2/3/06 - no changes)	Nonconformance and Corrective Action
Quality Assurance	CORP-QA-0013NC	1.1	11/28/04	Employee Orientation and Training
Wet Chemistry	CORP-WC-0002NC	2.3	10/08/04	Determination of Solids in Water and Wastes MCAWW and SM
Wet Chemistry	CORP-WC-0003	1.2	11/05/04	HEM/SGT-HEM by Method EPA1664
Administration	NC-AD-0001	1.1	05/03/05	Project Management
GC	NC-GC-0003	3	11/19/04	GC Halogenated/Aromatic VOCs EPA601, EPA602
GC	NC-GC-0006	4	01/30/07	Analysis of Polynuclar Aromatic Hydrocarbons EPA Method 610
GC	NC-GC-0007	3	02/27/07	Analysis of Pesticides and PCBs EPA608
GC	NC-GC-0013	2.1	11/17/04	TPH by Wisconsin DNR Modified DRO
GC	NC-GC-0025	4.1	01/09/04	TPH as Gasoline 8015B-Modified
GC	NC-GC-0031	1	11/21/04	Modified GRO Method for Determining Gasoline Range Organics, Wisconsin DNR
GC	NC-GC-0032	2	02/01/07	Analysis of Dissolved Gases in Groundwater by Modified Method RSK-175
GC	NC-GC-0033	0	08/13/04	Vapor Space Organics by Headspace GC/FID

Table 8.2-2
Standard Operating Procedures Listing [1, 2, 3] (Cont'd)

Dept	SOP No.	Revision	Active Date	SOP Title
GC	NC-GC-0035	1	01/31/07	Formaldehyde by High Performance Liquid Chromatography (HPLC), SW846 8315A
GC	NC-GC-0036	1	02/01/07	Aromatic Acids by High Performance Liquid Chromatography (HPLC)
Inorganic Prep	NC-IP-0001	3	05/13/04	Particle Size Reduction of Solid Matrices
Organic Prep	NC-IP-0009	2	06/26/07	DI Leachate Procedure for Solids Internal (RAS) ASTM D3987-85
GC/MS	NC-MS-0003	3	03/13/06	GC/MS Semivolatile Organic Compounds Capillary Column Technique EPA Method 625
GC/MS	NC-MS-0005	1.2	03/24/06	Dioxin Screen
GC/MS	NC-MS-0006	2	02/24/04	GC/MS Volatile Organic Compounds Method 624
GC/MS	NC-MS-0015	3	08/14/06	Analysis of Polynuclear Aromatic Hydrocarbons by Selective Ion Monitoring 8270C
GC/MS	NC-MS-0017	0	04/27/07	Analysis of Volatile Organic Compounds by Selective Ion Monitoring
Metals	NC-MT-0001	5	02/15/05	Prep and Analysis of Mercury in Aqueous and Solid Samples by Cold Vapor Atomic Fluorescence, Method 1631B, 1631C, 1631E & MCAWW 245.7
Metals	NC-MT-0002	4.2	01/08/04	Inductively Coupled Plasma-Mass Spectrometry EPA 6020 & 200.8
Metals	NC-MT-0010	1.3	04/23/07	Hardness by Calculation SM2340B, SW846 Method 6010B
Organic Prep	NC-OP-0021	1.4	04/24/07	Wipe Extraction Method for PCBs
Organic Prep	NC-OP-0024	1.2	06/26/07	Extraction of Organic Compounds From Waters and Soils, Based on Modified DRO Method for Determining Diesel Range Organics, Wisconsin DNR
Q.A.	NC-QA-0004	4	09/30/04	Micropipette Calibration
Q.A	NC-QA-0012	2.4	07/10/07	Shipping Department
Q.A.	NC-QA-0013	1.2	02/20/07	Inventory/Warehouse Control
Q.A	NC-QA-0014	6	02/16/07	Glassware Washing
Q.A.	NC-QA-0015	7	02/14/06	Equipment Monitoring and Thermometer Calibration
Q.A	NC-QA-0016	9.3	04/25/07	Supplemental Practices for Navy Project Work
Q.A.	NC-QA-0017	5.2	03/22/07	Standards and Reagents
Q.A	NC-QA-0018	8	02/15/07	Statistical Evaluation of Data and Development of Control Charts
Q.A.	NC-QA-0019	6	02/20/07	Records Information Management
Q.A	NC-QA-0020	1.2	07/07/07	Laboratory Holding Blanks
Q.A.	NC-QA-0021	5	04/25/07	Evaluation of Method Detection Limits for Chemical Tests
Q.A	NC-QA-0025	1	12/10/04	Performance Checks on Spectronic Model 21 and Model 1001 Spectrophotometers
Q.A.	NC-QA-0027	1.2	07/15/07	Preparation and Management of Standard Operataing Procedures

Table 8.2-2
Standard Operating Procedures Listing [1, 2, 3] (Cont'd)

Dept	SOP No.	Revision	Active Date	SOP Title
Sample Control	NC-SC-0005	6.4	10/02/06	Sample Receiving and Sample Control
Sample Control	NC-SC-0006	2	05/28/03	Sample Procurement Protocol
Wet Chemistry	NC-WC-0001	5	01/31/06	Nitrite, Nitrate/Nitrite, Nitrate Automated EPA353.2
Wet Chemistry	NC-WC-0003 (Also select NC-WC-0006)	2.2	12/07/04	Alkalinity-Carbonate, Bicarbonate and Hydroxide SM2320B, SM4500-CO2D
Wet Chemistry	NC-WC-0004	3.2	02/02/06	Total Solids, Percent Moisture, Total Settleable Solids, Ash, and Total Volatile Solids
Wet Chemistry	NC-WC-0006 (Also select NC-WC-0003)	6	01/31/06	Alkalinity (Total) EPA 310.1 SM2320B
Wet Chemistry	NC-WC-0007	2.5	05/10/07	Phenolics (Manual Sectrophotometer) - EPA420.1, SW9065
Wet Chemistry	NC-WC-0008	3.4	10/28/04	Demand, Biochemical Oxygen (BOD), Carbonaceous (CBOD) SM5210B, EPA405.1
Wet Chemistry	NC-WC-0009	2	10/25/04	pH Paper Method SW9041B
Wet Chemistry	NC-WC-00090	1.1	12/16/04	Preparation and Analysis of Inorganics Arsenic Species in Aqueous Samples, Soils and Sediments by Anodic Stripping Voltammetry Modified Method 7063
Wet Chemistry	NC-WC-0010	7	03/23/06	pH Electrometric Method SW9040B, SW9045C, EPA150.1
Wet Chemistry	NC-WC-0011	2	02/20/04	Acidity
Wet Chemistry	NC-WC-0013	2.2	11/08/04	Chloride (Automated Ferricyanide) EPA325.2, SW9251
Wet Chemistry	NC-WC-0014	2	11/06/04	Dissolved Oxygen
Wet Chemistry	NC-WC-0017	2.3	12/16/04	Carbon, Total Organic EPA415.1, SW9060, SM5310D
Wet Chemistry	NC-WC-0018	2.2	10/26/04	Carbon, Total Organic (TOC - Analysis for Non-Waters) Walkley-Black
Wet Chemistry	NC-WC-0019	5.2	10/11/04	Demand, Chemical Oxygen (COD) (Colorimetric) SM5220D, EPA410.4
Wet Chemistry	NC-WC-0021	1.2	12/06/04	Chlorine Total (Residual) and Free EPA330.5, SM 4500-CIG
Metals	NC-WC-0023	2.2	10/28/04	Cation-Exchange Capacity
Wet Chemistry	NC-WC-0024	3.3	10/08/04	Chromium, Hexavalent (Colorimetric) SW7196A, SM3500-CR
Wet Chemistry	NC-WC-0025	2.2	10/27/04	Iron, Ferrous SM3500-Fe D
Wet Chemistry	NC-WC-0031 (Also select NC-WC-0032)	8	11/08/04	Cyanide, Automated Pyridine-Barbituric Acid SW9012A, EPA335.1, 335.2, SM4500CN-I CLP
Wet Chemistry	NC-WC-0032 (Also select NC-WC-0031)	8.4	12/08/04	Cyanide, Preparation Method SW9012A, EPA335.1, 335.2, SM4500CN-I, SM4500CN-E, CLP ILM03.0
Wet Chemistry	NC-WC-0034	1	09/25/03	Flashpoint (Closed Cup) ASTM D93-85, SW846 Method 1010

Table 8.2-2
Standard Operating Procedures Listing [1, 2, 3] (Cont'd)

Dept	SOP No.	Revision	Active Date	SOP Title
Wet Chemistry	NC-WC-0035	3	11/02/04	Fluoride (ISE) - (analysis, all matrices) - EPA 340.2
Wet Chemistry	NC-WC-0036	3.1	11/06/04	Hardness, Total (mg/L as CaCO3) EPA 130.2
Wet Chemistry	NC-WC-0038	4.2	02/08/06	Nitrogen, Ammonia (ISE) - (Water) EPA350.3
Wet Chemistry	NC-WC-0039	2.3	05/03/07	Nitrogen, Ammonia Distillation/Titration (Solid) - EPA350.2
Wet Chemistry	NC-WC-0040	1.3	10/27/04	Nitrogen, Total Kjeldahl (TKN) EPA351.3
Wet Chemistry	NC-WC-0041	1	12/09/04	Nitrogen, Total Organic (TON) EPA350.2, 351.3
Wet Chemistry	NC-WC-0046	1	09/25/03	Paint Filter SW9095
Wet Chemistry	NC-WC-0050	2.6	11/04/04	Phosphorous: Total,Ortho and Organic - EPA365.2/365.3 SM4500P
Wet Chemistry	NC-WC-0058	8	11/06/04	Sulfate (Turbidimetric) EPA375.4, SW846 Method 9038
Wet Chemistry	NC-WC-0060	4	09/02/04	Sulfide - SW846 9030A, EPA 376.1
Wet Chemistry	NC-WC-0063	1	12/13/04	Sulfite (Titrimetric)
Wet Chemistry	NC-WC-0067	4.1	04/24/07	Halogens, Total Organic (TOX) SW9020A, EPA450.1
Wet Chemistry	NC-WC-0068	4	11/08/04	Turbidity EPA 180.1
Wet Chemistry	NC-WC-0070	1	11/06/04	Decanting Procedure for Aqueous Sample
Wet Chemistry	NC-WC-0071	1.2	11/04/04	Gravity, Specific SM2710F
Wet Chemistry	NC-WC-0079	4	09/30/04	Conductivity (Specific Conductance) EPA120.1, SM2510B, SW9050
Wet Chemistry	NC-WC-0083	1.2	11/08/04	Demand, Chemical Oxygen (COD - Low Level) SM5220D, EPA410.4
Wet Chemistry	NC-WC-0084	5	06/28/05	Ion Chromatography, Determination of Inorganic Anions by EPA300.0
Wet Chemistry	NC-WC-0086	1.1	12/06/04	Alkaline Digestion For Hexavalent Chromium SW846 3060A, ASTM Method D1498
Wet Chemistry	NC-WC-0087	1.1	10/06/03	Spreadsheet Upload SOP, Wet Chemistry
Wet Chemistry	NC-WC-0089	0	09/24/04	Extractable Organic Halides (EOX) in Solids SW846 Method 9023
Information Technology	P-I-0005R2	2	10/11/04	Software Licensing Policy
IT Policy	P-I-001	2	05/20/04	Internet Use Policy
IT Policy	P-I-002	2	05/20/04	Electronic Mail (Email) Use
IT Policy	P-I-003	2	05/20/04	Computer Systems Account and Naming Policy
IT Policy	P-I-004	2	05/20/04	Computer Systems Password Policy
IT Policy	P-I-006	2	10/11/04	Virus Protection Policy
IT Policy	P-I-007	2	10/11/04	Data Backup Policy
IT Policy	P-I-008	2	10/11/04	Internet Security Policy
IT Policy	P-I-009	1	06/17/04	Cellular Phone Policy

Table 8.2-2
Standard Operating Procedures Listing [1, 2, 3] (Cont'd)

Dept	SOP No.	Revision	Active Date	SOP Title
Information Technology	P-ITQ-013R1	1	01/27/03	Software Quality Assurance Policy
Legal Policy	P-L-001	1	07/27/04	Record Retention
Legal Policy	P-L-002	2	04/28/04	Subpoenas
Legal Policy	P-L-003	1.1	02/03/05	Internal Investigations
Legal Policy	P-L-004	1	09/16/04	Organizational Conflict of Interest
Legal Policy	P-L-006	4.1	02/03/05	TestAmerica Ethics Policy
Q.A.	P-T-001	3	09/08/04	Selection of Calibration Points
Q.A.	QA-003	5	09/27/04	TestAmerica North Canton Quality Control Program
Q.A.	QA-010	4	06/26/07	Maintaining Time Integrity
Q.A.	QA-014	2	12/03/04	Instrument Detection Limit, Determination of
Q.A.	QA-020	2	10/29/04	Data Validation Response
Information Technology	S-ITQ-0001R1	1	02/05/03	Software and Hardware Change Management
Information Technology	S-ITQ-0005R1	1	02/05/03	QuantIMS User Profile Setup and Maintenance
Information Technology	S-ITQ-0007R1	1	01/27/03	Software Testing Validation Verification
Q.A.	S-Q-002	3.1	02/03/05	Systems Audits
Q.A.	S-Q-001	5	12/15/06	Official Document Control and Archive
Q.A.	S-Q-003	2	10/17/04	MDL Policy
Q.A.	S-Q-004	3	12/15/06	Manual Integrations Procedure
Q.A.	S-Q-005	1	07/14/04	Data Recall Process
Q.A.	S-Q-007	1.1	02/03/05	Data Authenticity Audit Procedure
Q.A.	S-T-002	2	11/08/04	Reporting Limits for QuantIMS R1
Q.A.	S-T-001	2	11/08/04	Testing of Solvents and Acids
Finance,Purch, & Procurement	S-Pu-002	2	08/29/05	Returns - Vendor Performance
Finance,Purch, & Procurement	P-Pu-006	1	08/29/05	Procurement and Contracts Policy
Finance,Purch, & Procurement	P-Pu-007	0	08/29/05	Vendor Selection Procedure

[1] This listing is not all inclusive of all Corporate Policies and SOPs.
[2] SOPs and Policies are listed in electronic directories, where the latest version is always available.
[3] This SOP table lists the latest SOPs and Policies available at the time of this document's publication.

TABLE 8.2-3
Wet Chemistry Methods [1]

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Acidity	Water	305.[2] SM 2310 B	--	--
Alkalinity, Bicarbonate, Carbonate	Water	305.[2] SM 2320 B		
	Solid	EPA 310.1[2] (M)	--	--
Arsenic (ASV) Anodic Stripping Voltammetry	Water	--	EPA 7063	--
Ash Content	Solid	--	--	ASTM D29-74
Biochemical Oxygen Demand, Carbonaceous	Water	EPA 405.1 SM 5210 B	--	--
Bromide	Water	EPA 300.0A	EPA 9056A	--
	Waste	EPA 300.0A	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A	--
Cation-Exchange Capacity	Solid	--	EPA 9081	--
Chemical Oxygen Demand	Water	EPA 410.4 SM 5220D	--	--
	Waste	EPA 410.4	--	--
Chloride	Water	EPA 300.0A EPA 325.2[2]	EPA 9056A EPA 9251	EPA 325.2[2]
	Waste	EPA 300.0A	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A EPA 9251(M)	--
Chromium, Hexavalent	Water	EPA 3500-Cr-D	EPA 7196A	--
	Waste	EPA 3500-Cr-D	EPA 7196A	--
	Solid	--	EPA 3060A EPA 7196A	--

[1] Any matrix not listed is not applicable for the associated method
[2] Removed from 40CFR

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Specific Conductance	Water	EPA 120.1 SM 2510B	EPA 9050A	--
	Waste	EPA 120.1	EPA 9050A	--
	Solid	--	EPA 9050A	--
Chlorine, Residual	Water	EPA 330.5[2] SM 3500 CL-G	--	--
Cyanide (Amenable)	Water	EPA 335.1[2] SM 4500 CN-G	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Total)	Water	SM 4500-CN E EPA 335.4	EPA 9012A	---
	Waste	--	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Weak and Dissociable) (Free)	Water	SM 4500-CN I	--	--
Dissolved Oxygen	Water	360.1[2] SM 4500 O-G	--	--
Flash Point	Waste	--	EPA 1010	ASTM D93-9
	Solid	--	EPA 1010	ASTM D93-9
Fluoride	Water	EPA 300.0A EPA 340.2[2]	EPA 9056A	SM 4500 F-C, ISE
	Waste	EPA 340.2 (M)[2] EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 340.2 (M)[2] EPA 300.0A (M)	EPA 9056A	--
Iron, Ferrous & Ferric	Water	SM 3500 FE D	--	--
Hardness	Water	EPA 130.2[2]	--	SM 2340B
Moisture	Solid	---	EPA 160.3 (M) ASTM D2216-90	---
Nitrogen, Ammonia	Water	EPA 350.1	--	EPA 350.2[2]
	Waste	EPA 350.1	--	EPA 350.2[2]
	Solid	EPA 350.1	--	EPA 350.2[2]
	Water	SM 4500 NH_3-B (Distillation)	--	--
	Water	SM 4500 NH_3-E (Titration)	--	--
	Water	SM 4500 NH_3-F (ISE)	--	--

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Nitrite (NO_2)	Water	EPA 300.0A	EPA 9056A	--
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A	--
Nitrate (NO_3)	Water	EPA 300.0A	EPA 9056A	SM 4500 NO_3-E
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M)	--	--
Nitrate plus Nitrite NO_2/NO_3	Water	EPA 353.2	--	--
	Waste	EPA 353.2	--	--
Total Kjeldahl Nitrogen (TKN)	Water	EPA 351.3	--	SM 4500 NO_3
	Waste	EPA 351.3	--	--
	Solid	EPA 351.3	--	--
Oil and Grease (Hexane Extractable Material)	Water	EPA 1664A	EPA 9071B	--
	Waste	EPA 1664A	EPA 9071B	--
	Solid	--	EPA 9071B	--
Ortho-phosphate o-PO_4	Water	EPA 300.0A EPA 365.1	EPA 9056A	SM 4500 P-E
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M) EPA 365.1	EPA 9056A	--
pH	Water	EPA 150.1[2]	EPA 9040B	EPA 9041
	Waste	SM 4500 H-B	EPA 9045C	--
	Solid	---	EPA 9045C	--
Paint Filter	Water	--	EPA 9095A	--
Phenolics	Water	EPA 420.1	--	--
	Waste	--	EPA 9065	--
	Solid	--	EPA 9065	--
Phosphorus (Total)	Water	EPA 365.1	--	SM 4500 P-E
	Waste	EPA 365.1	--	--
	Solid	EPA 365.1	--	--
Sulfate (SO_4)	Water	EPA 300.0A EPA 375.4[2]	EPA 9056A EPA 9038	--
	Waste	EPA 300.0A (M) EPA 375.4[2]	EPA 9056A EPA 9038	--
	Solid	EPA 300.0A (M)	EPA 9056A EPA 9038 (M)	--

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA	Other
Sulfide	Water	EPA 376.1[2]	EPA 9030A SM 4500	9030B/9034
Total Organic Carbon (TOC)	Water	EPA 415.1[2]	EPA 9060	SM 5310 D
	Waste	--	EPA 9060	--
	Solid	EPA 415.1 (M)	EPA 9060 (M)	Walkley-Black
Total Organic Halides (TOX)	Water	--	EPA 9020B EPA 9023(EOX)	EPA 450.1
	Waste	--	--	--
	Solid	--	EPA 9020B	--
Total Petroleum Hydrocarbons	Water	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Waste	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Solid	--	EPA 9071B	--
Total Solids	Water	EPA 160.3	--	--
	Waste	EPA 160.3	--	--
	Solid	EPA 160.3 (M)	--	--
Total Dissolved Solids	Water	EPA 160.1	--	2540E
Total Suspended Solids	Water	EPA 160.2	---	2540E
Volatile and Volatile Suspended Solids	Water	EPA 160.4	--	--
Settleable Solids	Water	EPA 160.5	--	--
Turbidity	Water	EPA 180.1	--	--

TABLE 8.2-4
Methods for Mercury by Cold Vapor Atomic Absorption

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Mercury (CVAA)	Water	EPA 245.1	EPA 7470A	--
	TCLP Leachate	--	EPA 7470A	--
	Waste	--	EPA 7471A	--
	Solid	EPA 254.5	EPA 7471A	--

TABLE 8.2-5
Methods for Mercury by Cold Vapor Atomic Fluororescence

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water	EPA 245.7	--	EPA 1631E

TABLE 8.2-6
Methods for Metals by ICP and ICPMS

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Aluminum	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Antimony	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Arsenic	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Barium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Beryllium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Boron	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	EPA 200.7	EPA 6010B	---
Calcium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	EPA 200.7	EPA 6010B	---

TABLE 8.2-6
Methods for Metals by ICP and ICPMS (cont'd)

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Cadmium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Cobalt	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Chromium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Copper	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Iron	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Lead	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Magnesium	Water	EPA 200.7	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7	EPA 6010B EPA 6020	---

TABLE 8.2-6
Methods for Metals by ICP and ICPMS (cont'd)

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Manganese	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Molybdenum	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Nickel	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Potassium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	---	EPA 6010B	---
Selenium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Silver	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Sodium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	---	EPA 6010B	---
Tin	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

TABLE 8.2-6
Methods for Metals by ICP and ICPMS (cont'd)

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Thallium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Titanium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	---	EPA 6010B	---
Vanadium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	---	EPA 6010B EPA 6020	---
Zinc	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

TABLE 8.2-7
Metals Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water	---	EPA 1311	---
	Waste	---	EPA 1311	---
	Solid	---	EPA 1311	---
ICP Metals	Water	EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate	---	EPA 3010A	---
	Waste	---	EPA 3050B	---
	Solid	---	EPA 3050B	---
ICPMS Metals	Water	EPA 200.8	EPA 3010A	---
	TCLP	---	EPA 3010A	---
	Waste	---	EPA 3050B	---
	Solid	---	EPA 3050B	---
CVAA Mercury	Water	EPA 245.1	EPA 7470A	---
	TCLP Leachate	---	EPA 7470A	---
	Waste	---	EPA 7471A	---
	Solid	---	EPA 7471A	
CVAFS Mercury Low Level	Water	EPA 245.7	---	EPA 1631E

TABLE 8.2-8
Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	EPA 624	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Halogenated Volatiles by GC	Water	EPA 601	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Aromatic Volatiles by GC	Water	EPA 602	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Semivolatiles by GC/MS	Water	EPA 625	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---
Pesticides/PCBs by GC	Water	EPA 608	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---

TABLE 8.2-8
Organic Sample Preparation Methods (cont'd)

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Herbicides by GC	Water	EPA 615	EPA 8151A	---
	Waste	---	EPA 8151A	---
	Solid	---	EPA 8151A	---
Total Petroleum Hydrocarbons (Gasoline Range) by GC	Water	---	EPA 5030B	WI GRO
	Waste	---	EPA 5030B EPA 5035	WI GRO
	Solid	---	EPA 5035 EPA 5035	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC	Water	---	EPA 3510C EPA 3520C	WI DRO
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3580A	WI DRO
	Solid	---	EPA 3550B	WI DRO

TABLE 8.2-9
Organic Methods of Analysis

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	EPA 624	EPA 8260B	---
	Waste	---	EPA 8260B	---
	Solid	---	EPA 8260B	---
Halogenated Volatiles by GC	Water	EPA 601	EPA 8021B	---
	Waste	---	EPA 8021B	---
	Solid	---	EPA 8021B	---
Aromatic Volatiles by GC	Water	EPA 602	EPA 8021B	---
	Waste	---	EPA 8021B	---
	Solid	---	EPA 8021B	---
Semivolatiles by GC/MS	Water	EPA 625	EPA 8270C	
	Waste	---	EPA 8270C	---
	Solid	---	EPA 8270C	---
Pesticides/PCBs by GC	Water	EPA 608	Pesticides 8081A PCBs 8082	---
	TCLP Leachate	---	Pesticides 8081A PCBs 8082	---
	Waste	---	Pesticides 8081A PCBs 8082	---
	Solid	---	Pesticides 8081A PCBs 8082	---

TABLE 8.2-9
Organic Methods (cont'd)

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Phenoxyacid Herbicides by GC	Water	---	EPA 8151A	---
	TCLP Leachate	---	EPA 8151A	---
	Waste	---	EPA 8151A	---
	Solid	---	EPA 8151A	---
Gasoline Range Organics by GC	Water	---	EPA 8015B (M)	WI GRO
	Waste	---	EPA 8015B (M)	---
	Solid	---	EPA 8015B (M)	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC/FID	Water	---	EPA 8015B (M)	WI DRO
	Waste	---	EPA 8015B (M)	---
Dissolved Gases RSK-175	Water	---	---	SOP
Formaldehyde Carbonyl Compounds	Water	---	EPA 8315	---

TABLE 8.2-10-1
General Chemistry
Reporting Limits (RL), Method Reference

Test Method	Analyte	RL	Units
305.1/SM2310B	Acidity	5	mg/L
310.1/SM2320B	Alkalinity, Total	5	mg/L
7063	Arsenic Speciation	2	ug/L
300.0 9056A	Bromide Chloride Nitrate Fluoride Sulfate Ortho-Phosphate Nitrite	0.5 1 0.1 1 1 0.5 0.1	mg/L mg/L mg/L mg/L mg/L mg/L mg/L
405.1/SM5210B	Biochemical Oxygen Demand	2	mg/L
410.4/SM5220D	Chemical Oxygen Demand (COD)	10	mg/L
325.2/9251	Chloride, Automated	1	mg/L
330.5 SM4500CL-G	Chlorine, Total Residue	0.2	mg/L
9012A/335.2 335.4 SM4500CN-E	Cyanide, Total	0.01	mg/L
340.2 SM4500F-C, ISE	Fluoride	0.1	mg/L
130.2/SM2340B	Hardness, as CaCO3	5	mg/L
1664A	n-Hexane Extractable Material n-Hexane Extractable Material, SGT	5 10	mg/L mg/L
7196A	Hexavalent Chromium	0.02	mg/L
3500 Cr D	Hexavalent Chromium	0.02	mg/L
353.2 SM4500NO_3E	Nitrite Nitrate Nitrate/Nitrite	0.1 0.1 0.1	mg/L mg/L mg/L
350.2 SM4500NH_3-E,F	Nitrogen, as Ammonia	1	mg/L

TABLE 8.2-10-1
General Chemistry
Reporting Limits (RL), Method Reference

Test Method	Analyte	RL	Units
9065	Phenols, Total	0.02	mg/L
9045B, C SM4500H-B	pH (solid)	--	No Units
4500	Phosphorus as Orthophosphate	0.1	mg/L
365.2 **365.1**	Ortho-Phosphate Phosphorus, as ortho-Phosphate ortho-Phosphate, Dissolved	0.1 0.1 0.1	mg/L mg/L mg/L
120.1 SM2510B	Specific Conductance	1	umhos/cm
9050A	Specific Conductance	1	umhos/cm
160.1 SM2540E	Solids (Residue), Dissolved Filterable Solids, Total Dissolved	10 10	mg/L mg/L
375.4	Sulfate	5	mg/L
9038	Sulfate	5	mg/L
376.1	Sulfide, Total	1	mg/L
9030A	Sulfide, Total	1	mg/L
9030A	Sulfide, Acid-Insoluble	1	mg/L
351.3	Total Kjeldahl Nitrogen	3	mg/L
415.1 SM5310D	Total Organic Carbon	1	mg/L
9060	Total Organic Carbon	1	mg/L
9020B	Total Organic Halogens	30	ug/L
180.1	Turbidity	0.5	NTU

TABLE 8.2-10-2
Metals – ICP, CVAA 6000, 7000, 200 Series
Reporting Limits (RL), Method Reference [1, 2]

	Analyte	RL	Units
Trace Water	Antimony	0.01	mg/L
	Arsenic	0.01	mg/L
	Cadmium	0.002	mg/L
	Chromium	0.005	mg/L
	Cobalt	0.007	mg/L
	Lead	0.003	mg/L
	Molybdenum	0.01	mg/L
	Selenium	0.005	mg/L
	Silver	0.005	mg/L
	Thallium	0.01	mg/L
	Vanadium	0.007	mg/L
Trace Solid	Antimony	1	mg/kg
	Arsenic	1	mg/kg
	Cadmium	0.2	mg/kg
	Chromium	0.5	mg/kg
	Cobalt	5	mg/kg
	Lead	0.3	mg/kg
	Molybdenum	1	mg/kg
	Selenium	0.5	mg/kg
	Silver	0.5	mg/kg
	Thallium	1	mg/kg
	Vanadium	5	mg/kg
Water	Aluminum	0.2	mg/L
	Antimony	0.06	mg/L
	Arsenic	0.3	mg/L
	Barium	0.2	mg/L
	Beryllium	0.005	mg/L
	Boron	0.2	mg/L
	Cadmium	0.005	mg/L
	Calcium	5	mg/L
	Chromium	0.01	mg/L
	Cobalt	0.05	mg/L
	Copper	0.025	mg/L
	Iron	0.1	mg/L
	Lead	0.1	mg/L
	Magnesium	5	mg/L
	Manganese	0.015	mg/L
	Molybdenum	0.04	mg/L
	Nickel	0.04	mg/L
	Potassium	5	mg/L
	Selenium	0.25	mg/L
	Silver	0.01	mg/L
	Sodium	5	mg/L
	Thallium	0.2	mg/L
	Tin	0.1	mg/L
	Titanium	0.05	mg/L
	Vanadium	0.05	Mg/L
	Zinc	0.02	mg/L

TABLE 8.2-10-2
Metals – ICP, CVAA 6000, 7000, 200 Series
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Solid	Aluminum	20	mg/kg
	Antimony	6	mg/kg
	Arsenic	30	mg/kg
	Barium	20	mg/kg
	Beryllium	0.5	mg/kg
	Boron	20	mg/kg
	Cadmium	0.5	mg/kg
	Calcium	500	mg/kg
	Chromium	1	mg/kg
	Cobalt	5	mg/kg
	Copper	2.5	mg/kg
	Iron	10	mg/kg
	Lead	10	mg/kg
	Magnesium	500	mg/kg
	Manganese	1.5	mg/kg
	Molybdenum	4	mg/kg
	Nickel	4	mg/kg
	Potassium	500	mg/kg
	Selenium	25	mg/kg
	Silver	1	mg/kg
	Sodium	500	mg/kg
	Thallium	200	mg/kg
	Tin	10	mg/kg
	Titanium	5	mg/kg
	Vanadium	5	mg/kg
	Zinc	2	mg/kg
Water	Mercury	0.0002	mg/L
Solid	Mercury	0.1	mg/kg

Table 8.2-10-2
Metals – ICPMS, 6020
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water	Aluminum	50	ug/L
	Antimony	2	ug/L
	Arsenic	5	ug/L
	Barium	1	ug/L
	Beryllium	1	ug/L
	Cadmium	1	ug/L
	Chromium	2	ug/L
	Cobalt	1	ug/L
	Copper	2	ug/L
	Iron	20	ug/L
	Lead	1	ug/L
	Manganese	1	ug/L
	Molybdenum*	10	ug/L
	Nickel	2	ug/L
	Selenium*	5	ug/L
	Silver	1	ug/L
	Thallium	1	ug/L
	Tin*	10	ug/L
	Vanadium*	5	ug/L
	Zinc	10	Ug/L

*Molybdenum, Selenium, Tin, and Vanadium are not on the 6020 method list

Solid	Antimony	0.2	mg/kg
	Arsenic	0.5	mg/kg
	Barium	0.1	mg/kg
	Beryllium	0.1	mg/kg
	Cadmium	0.1	mg/kg
	Chromium	0.2	mg/kg
	Cobalt	0.1	mg/kg
	Copper	0.2	mg/kg
	Lead	0.1	mg/kg
	Manganese	0.1	mg/kg
	Molybdenum*	0.2	mg/kg
	Nickel	0.1	mg/kg
	Selenium*	0.5	mg/kg
	Silver	0.1	mg/kg
	Thallium	0.1	mg/kg
	Tin*	1	mg/kg
	Vanadium*	0.5	mg/kg
	Zinc	1	mg/kg

*Molybdenum, Selenium, Tin, and Vanadium are not on the 6020 method list

Table 8.2-10-2
Metals – ICPMS, 200.8
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water	Aluminum	50	ug/L
	Antimony	2	ug/L
	Arsenic	5	ug/L
	Barium	1	ug/L
	Beryllium	1	ug/L
	Cadmium	1	ug/L
	Chromium	2	ug/L
	Cobalt	1	ug/L
	Copper	2	ug/L
	Iron	20	ug/L
	Lead	1	ug/L
	Manganese	1	ug/L
	Molybdenum	10	ug/L
	Nickel	2	ug/L
	Selenium	2	ug/L
	Silver	1	ug/L
	Thallium	1	ug/L
	Tin*	10	ug/L
	Vanadium	5	ug/L
	Zinc	10	ug/L

*Tin is not on the 200.8 method list

Table 8.2-10-2
Metals – Low-Level Mercury, 1631E & 245.7
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
1631E Water	Mercury	0.5	ng/L
245.7 Water	Mercury	5	ng/L

TABLE 8.2-10-3
MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water	Acenaphthene	10	ug/L
	Acenaphthylene	10	ug/L
	Anthracene	10	ug/L
	Benzo(a)anthracene	10	ug/L
	Benzo(b)fluoranthene	10	ug/L
	Benzo(k)fluoranthene	10	ug/L
	Benzo(ghi)perylene	10	ug/L
	Benzo(a)pyrene	10	ug/L
	Bis(2-chloroEthoxy)methane	10	ug/L
	bis(2-Chloroethyl) ether	10	ug/L
	bis(2-Ethylhexyl) phthalate	10	ug/L
	4-Bromophenyl phenyl ether	10	ug/L
	Butyl benzyl phthalate	10	ug/L
	Carbazole	10	ug/L
	4-Chloroaniline	10	ug/L
	4-Chloro-3-methylphenol	10	ug/L
	2-Chloronaphthalene	10	ug/L
	2-Chlorophenol	10	ug/L
	4-Chlorophenyl phenyl ether	10	ug/L
	Chrysene	10	ug/L
	Dibenz(a,h)anthracene	10	ug/L
	Dibenzofuran	10	ug/L
	Di-n-butyl phthalate	10	ug/L
	1,2-Dichlorobenzene	10	ug/L
	1,3-Dichlorobenzene	10	ug/L
	1,4-Dichlorobenzene	10	ug/L
	3,3'-Dichlorobenzidine	50	ug/L
	2,4-Dichlorophenol	10	ug/L
	Diethyl phthalate	10	ug/L
	2,4-Dimethylphenol	10	ug/L
	Dimethyl phthalate	10	ug/L
	4,6-Dinitro-2-methylphenol	50	ug/L
	2,4-Dinitrophenol	50	ug/L
	2,4-Dinitrotoluene	10	ug/L
	2,6-Dinitrotoluene	10	ug/L
	Di-n-octyl phthalate	10	ug/L
	Fluoranthene	10	ug/L
	Fluorene	10	ug/L
	Hexachlorobenzene	10	ug/L
	Hexachlorobutadiene	10	ug/L
	Hexachloro-cyclopentadiene	50	ug/L
	Hexachloroethane	10	ug/L
	Indeno(1,2,3-cd)pyrene	10	ug/L
	Isophorone	10	ug/L
	2-Methylnaphthalene	10	ug/L
	2-Methylphenol	10	ug/L
	4-Methylphenol	10	ug/L
	Naphthalene	10	ug/L
	2-Nitroaniline	50	ug/L

	Analyte	RL	Units
	3-Nitroaniline	50	ug/L
	4-Nitroaniline	50	ug/L
	Nitrobenzene	10	ug/L
	2-Nitrophenol	10	ug/L
	4-Nitrophenol	50	ug/L
	N-Nitrosodiphenylamine	10	ug/L
	N-Nitrosodi-n-propylamine	10	ug/L
	Pentachlorophenol	10	ug/L
	Phenanthrene	10	ug/L
	Phenol	10	ug/L
	2,2'-Oxybis(1-Chloropropane)	10	ug/L
	Pyrene	10	ug/L
	1,2,4-Trichlorobenzene	10	ug/L
	2,4,5-Trichlorophenol	10	ug/L
	2,4,6-Trichlorophenol	10	ug/L
Water Surrogates	2-Fluorobiphenyl		
	2-Fluorophenol		
	2,4,6-Tribromophenol		
	Nitrobenzene-d5		
	Phenol-d5		
	Terphenyl-d14		

TABLE 8.2-10-3
MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
	Acenaphthene	330	ug/kg
	Acenaphthylene	330	ug/kg
	Anthracene	330	ug/kg
	Benzo(a)anthracene	330	ug/kg
	Benzo(b)fluoranthene	330	ug/kg
	Benzo(k)fluoranthene	330	ug/kg
	Benzo(ghi)perylene	330	ug/kg
	Benzo(a)pyrene	330	ug/kg
	bis(2-Chloroethoxy)-methane	330	ug/kg
	bis(2-Chloroethyl) ether	330	ug/kg
	bis(2-Ethylhexyl) phthalate	330	ug/kg
	4-Bromophenyl phenyl ether	330	ug/kg
	Butyl benzyl phthalate	330	ug/kg
	Carbazole	330	ug/kg
	4-Chloroaniline	330	ug/kg
	4-Chloro-3-methylphenol	330	ug/kg
	2-Chloronaphthalene	330	ug/kg
	2-Chlorophenol	330	ug/kg
	4-Chlorophenyl phenyl ether	330	ug/kg
	Chrysene	330	ug/kg
	Dibenz(a,h)anthracene	330	ug/kg
	Dibenzofuran	330	ug/kg
Solid	Di-n-butyl phthalate	330	ug/kg
	1,2-Dichlorobenzene	330	ug/kg
	1,3-Dichlorobenzene	330	ug/kg
	1,4-Dichlorobenzene	330	ug/kg
	3,3'-Dichlorobenzidine	1600	ug/kg
	2,4-Dichlorophenol	330	ug/kg
	Diethyl phthalate	330	ug/kg
	2,4-Dimethylphenol	330	ug/kg
	Dimethyl phthalate	330	ug/kg
	4,6-Dinitro-2-methylphenol	1600	ug/kg
	2,4-Dinitrophenol	1600	ug/kg
	2,4-Dinitrotoluene	330	ug/kg
	2,6-Dinitrotoluene	330	ug/kg
	Di-n-octyl phthalate	330	ug/kg
	Fluoranthene	330	ug/kg
	Fluorene	330	ug/kg
	Hexachlorobenzene	330	ug/kg
	Hexachlorobutadiene	330	ug/kg
	Hexachloro-cyclopentadiene	1600	ug/kg
	Hexachloroethane	330	ug/kg
	Indeno(1,2,3-cd)pyrene	330	ug/kg
	Isophorone	330	ug/kg
	2-Methylnaphthalene	330	ug/kg
	2-Methylphenol	330	ug/kg
	4-Methylphenol	330	ug/kg
	Naphthalene	330	ug/kg
	2-Nitroaniline	1600	ug/kg
	3-Nitroaniline	1600	ug/kg

	Analyte	RL	Units
	4-Nitroaniline	1600	ug/kg
	Nitrobenzene	330	ug/kg
	2-Nitrophenol	330	ug/kg
	4-Nitrophenol	1600	ug/kg
	N-Nitrosodiphenylamine	330	ug/kg
	N-Nitrosodi-n-propylamine	330	ug/kg
	Pentachlorophenol	330	ug/kg
	Phenanthrene	330	ug/kg
	Phenol	330	ug/kg
	2,2'-Oxybis(1-Chloropropane)	330	ug/kg
	Pyrene	330	ug/kg
	1,2,4-Trichlorobenzene	330	ug/kg
	2,4,5-Trichlorophenol	330	ug/kg
	2,4,6-Trichlorophenol	330	ug/kg
Solid Surrogates	2-Fluorobiphenyl		
	2-Fluorophenol		
	2,4,6-Tribromophenol		
	Nitrobenzene-d5		
	Phenol-d5		
	Terphenyl-d14		

TABLE 8.2-10-3
MS Semivolatiles – Method 625
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water	Acenaphthene	10	ug/L
	Acenaphthylene	10	ug/L
	Anthracene	10	ug/L
	Benzo(a)anthracene	10	ug/L
	Benzo(b)fluoranthene	10	ug/L
	Benzo(k)fluoranthene	10	ug/L
	Benzo(ghi)perylene	10	ug/L
	Benzo(a)pyrene	10	ug/L
	bis(2-Chloroethoxy)-methane	10	ug/L
	bis(2-Chloroethyl) ether	10	ug/L
	bis(2-Chloroisopropyl) ether	10	ug/L
	bis(2-Ethylhexyl) phthalate	10	ug/L
	4-Bromophenyl phenyl ether	10	ug/L
	Butyl benzyl phthalate	10	ug/L
	4-Chloro-3-methylphenol	10	ug/L
	2-Chloronaphthalene	10	ug/L
	2-Chlorophenol	10	ug/L
	4-Chlorophenyl phenyl ether	10	ug/L
	Chrysene	10	ug/L
	Dibenz(a,h)anthracene	10	ug/L
	Di-n-butyl phthalate	10	ug/L
	1,2-Dichlorobenzene	10	ug/L
	1,3-Dichlorobenzene	10	ug/L
	1,4-Dichlorobenzene	10	ug/L
	3,3'-Dichlorobenzidine	10	ug/L
	2,4-Dichlorophenol	10	ug/L
	Diethyl phthalate	10	ug/L
	2,4-Dimethylphenol	10	ug/L
	Dimethyl phthalate	10	ug/L
	2-Methyl-4,6-dinitrophenol	50	ug/L
	2,4-Dinitrophenol	50	ug/L
	2,4-Dinitrotoluene	10	ug/L
	2,6-Dinitrotoluene	10	ug/L
	Di-n-octyl phthalate	10	ug/L
	Fluoranthene	10	ug/L
	Fluorene	10	ug/L
	Hexachlorobenzene	10	ug/L
	Hexachlorobutadiene	10	ug/L
	Hexachloroethane	10	ug/L
	Indeno(1,2,3-cd)pyrene	10	ug/L
	Isophorone	10	ug/L
	Naphthalene	10	ug/L
	Nitrobenzene	10	ug/L
	2-Nitrophenol	10	ug/L
	4-Nitrophenol	50	ug/L
	N-Nitrosodi-n-propylamine	10	ug/L
	Pentachlorophenol	10	ug/L
	Phenanthrene	10	ug/L
	Phenol	10	ug/L
	Pyrene	10	ug/L

	Analyte	RL	Units
	1,2,4-Trichlorobenzene	10	ug/L
	2,4,6-Trichlorophenol	10	ug/L
Water Surrogates	2-Fluorobiphenyl		
	2-Fluorophenol		
	2,4,6-Tribromophenol		
	Nitrobenzene-d5		
	Phenol-d5		
	Terphenyl-d14		

TABLE 8.2-10-4
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water 25 mL	Benzene	1	ug/L
	Bromobenzene	1	ug/L
	Bromochloromethane	1	ug/L
	Bromodichloromethane	1	ug/L
	Bromoform	1	ug/L
	Bromomethane	1	ug/L
	n-Butylbenzene	1	ug/L
	sec-Butylbenzene	1	ug/L
	tert-Butylbenzene	1	ug/L
	Carbon tetrachloride	1	ug/L
	Chlorobenzene	1	ug/L
	Chlorodibromomethane	1	ug/L
	Chloroethane	1	ug/L
	Chloroform	1	ug/L
	Chloromethane	1	ug/L
	2-Chlorotoluene	1	ug/L
	4-Chlorotoluene	1	ug/L
	1,2-Dibromo-3-chloropropane	2	ug/L
	1,2-Dibromoethane	1	ug/L
	Dibromomethane	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Dichlorodifluoro-methane	1	ug/L
	1,1-Dichloroethane	1	ug/L
	1,2-Dichloroethane	1	ug/L
	cis-1,2-Dichloroethene	1	ug/L
	trans-1,2-Dichloroethene	1	ug/L
	1,1-Dichloroethene	1	ug/L
	1,2-Dichloropropane	1	ug/L
	1,3-Dichloropropane	1	ug/L
	2,2-Dichloropropane	1	ug/L
	1,1-Dichloropropene	1	ug/L
	Ethylbenzene	1	ug/L
	Hexachlorobutadiene	1	ug/L
	Isopropylbenzene	1	ug/L
	p-Isopropyltoluene	1	ug/L
	Methylene chloride	1	ug/L
	Naphthalene	1	ug/L
	n-Propylbenzene	1	ug/L
	Styrene	1	ug/L
	1,1,1,2-Tetrachloroethane	1	ug/L
	1,1,2,2-Tetrachloroethane	1	ug/L
	Tetrachloroethene	1	ug/L
	Toluene	1	ug/L
	1,2,3-Trichlorobenzene	1	ug/L
	1,2,4-Trichlorobenzene	1	ug/L
	1,1,1-Trichloroethane	1	ug/L
	1,1,2-Trichloroethane	1	ug/L
	Trichloroethene	1	ug/L
	Trichlorofluoromethane	1	ug/L

	Analyte	RL	Units
	1,2,3-Trichloropropane	1	ug/L
	1,2,4-Trimethylbenzene	1	ug/L
	1,3,5-Trimethylbenzene	1	ug/L
	Vinyl chloride	1	ug/L
	m-Xylene & p-Xylene	1	ug/L
	o-Xylene	1	ug/L
Water 25 mL Surrogates	4-Bromofluorobenzene		
	1,2-Dichloroethane-d4		
	Toluene-d8		
	Dibromofluoromethane		

TABLE 8.2-10-4
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
	Benzene	5	ug/L
	Bromobenzene	5	ug/L
	Bromochloromethane	5	ug/L
	Bromodichloromethane	5	ug/L
	Bromoform	5	ug/L
	Bromomethane	5	ug/L
	n-Butylbenzene	5	ug/L
	sec-Butylbenzene	5	ug/L
	tert-Butylbenzene	5	ug/L
	Carbon tetrachloride	5	ug/L
	Chlorobenzene	5	ug/L
	Chlorodibromomethane	5	ug/L
	Chloroethane	5	ug/L
	Chloroform	5	ug/L
	Chloromethane	5	ug/L
	2-Chlorotoluene	5	ug/L
	4-Chlorotoluene	5	ug/L
	1,2-Dibromo-3-chloropropane	10	ug/L
	1,2-Dibromoethane	5	ug/L
	Dibromomethane	5	ug/L
Water	1,2-Dichlorobenzene	5	ug/L
5 mL	1,3-Dichlorobenzene	5	ug/L
	1,4-Dichlorobenzene	5	ug/L
	Dichlorodifluoro-methane	5	ug/L
	1,1-Dichloroethane	5	ug/L
	1,2-Dichloroethane	5	ug/L
	cis-1,2-Dichloroethene	5	ug/L
	trans-1,2-Dichloroethene	5	ug/L
	1,1-Dichloroethene	5	ug/L
	1,2-Dichloropropane	5	ug/L
	1,3-Dichloropropane	5	ug/L
	2,2-Dichloropropane	5	ug/L
	1,1-Dichloropropene	5	ug/L
	Ethylbenzene	5	ug/L
	Hexachlorobutadiene	5	ug/L
	Isopropylbenzene	5	ug/L
	p-Isopropyltoluene	5	ug/L
	Methylene chloride	5	ug/L
	Naphthalene	5	ug/L
	n-Propylbenzene	5	ug/L
	Styrene	5	ug/L
	1,1,1,2-Tetrachloroethane	5	ug/L
	1,1,2,2-Tetrachloroethane	5	ug/L
	Tetrachloroethene	5	ug/L
	Toluene	5	ug/L
	1,2,3-Trichlorobenzene	5	ug/L
	1,2,4-Trichlorobenzene	5	ug/L
	1,1,1-Trichloroethane	5	ug/L
	1,1,2-Trichloroethane	5	ug/L
	Trichloroethene	5	ug/L
	Trichlorofluoromethane	5	ug/L

	Analyte	RL	Units
	1,2,3-Trichloropropane	5	ug/L
	1,2,4-Trimethylbenzene	5	ug/L
	1,3,5-Trimethylbenzene	5	ug/L
	Vinyl chloride	5	ug/L
	m-Xylene & p-Xylene	10	ug/L
	o-Xylene	5	ug/L
Surrogates	4-Bromofluorobenzene		
	1,2-Dichloroethane-d4		
	Toluene-d8		
	Dibromofluoromethane		

TABLE 8.2-10-4
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Encore	Benzene	250	ug/kg
	Bromobenzene	250	ug/kg
	Bromochloromethane	250	ug/kg
	Bromodichloromethane	250	ug/kg
	Bromoform	250	ug/kg
	Bromomethane	250	ug/kg
	n-Butylbenzene	250	ug/kg
	sec-Butylbenzene	250	ug/kg
	tert-Butylbenzene	250	ug/kg
	Carbon tetrachloride	250	ug/kg
	Chlorobenzene	250	ug/kg
	Chlorodibromomethane	250	ug/kg
	Chloroethane	250	ug/kg
	Chloroform	250	ug/kg
	Chloromethane	250	ug/kg
	2-Chlorotoluene	250	ug/kg
	4-Chlorotoluene	250	ug/kg
	1,2-Dibromo-3-chloropropane	500	ug/kg
	1,2-Dibromoethane	250	ug/kg
	Dibromomethane	250	ug/kg
	1,2-Dichlorobenzene	250	ug/kg
	1,3-Dichlorobenzene	250	ug/kg
	1,4-Dichlorobenzene	250	ug/kg
	Dichlorodifluoro-methane	250	ug/kg
	1,1-Dichloroethane	250	ug/kg
	1,2-Dichloroethane	250	ug/kg
	cis-1,2-Dichloroethene	250	ug/kg
	trans-1,2-Dichloroethene	250	ug/kg
	1,1-Dichloroethene	250	ug/kg
	1,2-Dichloropropane	250	ug/kg
	1,3-Dichloropropane	250	ug/kg
	2,2-Dichloropropane	250	ug/kg
	1,1-Dichloropropene	250	ug/kg
	Ethylbenzene	250	ug/kg
	Hexachlorobutadiene	250	ug/kg
	Isopropylbenzene	250	ug/kg
	p-Isopropyltoluene	250	ug/kg
	Methylene chloride	250	ug/kg
	Naphthalene	250	ug/kg
	n-Propylbenzene	250	ug/kg
	Styrene	250	ug/kg
	1,1,1,2-Tetrachloroethane	250	ug/kg
	1,1,2,2-Tetrachloroethane	250	ug/kg
	Tetrachloroethene	250	ug/kg
	Toluene	250	ug/kg
	1,2,3-Trichlorobenzene	250	ug/kg
	1,2,4-Trichlorobenzene	250	ug/kg
	1,1,1-Trichloroethane	250	ug/kg
	1,1,2-Trichloroethane	250	ug/kg
	Trichloroethene	250	ug/kg

	Analyte	RL	Units
	Trichlorofluoromethane	250	ug/kg
	1,2,3-Trichloropropane	250	ug/kg
	1,2,4-Trimethylbenzene	250	ug/kg
	1,3,5-Trimethylbenzene	250	ug/kg
	Vinyl chloride	250	ug/kg
	m-Xylene & p-Xylene	500	ug/kg
	o-Xylene	250	ug/kg
Surrogates	4-Bromofluorobenzene		
	1,2-Dichloroethane-d4		
	Toluene-d8		
	Dibromofluoromethane		

TABLE 8.2-10-4
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
	Benzene	5	ug/kg
	Bromobenzene	5	ug/kg
	Bromochloromethane	5	ug/kg
	Bromodichloromethane	5	ug/kg
	Bromoform	5	ug/kg
	Bromomethane	5	ug/kg
	n-Butylbenzene	5	ug/kg
	sec-Butylbenzene	5	ug/kg
	tert-Butylbenzene	5	ug/kg
	Carbon tetrachloride	5	ug/kg
	Chlorobenzene	5	ug/kg
	Chlorodibromomethane	5	ug/kg
	Chloroethane	5	ug/kg
	Chloroform	5	ug/kg
	Chloromethane	5	ug/kg
	2-Chlorotoluene	5	ug/kg
	4-Chlorotoluene	5	ug/kg
	1,2-Dibromo-3-chloropropane	10	ug/kg
	1,2-Dibromoethane	5	ug/kg
Low Level Encore	Dibromomethane	5	ug/kg
	1,2-Dichlorobenzene	5	ug/kg
	1,3-Dichlorobenzene	5	ug/kg
	1,4-Dichlorobenzene	5	ug/kg
	Dichlorodifluoro-methane	5	ug/kg
	1,1-Dichloroethane	5	ug/kg
	1,2-Dichloroethane	5	ug/kg
	cis-1,2-Dichloroethene	5	ug/kg
	trans-1,2-Dichloroethene	5	ug/kg
	1,1-Dichloroethene	5	ug/kg
	1,2-Dichloropropane	5	ug/kg
	1,3-Dichloropropane	5	ug/kg
	2,2-Dichloropropane	5	ug/kg
	1,1-Dichloropropene	5	ug/kg
	Ethylbenzene	5	ug/kg
	Hexachlorobutadiene	5	ug/kg
	Isopropylbenzene	5	ug/kg
	p-Isopropyltoluene	5	ug/kg
	Methylene chloride	5	ug/kg
	Naphthalene	5	ug/kg
	n-Propylbenzene	5	ug/kg
	Styrene	5	ug/kg
	1,1,1,2-Tetrachloroethane	5	ug/kg
	1,1,2,2-Tetrachloroethane	5	ug/kg
	Tetrachloroethene	5	ug/kg
	Toluene	5	ug/kg
	1,2,3-Trichlorobenzene	5	ug/kg
	1,2,4-Trichlorobenzene	5	ug/kg
	1,1,1-Trichloroethane	5	ug/kg
	1,1,2-Trichloroethane	5	ug/kg
	Trichloroethene	5	ug/kg
	Trichlorofluoromethane	5	ug/kg

	Analyte	RL	Units
	1,2,3-Trichloropropane	5	ug/kg
	1,2,4-Trimethylbenzene	5	ug/kg
	1,3,5-Trimethylbenzene	5	ug/kg
	Vinyl chloride	5	ug/kg
	m-Xylene & p-Xylene	10	ug/kg
	o-Xylene	5	ug/kg
Surrogates	4-Bromofluorobenzene		
	1,2-Dichloroethane-d4		
	Toluene-d8		
	Dibromofluoromethane		

TABLE 8.2-10-4
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Frozen Encore	Benzene	5	ug/kg
	Bromobenzene	5	ug/kg
	Bromochloromethane	5	ug/kg
	Bromodichloromethane	5	ug/kg
	Bromoform	5	ug/kg
	Bromomethane	5	ug/kg
	n-Butylbenzene	5	ug/kg
	sec-Butylbenzene	5	ug/kg
	tert-Butylbenzene	5	ug/kg
	Carbon tetrachloride	5	ug/kg
	Chlorobenzene	5	ug/kg
	Chlorodibromomethane	5	ug/kg
	Chloroethane	5	ug/kg
	Chloroform	5	ug/kg
	Chloromethane	5	ug/kg
	2-Chlorotoluene	5	ug/kg
	4-Chlorotoluene	5	ug/kg
	1,2-Dibromo-3-chloropropane	10	ug/kg
	1,2-Dibromoethane	5	ug/kg
	Dibromomethane	5	ug/kg
	1,2-Dichlorobenzene	5	ug/kg
	1,3-Dichlorobenzene	5	ug/kg
	1,4-Dichlorobenzene	5	ug/kg
	Dichlorodifluoro-methane	5	ug/kg
	1,1-Dichloroethane	5	ug/kg
	1,2-Dichloroethane	5	ug/kg
	cis-1,2-Dichloroethene	5	ug/kg
	trans-1,2-Dichloroethene	5	ug/kg
	1,1-Dichloroethene	5	ug/kg
	1,2-Dichloropropane	5	ug/kg
	1,3-Dichloropropane	5	ug/kg
	2,2-Dichloropropane	5	ug/kg
	1,1-Dichloropropene	5	ug/kg
	Ethylbenzene	5	ug/kg
	Hexachlorobutadiene	5	ug/kg
	Isopropylbenzene	5	ug/kg
	p-Isopropyltoluene	5	ug/kg
	Methylene chloride	5	ug/kg
	Naphthalene	5	ug/kg
	n-Propylbenzene	5	ug/kg
	Styrene	5	ug/kg
	1,1,1,2-Tetrachloroethane	5	ug/kg
	1,1,2,2-Tetrachloroethane	5	ug/kg
	Tetrachloroethene	5	ug/kg
	Toluene	5	ug/kg
	1,2,3-Trichlorobenzene	5	ug/kg
	1,2,4-Trichlorobenzene	5	ug/kg
	1,1,1-Trichloroethane	5	ug/kg
	1,1,2-Trichloroethane	5	ug/kg
	Trichloroethene	5	ug/kg

	Analyte	RL	Units
	Trichlorofluoromethane	5	ug/kg
	1,2,3-Trichloropropane	5	ug/kg
	1,2,4-Trimethylbenzene	5	ug/kg
	1,3,5-Trimethylbenzene	5	ug/kg
	Vinyl chloride	5	ug/kg
	m-Xylene & p-Xylene	10	ug/kg
	o-Xylene	5	ug/kg
Surrogates	4-Bromofluorobenzene		
	1,2-Dichloroethane-d4		
	Toluene-d8		
	Dibromofluoromethane		

TABLE 8.2-10-4
MS Volatiles – Method 624
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water	Benzene	5	ug/L
	Bromodichloro-methane	5	ug/L
	Bromoform	5	ug/L
	Bromomethane	55	ug/L
	Carbon tetrachloride	5	ug/L
	Chlorobenzene	5	ug/L
	Dibromochloro-methane	5	ug/L
	Chloroethane	5	ug/L
	2-Chloroethyl vinyl ether	--	ug/L
	Chloroform	5	ug/L
	Chloromethane	5	ug/L
	1,2-Dichlorobenzene	5	ug/L
	1,3-Dichlorobenzene	5	ug/L
	1,4-Dichlorobenzene	5	ug/L
	1,1-Dichloroethane	5	ug/L
	1,2-Dichloroethane	5	ug/L
	trans-1,2-Dichloroethene	5	ug/L
	1,1-Dichloroethene	5	ug/L
	1,2-Dichloropropane	5	ug/L
	cis-1,3-Dichloropropene	5	ug/L
	trans-1,3-Dichloropropene	5	ug/L
	Ethylbenzene	5	ug/L
	Methylene chloride	5	ug/L
	1,1,2,2-Tetrachloroethane	5	ug/L
	Tetrachloroethene	5	ug/L
	Toluene	5	ug/L
	1,1,1-Trichloroethane	5	ug/L
	1,1,2-Trichloroethane	5	ug/L
	Trichloroethene	5	ug/L
	Trichlorofluoro-methane	5	ug/L
	Vinyl chloride	5	ug/L
Surrogates	Bromofluorobenzene		
	1,2-Dichloro-ethane-d4		
	Toluene-d8		

TABLE 8.2-10-5
GC Semivolatiles – Method 8081A
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water	Aldrin	0.05	ug/L
	alpha-BHC	0.05	ug/L
	beta-BHC	0.05	ug/L
	delta-BHC	0.05	ug/L
	gamma-BHC (Lindane)	0.05	ug/L
	alpha-Chlordane	0.05	ug/L
	gamma-Chlordane	0.05	ug/L
	4,4'-DDD	0.05	ug/L
	4,4'-DDE	0.05	ug/L
	4,4'-DDT	0.05	ug/L
	Dieldrin	0.05	ug/L
	Endosulfan I	0.05	ug/L
	Endosulfan II	0.05	ug/L
	Endosulfan sulfate	0.05	ug/L
	Endrin	0.05	ug/L
	Endrin aldehyde	0.05	ug/L
	Endrin ketone	0.05	ug/L
	Heptachlor	0.05	ug/L
	Heptachlor epoxide	0.05	ug/L
	Methoxychlor	0.1	ug/L
	Toxaphene	2	ug/L
Surrogates	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

TABLE 8.2-10-5
GC Semivolatiles – 8081A
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Solid	Aldrin	1.7	ug/kg
	alpha-BHC	1.7	ug/kg
	beta-BHC	1.7	ug/kg
	delta-BHC	1.7	ug/kg
	gamma-BHC (Lindane)	1.7	ug/kg
	alpha-Chlordane	1.7	ug/kg
	gamma-Chlordane	1.7	ug/kg
	4,4'-DDD	1.7	ug/kg
	4,4'-DDE	1.7	ug/kg
	4,4'-DDT	1.7	ug/kg
	Dieldrin	1.7	ug/kg
	Endosulfan I	1.7	ug/kg
	Endosulfan II	1.7	ug/kg
	Endosulfan sulfate	1.7	ug/kg
	Endrin	1.7	ug/kg
	Endrin aldehyde	1.7	ug/kg
	Endrin ketone	1.7	ug/kg
	Heptachlor	1.7	ug/kg
	Heptachlor epoxide	1.7	ug/kg
	Methoxychlor	3.3	ug/kg
	Toxaphene	67	ug/kg
Surrogates	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

TABLE 8.2-10-5
GC Semivolatiles – Method 8082
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water	Aroclor 1016	1	ug/L
	Aroclor 1221	1	ug/L
	Aroclor 1232	1	ug/L
	Aroclor 1242	1	ug/L
	Aroclor 1248	1	ug/L
	Aroclor 1254	1	ug/L
	Aroclor 1260	1	ug/L
	Aroclor 1262	1	ug/L
	Aroclor 1268	1	ug/L
Surrogates	Decachlorobi-phenyl		
	Tetrachloro-m-xylene		
Solid	Aroclor 1016	33	ug/kg
	Aroclor 1221	33	ug/kg
	Aroclor 1232	33	ug/kg
	Aroclor 1242	33	ug/kg
	Aroclor 1248	33	ug/kg
	Aroclor 1254	33	ug/kg
	Aroclor 1260	33	ug/kg
	Aroclor 1262	33	ug/kg
	Aroclor 1268	33	ug/kg
Surrogates	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

TABLE 8.2-10-5
GC Semivolatiles – Method 608
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water	Aldrin	0.05	ug/L
	alpha-BHC	0.05	ug/L
	beta-BHC	0.05	ug/L
	delta-BHC	0.05	ug/L
	gamma-BHC (Lindane)	0.05	ug/L
	Chlordane (technical)	0.5	ug/L
	4,4'-DDD	0.05	ug/L
	4,4'-DDE	0.05	ug/L
	4,4'-DDT	0.05	ug/L
	Dieldrin	0.05	ug/L
	Endosulfan I	0.05	ug/L
	Endosulfan II	0.05	ug/L
	Endosulfan sulfate	0.05	ug/L
	Endrin	0.05	ug/L
	Endrin aldehyde	0.05	ug/L
	Heptachlor	0.05	ug/L
	Heptachlor epoxide	0.05	ug/L
	PCB-1016	1	ug/L
	PCB-1221	1	ug/L
	PCB-1232	1	ug/L
	PCB-1242	1	ug/L
	PCB-1248	1	ug/L
	PCB-1254	1	ug/L
	PCB-1260	1	ug/L
	Toxaphene	2	ug/L
Surrogates	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

TABLE 8.2-10-5
GC Semivolatiles – Method 8151A
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water	2,4-D	4	ug/L
	Dalapon	2	ug/L
	2,4-DB	4	ug/L
	Dicamba	2	ug/L
	Dichlorprop	4	ug/L
	Dinoseb	0.6	ug/L
	MCPA	400	ug/L
	MCPP	400	ug/L
	2,4,5-TP (Silvex)	1	ug/L
	2,4,5-T	1	ug/L
Surrogate	2,4-Dichlorophenylacetic acid		
Solid	2,4-D	80	ug/kg
	Dalapon	40	ug/kg
	2,4-DB	80	ug/kg
	Dicamba	40	ug/kg
	Dichlorprop	80	ug/kg
	Dinoseb	12	ug/kg
	MCPA	8000	ug/kg
	MCPP	8000	ug/kg
	2,4,5-TP (Silvex)	20	ug/kg
	2,4,5-T	20	ug/kg
Surrogate	2,4-Dichlorophenylacetic acid		

TABLE 8.2-10-6
GC Volatiles - Method 8021B
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water 5 mL Purge	Benzene	1	ug/L
	Bromobenzene	1	ug/L
	Bromochloromethane	1	ug/L
	Bromodichloromethane	1	ug/L
	Bromoform	1	ug/L
	Bromomethane	1	ug/L
	n-Butylbenzene	1	ug/L
	sec-Butylbenzene	1	ug/L
	tert-Butylbenzene	1	ug/L
	Carbon tetrachloride	1	ug/L
	Chlorobenzene	1	ug/L
	Chlorodibromomethane	1	ug/L
	Chloroethane	1	ug/L
	Chloroform	1	ug/L
	Chloromethane	1	ug/L
	2-Chlorotoluene	1	ug/L
	4-Chlorotoluene	1	ug/L
	1,2-Dibromo-3-chloropropane (DBCP)	1	ug/L
	1,2-Dibromoethane (EDB)	1	ug/L
	Dibromomethane	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Dichlorodifluoromethane	1	ug/L
	1,1-Dichloroethane	1	ug/L
	1,2-Dichloroethane	1	ug/L
	cis-1,2-Dichloroethene	1	ug/L
	trans-1,2-Dichloroethene	1	ug/L
	1,1-Dichloroethene	1	ug/L
	1,2-Dichloropropane	1	ug/L
	1,3-Dichloropropane	1	ug/L
	2,2-Dichloropropane	1	ug/L
	cis-1,3-Dichloropropene	1	ug/L
	trans-1,3-Dichloropropene	1	ug/L
	1,1-Dichloropropene	1	ug/L
	Ethylbenzene	1	ug/
	Hexachlorobutadiene	1	ug/L
	Isopropylbenzene	1	ug/L
	p-Isopropyltoluene	1	ug/L
	Methylene chloride	5	ug/L
	Naphthalene	1	ug/L
	n-Propylbenzene	1	ug/L
	Styrene	1	ug/L
	1,1,1,2-Tetrachloroethane	1	ug/L
	1,1,2,2-Tetrachloroethane	1	ug/L
	Tetrachloroethene	1	ug/L
	Toluene	1	ug/L
	1,2,3-Trichlorobenzene	1	ug/L
	1,2,4-Trichlorobenzene	1	ug/L

TABLE 8.2-10-6
GC Volatiles - Method 8021B
Reporting Limits (RL), Method Reference [1,2]

Analyte	RL	Units
1,1,1-Trichloroethane	1	ug/L
1,1,2-Trichloroethane	1	ug/L
Trichloroethene	1	ug/L
Trichlorofluoromethane	1	ug/L
1,2,3-Trichloropropane	1	ug/L
1,2,4-Trimethylbenzene	1	ug/L
1,3,5-Trimethylbenzene	1	ug/L
Vinyl chloride	1	ug/L
m-Xylene & p-Xylene	2	ug/L
o-Xylene	1	ug/L
Xylenes (total)	3	ug/L
Surrogates		
1,4-Dichlorobutane		
Trifluorotoluene		

TABLE 8.2-10-6
GC Volatiles - Method 8021B
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Solid 5 g	Benzene	1	ug/kg
	Ethylbenzene	1	ug/kg
	Methyl tert-butyl ether (MTBE)	1	ug/kg
	Methyl tert-butyl ether	1	ug/kg
	Naphthalene	1	ug/kg
	Toluene	1	ug/kg
	1,2,4-Trimethylbenzene	1	ug/kg
	1,3,5-Trimethylbenzene	1	ug/kg
	m-Xylene & p-Xylene	2	ug/kg
	o-Xylene	1	ug/kg
	Xylenes (total)	3	ug/kg
Surrogates	Trifluorotoluene		

TABLE 8.2-10-6
GC Volatiles – Method 8021B
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Solid Encore, Methanol Prep	Benzene	50	ug/kg
	Ethylbenzene	50	ug/kg
	Methyl tert-butyl ether (MTBE)	50	ug/kg
	Naphthalene	250	ug/kg
	Toluene	50	ug/kg
	1,2,4-Trimethylbenzene	50	ug/kg
	1,3,5-Trimethylbenzene	50	ug/kg
	m-Xylene & p-Xylene	100	ug/kg
	o-Xylene	50	ug/kg
	Xylenes (total)	150	ug/kg
Surrogates	Trifluorotoluene		

TABLE 8.2-10-6
GC Volatiles – Method 8021B
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Frozen Encore	Benzene	1	ug/kg
	Ethylbenzene	1	ug/kg
	Methyl tert-butyl ether (MTBE)	1	ug/kg
	Methyl tert-butyl ether	1	ug/kg
	Naphthalene	1	ug/kg
	Toluene	1	ug/kg
	1,2,4-Trimethylbenzene	1	ug/kg
	1,3,5-Trimethylbenzene	1	ug/kg
	m-Xylene & p-Xylene	2	ug/kg
	o-Xylene	1	ug/kg
	Xylenes (total)	3	ug/kg
Surrogates	Trifluorotoluene		

TABLE 8.2-10-6
GC Volatiles – Method 601
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water	Bromodichloromethane	1	ug/L
	Bromoform	1	ug/L
	Bromomethane	1	ug/L
	Carbon tetrachloride	1	ug/L
	Chlorobenzene	1	ug/L
	Dibromochloromethane	1	ug/L
	Chloroethane	1	ug/L
	2-Chloroethyl vinyl ether	5	ug/L
	Chloroform	1	ug/L
	Chloromethane	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Dichlorodifluoromethane	1	ug/L
	1,1-Dichloroethane	1	ug/L
	1,2-Dichloroethane	1	ug/L
	trans-1,2-Dichloroethene	1	ug/L
	1,1-Dichloroethene	1	ug/L
	1,2-Dichloropropane	1	ug/L
	cis-1,3-Dichloropropene	1	ug/L
	trans-1,3-Dichloropropene	1	ug/L
	Methylene chloride	5	ug/L
	1,1,2,2-Tetrachloroethane	1	ug/L
	Tetrachloroethene	1	ug/L
	1,1,1-Trichloroethane	1	ug/L
	1,1,2-Trichloroethane	1	ug/L
	Trichloroethene	1	ug/L
	Trichlorofluoromethane	1	ug/L
	Vinyl chloride	1	ug/L
Surrogates	1,4-Dichlorobutane		

TABLE 8.2-10-6
GC Volatiles – Method 602
Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Water	Benzene	1	ug/L
	Chlorobenzene	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Ethylbenzene	1	ug/L
	Toluene	1	ug/L
Surrogates	Trifluorotoluene		

TABLE 8.2-10-7
Total Petroleum Hydrocarbons, Diesel & Gasoline Range Organics Reporting Limits (RL), Method Reference [1,2]

	Analyte	RL	Units
Diesel Range Organics - Water	TPH (as Diesel)	100	ug/L
Surrogate	C9 (nonane)		
Diesel Range Organics - Solid	TPH (as Diesel)	10	mg/kg
Surrogate	C9 (nonane)		
Gasoline Range Organics - Water	Gasoline Range Organics	100	ug/L
	TPH (as Gasoline)	100	ug/L
	TPH (Purgeables)	100	ug/L
Gasoline Range Organics - Solid	Gasoline Range Organics	100	ug/kg
	TPH (as Gasoline)	100	ug/kg
	TPH (Purgeables)	100	ug/kg
Surrogate	TFT		
Gasoline Range Organics – Medium Level	TPH (as Gasoline) Solid	1000	ug/kg
	TPH (as Gasoline)	1000	ug/kg
	TPH (Purgeables)	1000	ug/kg
Surrogate	TFT		

Footnote:

[1] *The latest MDLs, RLs, and Control Limits will be utilized at the time of sample analysis, and are available upon request.*

[2] *The compounds listed represent TAL/TCL compounds. Project specific or other regulatory lists may be available upon request.*

TABLE 8.4-1
FIELD QUALITY CONTROL SAMPLES

Type	Applicability		Accuracy and Precision Application	Introduced By
	Inorganic	Organic		
Trip Blank (volatiles)	No	Yes	Accuracy	Supplier of Containers
Field Blank	Yes	Yes	Accuracy	Field Sampler
Rinsate Blank	Yes	Yes	Accuracy	Field Sampler
Collocated Sample	Yes	Yes	Precision	Field Sampler
Split Sample	Yes	Yes	Precision	Field Sampler
Field Duplicate	Yes	Yes	Precision	Field Sampler
Field Matrix Spike	Yes	Yes	Accuracy	Field Sampler

TABLE 8.4-2
LABORATORY QUALITY CONTROL SAMPLES

Type	Frequency	Applicability		Accuracy and Precision Application	Introduced By
		Inorganic	Organic		
Analytical Spike	As specified in methods, or as needed	Yes	No	Accuracy	Analyst/ Prep
Duplicate	1 out of 20 or at least 1/month/run	Yes	Yes	Precision	Analyst/ Prep
Instrument Blank	As specified methods, or as needed	Yes	Yes	Accuracy	Analyst
Interference Check Sample	As specified in methods	Yes	No	Accuracy	Analyst
Internal Standard	Each sample and standard	Yes	Yes	Both	Analyst/ Prep
Laboratory Control Sample	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Matrix Spike	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Matrix Spike Duplicate	1 per each group of samples processed up to 20 samples.	Yes	Yes	Both	Analyst/ Prep
Method Blank	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Surrogate	All standards, method blanks, LCS, and samples.	No	Yes Method Dependent	Accuracy	Analyst/ Prep

TABLE 8.4-3
LABORATORY PERFORMANCE QUALITY CONTROL SAMPLES

Sample/Measurement	Purpose
Method Blanks	Demonstrates that the laboratory systems (*e.g.*, glassware cleaning procedures) and laboratory reagents used for the preparation and analysis of samples have not contributed to a false positive or negative measurement.
Instrument Blank	Demonstrates that the analytical system has not contributed to a false positive or negative measurement.
Laboratory Control Sample	Demonstrates the laboratory's ability to perform an analysis within the performance requirements of the method.

TABLE 8.4-4
MATRIX SPECIFIC QUALITY CONTROL SAMPLES

Quality Control Sample	Purpose
Duplicate Samples	Estimates the ability of the laboratory to obtain precise measurements on a sample. This measure is dependent on the homogeneity of the sample being duplicated. Solid samples often portray poor sample homogeneity and therefore often have poor duplication with regards to the sample result.
Matrix Spike Sample	Estimates the ability of the laboratory to obtain accurate measurements on a sample. The measure is dependent on the bias a sample matrix may cause regarding a given analyte.
Matrix Spike Duplicate Sample	In addition to verifying the accuracy of the matrix spike sample, the matrix spike duplicate can be used with the matrix spike sample as a measure of precision by calculating the relative percent difference (RPD).

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Alkalinity	Method Blank	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	310.1 2320B	Total alkalinity: 1 per batch of 20 samples	—	N/A
	Matrix Spike Duplicate	310.1 2320B	Total alkalinity: 1 per batch of 20 samples	—	N/A
	Duplicate	310.1 2320B	For carbonate, bicarbonate, hydroxide, alkalinity only. Frequency: 1 per batch of 10 samples Criteria 310.1: ≤ 20 % RPD(3) Criteria 2320B: ≤ 25 % RPD(3) Corrective Action: Flag data outside of limit.	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Ammonia	Method Blank	350.1 350.2 SM4500 NH-E,F	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	350.1 350.2 SM4500 NH-E,F	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	—	N/A
	Matrix Spike	350.1 350.2 SM4500 NH-E,F	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Ammonia (Cont'd)	Matrix Spike Duplicate	350.1 350.2 *SM4500* NH-E,F	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	350.1 350.2 SM4500 NH-E,F	N/A	—	N/A
Ammonia (TKN)	Method Blank	351.2 351.3 SM4500 NO_3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Ammonia (TKN) (Cont'd)	Laboratory Control Sample	351.2 351.3 SM4500 NO_3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits *Corrective Action: If not* within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	351.2 351.3 SM4500 NO_3	*Frequency: 1 per 20* samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	351.2 351.3 SM4500 NO_3	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	351.2 351.3 SM4500 NO_3	N/A	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
BOD	Method Blank	405.1 SM5210B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample and Laboratory Control Sample Duplicate	405.1 SM5210B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	405.1 SM5210B	N/A	—	N/A
	Matrix Spike Duplicate	405.1 SM5210B	N/A	—	N/A
	Duplicate	405.1 SM5210B	N/A	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Bromide	Method Blank	300.0A[(5)]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample and Laboratory Control Sample Duplicate	300.0A[(5)]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples
	Matrix Spike	300.0A[(5)]	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with MS outside of limit
	Matrix Spike Duplicate	300.0A[(5)]	N/A	9056A	N/A
	Duplicate	300.0A[(5)]	N/A	9056A	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chemical Oxygen Demand (COD)	Method Blank	410.4 SM5220D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample and Laboratory Control Sample Duplicate	410.4 SM5220D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	410.4 SM5220D	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	410.4 SM5220D	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	410.4 SM5220D	N/A	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chloride	Method Blank	300.0A [5] 325.2 325.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A [5] 325.2 325.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0A[(b)]	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Methods 9251Corrective Action: If not within laboratory control limits, rerun all associated samples Method 9056/9253 Corrective Action: Flag data associated with MS outside of limits

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chloride (cont'd)	Matrix Spike Duplicate	325.2 325.3	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits/< 20 % RPD(3) Corrective Action: Flag data outside of limit Method 9056: MSD is not applicable
	Duplicate	300.0A(5) 325.2 325.3	N/A	9056A	N/A
Chlorine, Residual	Method Blank	330.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	330.5	N/A	—	N/A
	Matrix Spike	330.5	N/A	—	N/A
	Matrix Spike Duplicate	330.5	N/A	—	N/A
	Duplicate	330.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD(3) Corrective Action: Flag data outside of limit.	—	Water

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chromium (Cr^{+6})	Method Blank	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples prepped Criteria: percent recovery for water must be within ± 15 % and for solids must be within ± 20% Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	3060A 7196A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chromium (Cr^{+6}) (Cont'd)	Matrix Spike Duplicate	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	3500 Cr-D	N/A	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD(3) limit Corrective Action: Flag data outside of limit.

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Conductivity, Specific	Method Blank	120.1 SM2510B	N/A	9050A	Not Applicable
	Laboratory Control Sample	120.1 SM2510B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9050A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	120.1 SM2510B	N/A	9050A	N/A
	Matrix Spike Duplicate	120.1 SM2510B	N/A	9050A	N/A
	Duplicate	120.1 SM2510B	Frequency: 1 with each batch of samples processed not to exceed 10 samples Criteria: ≤ 20 % RPD(3) Corrective Action: Flag data outside of limit.	9050A	Frequency: 1 with each batch of samples processed not to exceed 10 samples

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Cyanide (Amenable)	Method Blank	335.1 SM4500C N-G	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.1 SM4500C N-G	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.1 SM4500C N-G	N/A	9012A	N/A
	Matrix Spike Duplicate	335.1 SM4500C N-G	N/A	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within lab control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	335.1 SM4500C N-G	N/A	9012A	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Cyanide (Total)	Method Blank	335.2 335.3 335.4 4500-CN E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.2 335.3 335.4 4500-CN E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.2 335.3 335.4 4500-CN E	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Cyanide (Total) (cont'd)	Matrix Spike Duplicate	335.2 335.3 335.4 4500-CN E	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with *unacceptable Matrix Spike*
	Duplicate	335.2 335.3 335.4	N/A	9012A	N/A
Flashpoint	Method *Blank*	—	N/A	1010 ASTM D93-9	N/A
	Laboratory Control Sample	—	N/A	1010 ASTM D93-9	N/A
	Matrix Spike	—	N/A	1010 ASTM D93-9	N/A
	Matrix Spike *Duplicate*	—	N/A	1010 ASTM D93-9	N/A
	Duplicate	—	Frequency: 1 per batch of ≤20 samples per matrix Criteria: RPD[(3)] *must be* ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate	1010 ASTM D93-9	Frequency: 1 *per batch of* ≤20 samples per matrix Criteria: RPD[(3)] must be ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Fluoride	Method Blank	300.0A[(5)] 340.2 SM4500F-C,ISE	Frequency: 1 with each batch of samples processed not to exceed *20 samples* Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun *all samples associated* with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun *all samples associated* with unacceptable
	Laboratory Control Sample	300.0A[(5)] 340.2 SM4500F-C,ISE	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples
	Laboratory Control Sample Duplicate	300.0A[(5)] SM4500F-C,ISE	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Flouride (Cont'd)	Matrix Spike	300.0A[(5)] 340.2 SM4500F-C,ISE	Frequency: 1 per 10 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each *batch of samples* processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with *outside of limit*
	Matrix Spike Duplicate	340.2 SM4500F-C,ISE	Frequency: 1 per 20 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	340.2	N/A
	Duplicate	300.0A[(5)] 340.2 SM4500F-C,ISE	N/A	9056A	Frequency: 1 with each batch of samples processed Criteria: RPD[(3)] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[(3)] limits

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Hardness	Method Blank	130.2 2340B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	130.2 2340B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	130.2 2340B	Method 130.2: 1 per 20 samples Method 2340B: Frequency , Criteria, and Corrective Action: See ICP Metals Method 200.7 Requirements	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Hardness (cont'd)	Matrix Spike Duplicate	130.2 2340B	Method 130.2: 1 per 20 samples Method 2340B: Frequency, Criteria, and Corrective Action: See ICP Metals Method 200.7 Requirements	—	N/A
	Duplicate	130.2 2340B	N/A	—	N/A
Iron, Ferrous & Ferric	Method Blank	3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Iron, Ferrous & Ferric (Cont'd)	Matrix Spike	3500-Fe D	Frequency: 1 every 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	N/A
	Matrix Spike Duplicate	3500-Fe D	Frequency: 1 every 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	N/A
	Duplicate	3500-Fe D	N/A	—	N/A
Nitrate	Method Blank	300.0A(5) 353.2 SM4500-NO_3-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrate (Cont'd)	Laboratory Control Sample	300.0A[(5)] 353.2 SM4500-NO_3-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0A[(5)] 353.2 SM4500-NO_3-E	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within *laboratory control limits* Corrective Action: If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0A[(5)] 353.2 SM4500-NO_3-E	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrate (cont'd)	Duplicate	300.0A[(5)] 353.2 SM4500-NO_3-E	N/A	9056A	Frequency: 1 per 10 samples Criteria: RPD[(3)] must be *within laboratory control* limits Corrective Action: If not within laboratory control limits, flag all associated samples
Nitrite	Method Blank	300.0A[(5)] 353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A[(5)] 354.1 353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control *limits, rerun all* associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrite (Cont'd)	Matrix Spike	300.0A[(5)] 354.1 353.2	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0A[(5)] 354.1 353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	N/A
	Duplicate	300.0A[(5)] 354.1 353.2	N/A	9056A	Frequency: 1 per 10 samples Criteria: RPD[(3)] must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrate-Nitrite	Method Blank	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	353.2	N/A	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
pH	Method Blank	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Laboratory Control Sample	150.1 SM4500H-B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9040B 9045C	N/A
	Matrix Spike	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Matrix Spike Duplicate	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Duplicate	150.1 SM4500H-B	Frequency: 1 with each batch of samples processed not to exceed 10 samples per matrix Criteria: ≤ 20 % RPD[(3)] limit Corrective Action: Flag data outside of limit.	9040B 9045C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are ≤ 20% RPD[(3)] Corrective Action: Flag data associated with unacceptable Duplicate
Phenolics	Method Blank	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the *reporting limit* Corrective Action: Rerun all samples associated with unacceptable blank	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Phenolics (Cont'd)	Laboratory Control Sample	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable matrix spike	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Phosphorus (Total and Ortho-phosphate)	Method Blank	300.0 [4,5] 365.3 365.1 SM4500P-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0 [4,5] 365.3 365.1 SM4500P-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0 [4,5] 365.3 365.1 SM4500P-E	Frequency: 1 per 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data associated with MS outside of limits
	Matrix Spike Duplicate	365.3 365.1 SM4500P-E	Frequency: 1 per 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056A	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Phosphorus (Total and Ortho-phosphate) (Cont'd)	Duplicate	300.0[4,5] 365.3 365.1 SM4500P-E	N/A	9056A	Frequency: 1 with each batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[3] limits
Solids	Method Blank	160.1 160.2 160.3 160.4 160.5 SM2540E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: If analyte level in method blank is ≥ RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are reprepared and reanalyzed.	—	N/A
	Laboratory Control Sample	160.1 160.2 160.3 160.4 160.5 SM2540E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, reprepare and rerun all associated samples	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Solids (Cont'd)	Matrix Spike	160.1 160.2 160.3 160.4 160.5 SM4500P-E	N/A	—	N/A
	Matrix Spike Duplicate	160.1 160.2 160.3 160.4 160.5 SM4500P-E	N/A	—	N/A
	Duplicate	160.1 160.2 160.3 160.4 160.5 SM4500P-E	Frequency: 1 with each batch of samples processed not to exceed 10 samples Criteria: Sample results should agree within 20% if both the sample and sample duplicate results are > 5 X RL Corrective Action: Flag data outside of limit	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfate	Method Blank	300.0A[5] 375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9038 9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A[5] 375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9038 9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Method 9038 Criteria: Percent recovery must be within ± 15 % Method 9056 Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfate (cont'd)	Matrix Spike	300.0A[5] 375.4	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9038 9056A	Frequency: 1 with each batch of samples processed not to exceed 10 samples (9038) or 20 samples (9056) Method 9038 Criteria: Limits are 75% - 125% recovery Method 9056 Criteria: Percent recovery must be within laboratory 0control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	300.0A[5] 375.4	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9038 9056A	N/A
	Duplicate	300.0A[5] 375.4	N/A	9038 9056A	N/A
Sulfide	Method Blank	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfide (Cont'd)	Laboratory Control Sample	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data Method 9034: Not Applicable
	Duplicate	376.1	N/A	9030A	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Total Organic Carbon (TOC)	Method Blank	415.1 SM5310D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	415.1 SM5310D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	415.1 SM5310D	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Total Organic Carbon (TOC) (cont'd)	Matrix Spike Duplicate	415.1 SM5310D	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike Duplicate
	Duplicate	415.1 SM5310D	N/A	9060 Walkley-Black	Not Applicable
Total Organic Halides (TOX)	Method Blank	450.1 [5]	Frequency: 1 with each set of 8 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9020B	Frequency: Run in duplicate between each group of 8 analytical determinations Criteria: Concentration less than reporting limit or less than 2 X MDL or RL whichever is lower Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	450.1 [5]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery of analyte must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)	9020B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery of analyte must be within 90-110% Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Total Organic Halides (TOX) (cont'd)	Matrix Spike	450.1 [(5)]	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike	9020B	Frequency: 1 per batch of 10 samples Criteria: Must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-WC-0001
	Matrix Spike Duplicate	450.1[(5)]	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike	9020B	N/A
	Duplicate	450.1[(5)]	N/A	9020B	N/A
Turbidity	Method Blank	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Turbidity (cont'd)	Laboratory Control Sample	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	180.1	N/A	—	N/A
	Matrix Spike Duplicate	180.1	N/A	—	N/A
	Duplicate	180.1	Frequency: 1 per 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit Not Applicable.	—	N/A
Mercury by CVAA & CVAFS	Method Blank	200 series 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP CORP-MT-0003. Exception: If blank is above RL, and samples are ND.	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP CORP-MT-0003. Exception: If blank is above RL and samples are ND.

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Mercury by CVAA & CVAFS (Cont'd)	Laboratory Control Sample	200 series 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS SOP CORP-MT-0003. Exception: If samples are ND, results are reported.	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS SOP CORP-MT-0003. Exception: If samples are ND, results are reported.
	Matrix Spike	200 series 1631E	Frequency: with each batch of samples processed not to exceed 20 samples Criteria: Recovery should be within 75-125 % Corrective Action: Flag data associated with unacceptable MS. (See SOP CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery should be within 75-125 % Corrective Action: Flag data associated with unacceptable MS. (See SOP CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)
	Matrix Spike Duplicate	200 series 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery should be within 75-125 % , RPD[3] should be within 20% Corrective Action: Flag data associated with unacceptable MSD SOP CORP-MT-0003	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery should be within 75-125 % , RPD[3] should be within 20% Corrective Action: Flag data associated with unacceptable MSD SOP CORP-MT-0003

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Mercury by CVAA & CVAFS (Cont'd)	*Duplicate*	*200 series* 1631E	N/A	7000A series	N/A
ICP Metals	Method Blank	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid. Corrective Action: Rerun all samples associated with unacceptable blank SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit. Concentration less than *reporting with the* exception of lab common contaminants. Sample results <RL are also valid. Corrective Action: Rerun all samples associated with unacceptable blank SOP CORP-MT-0001
	Laboratory Control Sample	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 85-115%. If LCS is biased high and samples are <RL, the results are valid. Corrective Action: Rerun all samples associated with unacceptable LCS SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 20 %. If LCS is biased high and samples are <RL, the results are valid. Corrective Action: Rerun all samples associated with unacceptable LCS SOP CORP-MT-0001. If samples are ND, results are reported.

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
ICP Metals (Cont'd)	Matrix Spike	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125% Corrective Action: Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125% Corrective Action: Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001
	Matrix Spike Duplicate	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125%, RPD[3] must be within 20% Corrective Action: Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125%, RPD[3] must be within 20% Corrective Action: Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001
	Duplicate	200.7 200.8	Not Applicable	6010B 6020	Not Applicable
	Serial Dilution	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10% difference. 10% difference only applied if sample results are >50 times IDL. Corrective Action: Flag data associated with unacceptable Serial Dilution SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10% difference. 10% difference only applied if sample results are >50 times IDL. Corrective Action: Flag data associated with unacceptable Serial Dilution SOP CORP-MT-0001

TABLE 8.4-5
INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Footnotes

[1] National Pollutant Discharge Elimination System

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[3] RPD-Relative Percent Difference

[4] Orthophosphate only

[5] Method not listed in 40 CFR Part 136. Method 300.0 is a proposed 40CFR method. Specific state and/or region approval is required for NPDES.

[6] Current promulgated method is a Guidance Method Only, SW-846, Final Update III, Rev.3, 12/96.

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Aromatic Volatiles by GC	Method Blank	602	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	602	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	602	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Aromatic Volatiles by GC (Cont'd)	Matrix Spike Duplicate	602	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	602	N/A	8021B	N/A
	Surrogates	602	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Reprepare and reanlayze samples or flag sample data not meeting surrogate criteria
	Internal Standards	602	Optional: Internal standards are added to the method blank and all samples (QC included).	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Halogenated Volatiles by GC	Method Blank	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Halogenated Volatiles by GC (Cont'd)	Duplicate	--	N/A	8021B	N/A
	Surrogates	--	N/A	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Reprepare and reanalyze samples or flag sample data not meeting surrogate criteria.
	Internal Standards	--	N/A	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Herbicides	Laboratory Control Sample	615 [3]	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract all samples associated with unacceptable LCS	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS
	Matrix Spike	615 [3]	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	615 [3]	N/A	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable matrix spike sample

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Herbicides (cont'd)	Duplicate	615 [3]	N/A	8151A	N/A
	Surrogates	615 [3]	N/A	8151A	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must fall within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	615 [3]	N/A	8151A	Optional
Pesticides/ PCBs	Method Blank	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Reprepare and reanalyze all samples associated with unacceptable blank

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Pesticides/ PCBs (Cont'd)	Laboratory Control Sample	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	608	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Pesticides/ PCBs (cont'd)	Matrix Spike Duplicate	608	N/A	8081A 80882	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	608	N/A	8081A 8082	N/A
	Surrogates	608	Not specified in method	8081A 8082	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
Petroleum Hydro-carbons	Method Blank	1664A	Frequency: 1 with each preparation batch Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9071B	Frequency: 1 with each preparation batch Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Petroleum Hydro-carbons (Cont'd)	Laboratory Control Sample	1664A	Frequency: 1 with each analytical batch Criteria: Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS	9071B	Frequency: 1 with each analytical batch Criteria: Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	1664A	Frequency: 1 with every 10 samples per site Criteria: See percent recovery limits in SOP, NC-WC-0084 Corrective Action: See SOP, NC-WC-0084	9071B	Frequency: 1 with every 10 samples per site Criteria: See percent recovery limits in SOP, NC-WC-0084 Corrective Action: See SOP, NC-WC-0084
	Matrix Spike Duplicate	1664A	Frequency: 1 with every 20 samples per site Criteria: See percent recovery and RPD limits in SOP, NC-WC-0084 Corrective Action: See NC-WC-0084	9071B	Frequency: 1 with every 10 samples per site Criteria: See percent recovery and RPD limits in SOP, NC-WC-0084 Corrective Action: See NC-WC-0084
	Duplicate	1664A	N/A	9071B	N/A

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Purgeable Halocarbons by GC	Method Blank	601	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	601	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	601	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Purgeable Halocarbons by GC (Cont'd)	Matrix Spike Duplicate	601	N/A	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	601	N/A	8021B	N/A
	Surrogates	601	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	601	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Semivolatiles	Matrix Spike	625	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	625	N/A	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Semivolatiles (Cont'd)	Duplicate	625	N/A	8270C	N/A
	Surrogates	625	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8270C	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	625	Frequency: Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8270C	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS	Method Blank	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS (Cont'd)	Matrix Spike	624	Frequency: 1 per ≤ 20 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	624	N/A	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS (cont'd)	Duplicate	624	N/A	8260B	N/A
	Surrogates	624	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8260B	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	624	Frequency: Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8260B	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

Footnotes

[1] National Pollutant Discharge Elimination System

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996)

[3] Method not listed in 40 CFR Part 136

TABLE 8.5-1
INORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Acidity	Water	100 mL	305.1 SM2310B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
Alkalinity, Bicarbonate, Carbonate	Water	100 mL	310.1 2320B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Ammonia	Water	100 mL	350.1 350.2 SM4500NH$_3$-E SM4500NH$_3$-F	500 mL plastic or glass. Cool to 4°C H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Arsenic (ASV) Anodic Stripping Voltammetry	Water	100 mL	---	N/A	7063	250 mL plastic. Cool to 4°C. Hcl to pH <2, 28 days
Biochemical Oxygen Demand (BOD), Carbonaceous	Water	1000 mL	405.1 5210B	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Bromide	Water	50 mL	300.0A [7]	250 mL plastic or glass. No preservative required, 28 days	9056A	Cool to 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Cation Exchange	Solid	8 oz	---	N/A	9081	8 or 16 oz glass. Cool to 4°C, 6 months
Chemical Oxygen Demand (COD)	Water	100 mL	410.4 5220D	250 mL glass or plastic. Cool to 4°C, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

TABLE 8.5-1
INORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Chloride	Water	50 mL	300.0A [7] 325.2	250 mL plastic or glass. No preservative required, 28 days	9056A 9251	Method 9056: Cool to 4°C. Analyze ASAP after collection. Method 9251: 250ml plastic or glass, no preservative required, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chlorine, Residual	Water	100 mL	330.5	250 mL glass or plastic. Cool to 4°C, analyze immediately	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chromium (Cr^{+6})	Water	100 mL	3500 Cr-D	Method 218.4: 200 mL plastic or glass. Cool to 4°C, 24 hours. Method 3500 Cr-D: 200 mL quartz, TFE, or polypropylene HNO_3 to pH <2. Cool to 4°C. Analyze ASAP after collection	7196A	200 mL plastic or glass. Cool to 4°C, 24 hours
	Solid	20 g	---	N/A	7196A 3060A	250 mL plastic or glass, 30 days to digestion, 96 hours after digestion
	Waste	N/A	---	N/A	---	N/A
Conductivity	Water	100 mL	120.1 2510B	200 mL glass or plastic. Cool to 4°C, 28 days	9050A	200 mL glass or plastic. Cool to 4°C, 24 hours
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

TABLE 8.5-1
INORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Cyanide (Amenable)	Water	250 mL	335.1 SM4500CN-G	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[5]. Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6]. Cool to 4°C, 14 days
	Solid	50g	---	N/A	9012A	Not Specified
	Waste	50g	---	N/A	9012A	Not Specified
Cyanide (Total)	Water	1L	335.2 335.3 335.4 [(7)] SM4500CN-E	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6]. Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6]. Cool to 4°C, 14 days.
	Solid	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C, 14 days
	Waste	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C
Flashpoint (Ignitability)	Liquid	100 mL	---	N/A	1010 ASTM D93-9	No requirements, 250 mL amber glass. Cool to 4°C recommended
	Solid	100 g	--	N/A	---	N/A
	Waste	100 mL	--	N/A	---	N/A
Fluoride	Water	300 mL	300.0 / 340.2	500 mL plastic. No preservation required, 28 days.	9056A	Cool to 4°C. Analyze ASAP after collection.
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

TABLE 8.5-1
INORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Hardness (Total)	Water	50 mL	130.2 2340B	250 mL glass or plastic, HNO_3 to pH < 2, 6 months	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Iron (Ferrous)	Water	100 mL	3500-Fe D	1 liter glass or polyethylene container, 6 months. **This test should be performed in the field.**	-	N/A
	Solid	N/A	-	N/A	-	N/A
	Waste	N/A	-	N/A	-	N/A
Nitrate	Water	50 mL	300.0A [7] SM4500NO_3-E	Method 300.0: 250 mL plastic or glass. Cool to 4°C, 48 hours.	9056A	Method 9056: Cool to 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	Not Specified
Nitrite	Water	50 mL	300.0A [7] 354.1	250 mL plastic or glass. Cool, 4°C, 48 hours	9056A	Cool, 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Nitrate-Nitrite	Water	50 mL	353.2	250 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Ortho-phosphate	Water	50 mL	300.0A [7] 365.1 SM4500P-E	100 mL plastic or glass. Filter on site. Cool to 4°C, 48 hours	9056A	Cool to 4°C. Analyze ASAP collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

TABLE 8.5-1
INORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
pH	Water	50 mL	150.1 SM4500H-B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.**	9040B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.** [8]
	Solid	N/A	---	N/A	9045C	4 oz glass or plastic. Cool to 4°C. Analyze as soon as possible. [8]
	Waste	N/A	---	N/A	9045C	4 oz glass or plastic. Cool to 4°C. Analyze as soon as possible. [8]
Phenolics	Water	100 mL	420.1	500 mL glass, Cool to 4°C, H_2SO_4 to pH < 2, 28 days	9065	1 liter glass recommended, Cool to 4°C, H_2SO_4 to pH < 4, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	9065	Not Specified
Phosphate	Water	50 mL	---	N/A	9056A	Cool to 4°C, analyze ASAP collection
	Solid	N/A	---	N/A	9056A	N/A
	Waste	N/A	---	N/A	9056A	N/A

TABLE 8.5-1
INORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Phosphorus (Total)	Water	100 mL	365.2 365.1 SM4500P-E	100 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Settleable Solids	Water	1000 mL	160.5	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Specific Conductance	Water	50 mL	120.1 2510B	250 mL plastic or glass. Cool to 4°C, 24 hours	9050A	250 mL plastic or glass. Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Sulfate (SO_4)	Water	50 mL	300.0A [7] 375.4	100 mL plastic or glass. Cool to 4°C, 28 days	9056A 9038	Method 9056: Cool to 4°C, Analyze ASAP collection. Method 9038: 200 mL plastic or glass, Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	100 mL	---	N/A	9038	200 mL plastic or glass. Cool to 4°C, 28 days

TABLE 8.5-1
INORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Sulfide	Water	250 mL	376.1	500 mL plastic or glass. Cool to 4°C, Add 2 mL zinc acetate plus NaOH to pH > 9, 7 days	9030A 9030B/ 9034	500 mL plastic, no headspace. Cool to 4°C. Add 4 drops of 2N zinc acetate per 100 mL of sample, adjust the pH to > 9 with 6 N NaOH solution, 7 days
	Solid	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
	Waste	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
Total Dissolved Solids (Filterable)	Water	100 mL	160.1 2540C	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

TABLE 8.5-1
INORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Total Kjeldahl Nitrogen (TKN)	Water	100 mL	351.3	500 mL plastic or glass. Cool to 4°C, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Organic Carbon (TOC)	Water	100 mL	415.1 SM5310D	100 mL plastic or glass. Cool to 4°C, H_2SO_4 to pH < 2, 28 days	9060 Walkley-Black	100 mL glass or 40 mL VOA vials, Cool to 4°C, H_2SO_4 or HCl to pH < 2, 28 days
	Solid	N/A	---	N/A	9060 Walkley-Black	Not Specified
	Waste	N/A	---	N/A	9060 Walkley-Black	Not Specified
Total Organic Halides (TOX) (EOX)	Water	100 mL	450.1 [7]	500 mL amber glass, Teflon®-lined lid. Cool to 4°C, HNO_3 to pH <2, no headspace, 28 days	9020B 9023 (EOX)	500 mL amber glass, Teflon®-lined lid. Cool to 4°C, H_2SO_4 to pH < 2, no headspace, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Solids	Water	100 mL	160.3	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Suspended Solids (Nonfilterable)	Water	100 mL	160.2	250 mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

TABLE 8.5-1
INORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Turbidity	Water	50 mL	180.1	250 mL plastic or glass. Cool, 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Volatile Solids	Water	100 mL	160.4	250 mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Metals (excludes Hg)	Water	100 mL	200 series	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 6 months	6010B 6020	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 6 months
	Solid	200 g	200 series	2, 8, or 16 oz glass or polyethylene container storage at 4 °C	6010B 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
	Waste	200 g	200 series	N/A	6010B 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
Mercury (CVAA) (CVAFS)	Water	100 mL	245.1 245.7 1631E	250 mL glass or polyethylene container, HNO_3 to pH ≤ 2, 28 days	7470A	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 28 days
	Solid	200 g	--	2, 8, or 16 oz glass or polyethylene container. Cool to 4°C, 28 days	7471A	8 or 16 oz glass or polyethylene container. Cool to 4°C, 28 days (CORP-MT-0007)
	Waste	200 g	--	N/A	7471A	8 or 16 oz glass or polyethylene container. Cool, 4°C, 28 days (CORP-MT-0007)

TABLE 8.5-1
INORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES (Cont'd)

Footnotes

1 Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.

2 National Pollutant Discharge Elimination System - MCAWW, March 1983.

3 Holding times are calculated from date of collection.

4 Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA, (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

5 Solid matrix type includes soil, sediment, sludge and other solid materials not classified as waste.

6 Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.

7 Method not listed in 40 CFR Part 136.

8 If not done in the field (ASAP) per the method and requested by client, analyze in lab within 48 hours.

9 EPA issued memo recommending not to use reactive cyanide and sulfide methods.

TABLE 8.5-2
ORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2,3]		RCRA (SW846) [3,4]	
			Method	Requirements	Method [6]	Requirements
Aromatic Volatiles	Water	40 mL	602	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 7 days with pH > 2,14 days with pH ≤ 2	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 1:1 HCl to pH <2, 14 days with pH ≤ 2
	Solid [5]	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool, 4°C [12]
	Waste	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Max holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol). Cool to 4°C [12]

TABLE 8.5-2
ORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES (cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Halogenated Volatiles by GC	Water	40 mL	--	N/A	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 1:1 HCl to pH < 2, 14 days
	Solid [5]	5 g or 25 g	--		8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool to 4°C [12]
	Waste	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool, 4°C [12]

TABLE 8.5-2
ORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES (Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Herbicides	Water	1L	615 [10]	1 liter amber glass with Teflon®-lined lid, Sodium thiosulfate or ascorbic acid if residual chlorine present. Cool to 4°C, Extraction, 7 days. Analysis, 40 days after extraction	8151A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool to 4°C. Extraction, 7 days. Analysis, 40 days of the start of extraction.
	Solid	50 g	--	N/A	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.
	Waste	50 g	--	N/A	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.

TABLE 8.5-2
ORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Pesticides/ PCBs	Water	1L	608	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C. Extraction, 7 days. Analysis, 40 days after extraction.	8081A 8082	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, 40 days of the start of the extraction.
	Solid	50 g	---	N/A	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.
	Waste	50 g	---	N/A	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.

TABLE 8.5-2
ORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2,3]		RCRA (SW846) [3,4]	
			Method	Requirements	Method [6]	Requirements
Petroleum Hydrocarbons/ Oil and Grease	Water	1 L	1664A[(7)]	1 liter glass, Cool, 4°C HCl or H_2SO_4 to pH <2 28 days	9071B	1 liter glass, Cool, 0-4°C HCl or H_2SO_4 to pH <2 28 days
	Solid	30 g	1664A[(7)]	8 or 16 oz. Wide mouth glass jar, Cool, 4°C, 28 days	9071B	8 or 16 oz. wide mouth glass jar, Cool, 0-4°C, 28 days
	Waste	---	---	N/A	9071B	N/A
Purgeable Halocarbons by GC	Water	40 mL	601	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 14 days.	8021B	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 1:1 HCl to pH ≤ 2, 14 days.
	Solid	5 g or 25 g	---	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol). Cool, 4°C[12].

TABLE 8.5-2
ORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Purgeable Halocarbons by GC (cont'd)	Waste	5 g or 25 g	---	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore™ sampler is 48 hours (before the sample is added to methanol). Cool, 4°C [12]
Semivolatiles	Water	1L	625	1 liter amber glass with Teflon®-lined lid. Cool, 4°C. Extraction, 7 days. Analysis, 40 days.	8270C	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, within 40 days of extraction.
	Solid	50 g	---	N/A	8270C	8 or 16 oz glass wide mouth with Teflon-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.
	Waste	50 g	---	N/A	8270C	8 or 16 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.

TABLE 8.5-2
ORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Volatile Organics	Water	40 mL	624	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2[8].	8260B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2[9].
	Solid[5]	5 g or 25 g	--	N/A	8260B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol or sodium bisulfate). Cool to 4°C[(12)]
	Waste	5 g or 25 g	--	N/A	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for Encore™ sampler is 48 hrs (before sample is added to methanol or sodium bisulfate). Cool to 4°C[12]

TABLE 8.5-2
ORGANIC SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(Cont'd)

Footnotes

[1] Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.

[2] National Pollutant Discharge Elimination System - 40 CFR Part 136, Appendix A.

[3] Holding times are calculated from the date of collection.

[4] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[5] Solid matrix type includes soil, sediment, sludge or other solids not classified as waste.

[6] Only one determination method is listed when separate methods are required for preparation and analysis.

[7] Method 1664 was promulgated by the EPA with an effective date of June 14, 1999.

[8] For acrolein and acrylonitrile the pH should be adjusted to 4-5. This pH adjustment is not required if acrolein is not measured. Samples requiring analysis of acrolein that received no pH adjustment must be analyzed within three days of sampling.

[9] For acrolein and acrylonitrile the pH should be adjusted to 4-5.

[10] Method not listed in 40 CFR Part 136.

[11] Should only be used in the presence of residual chlorine.

[12] Depending on regulatory programs, EnCore™ samplers may be preserved for up to 14 days from sampling by freezing at -5 to-12°C until analysis. Alternatively the EnCore™ sample may be transferred to a 40-ml VOA vial and preserved by freezing at -5 to -12°C until analysis. Some regulatory agencies may require 4 or 8 oz glass with Teflon®-lined lid, Cool 4°C, 14 days. This technique is not recommended, but will be supported where required. (Preservation and holding times are subject to client specifications.)

TABLE 8.5-3
SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES FOR TCLP[1] AND SPLP[2]

Analytical Parameters	Matrix	Minimum Sample Size	TCLP Method 1311 and SPLP Method 1312 Requirements	
			From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Analysis
Mercury	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 28 days	Glass or polyethylene 28 days
Metals (except mercury)	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 180 days	Glass or polyethylene 180 days
Semivolatiles	Liquid Solid Waste	1L	1L glass, Cool 4°C, 14 days	1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days
Volatiles	Liquid Solid Waste	6 oz	4 oz glass, Cool 4°C, 14 days	40 mL glass, 14 days

Footnotes

1 TCLP = Toxicity Characteristic Leaching Procedure
2 SPLP = Synthetic Precipitation Leaching Procedure

TABLE 8.5-4
PERIODIC EQUIPMENT CALIBRATIONS

Type of Equipment	Calibration Requirements
Balances	• Must be serviced and calibrated annually by an approved vendor. • Calibration must be checked daily or before use by analyst with weight(s) classified as Class 1 (formerly termed Class S) by NIST or Class 1 traceable. Acceptance criteria vary according to weight used and accuracy of balance. Acceptance criteria must be documented in the log. • All Class 1 weights must be certified by an outside vendor every three years. • All non-Class 1 weights must be checked annually against NIST Class 1 weights annually.
Thermometers	• Working glass thermometers must be calibrated against a certified. • NIST thermometer at least annually as described in operation-specific SOPs. • Working non-glass thermometers must be calibrated against a certified NIST thermometer annually as described in operation-specific SOPs. • The NIST thermometer must be recertified every three years.
Refrigerators/ Freezers	• Thermometers must be immersed in a liquid such as mineral oil or glycol. • Temperature of units used for sample or standard storage must be checked daily as described in operation-specific SOPs. Refrigerator acceptance limits: 4°C ± 2°C. Freezer acceptance limits: < - 10°C
Ovens	• Temperature of units must be checked daily or before use. • Acceptance limits vary according to use as described in operation-specific SOPs, andmust be documented in the temperature log.
Micropipettes	• Calibrations are checked gravimetrically as required by the operation-specific SOP. • Must be calibrated at the frequency (normally quarterly) required by the manufacturer at a minimum.
Syringes, Volumetric Glassware and Graduated Glassware	• All syringes and volumetric glassware are purchased as Class A items. • Class A items are certified by the manufacturer to be within ± 1% of the measured volume; therefore, calibration of these items by TestAmerica laboratories is not required. • All analysts are trained in the proper use and maintenance of measuring devices to ensure the measurement of standards, reagents, and sample volumes are within method tolerances.

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Acidity	Initial	350.1	Two-level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value	9040B 9045C	2 point calibration ± 0.05 pH units of true value
	Continuing	350.1	One buffer check every 10 samples ± 5% of true value	9040B 9045C	N/A
	Other	350.1	Third point check	9040B 9045C	Third point check
	Ending	350.1	One buffer check ± 5% of true value	9040B 9045C	N/A
Alkalinity, Bicarbonate, Carbonate	Initial	310.1 2320B	2 point calibration of pH meter ± 0.05 pH units of true value	--	N/A
	Continuing	310.1 2320B	N/A	--	N/A
	Ending	310.1 2320B	N/A	--	N/A
Ammonia	Initial	350.1	6 levels including blank, "r" [3] ≥ 0.995	--	N/A
	Continuing	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
	Ending	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
Arsenic Speciation		N/A	N/A	7063	* Refer to Section 10 of SOP NC-WC-0090

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Biochemical Oxygen Demand (BOD)	Initial	405.1 SM5210B	a. Winkler titration: Iodometric with standard thiosulfate b. Membrane electrode: Read in air and in water with zero dissolved oxygen	--	N/A
	Continuing	405.1 SM5210B	N/A	--	N/A
	Ending	405.1 SM5210B	N/A	--	N/A
Bromide	Initial	300.0A	5 levels plus a blank, "r"$^3 \geq 0.995$	9056A	5 levels plus a blank, "r" $^3 \geq 0.995$
	Continuing	300.0A	Level every 10 samples ± 10% of true value	9056A	N/A
	Ending	300.0A	N/A	9056A	N/A
Cation Exchange	Initial	N/A	N/A	9081	N/A
	Continuing	N/A	N/A	9081	N/A
	Ending	N/A	N/A	9081	N/A
Chemical Oxygen Demand (COD)	Initial	410.4 SM5220D	5 levels plus a blank"r" $^3 \geq 0.995$	--	N/A
	Continuing	410.4 SM5220D	One level every 10 samples ± 10% of true value	--	N/A
	Ending	410.4 SM5220D	One level ± 10% of true value	--	N/A

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Chloride	Initial	300.0A 325.2	5 levels plus blank "r" [3] ≥ 0.995	9056A 9251	Method 9056: 3 levels plus a blank Method 9252: 5 levels plus blank "r" [3] ≥ 0.995
	Continuing	300.0A 325.2	One level every 10 samples ± 10% of true value	9056A 9251	Method 9056: One per batch of 20 samples, ± 10% of true value Method 9252: One level every 10 samples, ± 10% of true value
	Ending	300.0A 325.2	One level every 10 samples ± 10% of true value	9056A 9251	Method 9056 : N/A Method 9252: One level ± 10% of true value
Chromium Cr^{+6}	Initial	3500 Cr-D	3 levels plus blank	7196A	5 levels plus blank "r" [3] ≥ 0.995
	Continuing	3500 Cr-D	One level every 10 samples ± 10% of true value	7196A	One level every 10 samples ± 15%
	Ending	3500 Cr-D	One level ± 10% of true value	7196A	One level ± 15%
Chlorine, Residual	Initial	330.5 SM3500CL-G	N/A	--	N/A
	Continuing	330.5 SM3500CL-G	N/A	--	N/A
	Ending	330.5 SM3500CL-G	N/A	--	N/A

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Conductivity	Initial	120.1 SM2510B	Standard KCl solution	9050A	One level to determine cell constant
	Continuing	120.1 SM2510B	N/A	9050A	N/A
	Ending	120.1 SM2510B	N/A	9050A	N/A
Cyanide (Amenable)	Initial	335.1 SM4500CN-G	6 levels plus blank "r" [3] ≥ 0.995	9012A	6 levels plus blank "r" [3] ≥ 0.995
	Continuing	335.1 SM4500CN-G	One level every 10 samples ± 10% of true	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.1 SM4500CN-G	One level ± 10 % of true value	9012A	± 15% of true value
Cyanide (Total)	Initial	335.1/335.2 335.3/335.4 SM4500CU-E	6 levels plus blank "r" [3] ≥ 0.995	9012A	6 levels plus blank "r" [3] ≥ 0.995
	Continuing	335.1/335.2 335.3/335.4 SM4500CU-E	One mid-level every 10 samples ± 10 % of true value	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.1/335.2 335.3/335.4 SM4500CU-E	One mid-level ± 10 % of true value	9012A	± 15% of true value
Flashpoint	Initial	--	N/A	1010 ASTM D93-9	p-Xylene reference standard must have flashpoint of 81°F ±2°F
	Continuing	--	N/A	1010 ASTM D93-9	N/A
	Ending	--	N/A	1010 ASTM D93-9	N/A

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Fluoride	Initial	300.0A 340.2	Method 300.0A: 5 levels plus a blank, "r" [3] ≥ 0.995 Method 340.2: 5 levels "r" [3] ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 340.2	One mid-level every 10 samples ± 10% of true value	9056A	*One per batch of 20* samples ± 10% of true value
	Ending	300.0A 340.2	One mid-level ± 10% of true value	9056A	N/A
Hardness	Initial	130.2 2340B	Method 130.2: Standardize titrant Method 2340B: See ICP Metals 200.7	--	N/A
	Continuing	130.2 2340B	Method 130.2: N/A Method 2340B: See ICP Metals 200.7	--	N/A
	Ending	130.2 2340B	Method 130.2: N/A Method 2340B: See ICP Metals 200.7	--	N/A
Iron (Ferrous)	Initial	3500-Fe D	3 levels plus a blank, "r" [3] ≥ 0.995	-	N/A
	Continuing	3500-Fe D	*One mid-level every* 10 samples ± 10% of true value	-	N/A
	Ending	3500-Fe D	One mid-level ± 10% of true value	-	N/A

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Nitrate	Initial	300.0A 353.2 SM4500NO_3-E	5 levels plus a blank "r" [3] ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 353.2 SM4500NO_3-E	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 353.2 SM4500NO_3-E	One mid-level ± 10% of true value	9056A	N/A
Nitrite	Initial	300.0A 354.1	5 levels plus a blank "r" [3] ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 354.1	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 354.1	One mid-level ± 10% of true value	9056A	N/A
Nitrate-Nitrite	Initial	300.0A 353.2	5 levels plus blank "r" [3] ≥ 0.995	--	N/A
	Continuing	300.0A 353.2	One level every 10 samples ± 10% of true value	--	N/A
	Ending	300.0A 353.2	One mid-level ± 10% of true value	--	N/A

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Phosphorus (Total and Ortho-phosphate)	Initial	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: 3 levels plus a blank Method 365.2: 5 levels plus a blank	--	N/A
	Continuing	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: *One level every 10* samples ± 10% of true value Method 365.2: Blank *and 2 standards with* each series of samples, ± 2% of true value or recalibrate	--	N/A
	Ending	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: ± 10% of true value Method 365.2: N/A	--	N/A
pH	Initial	150.1 SM4500H-B	2 level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value	9040B 9045C	2 point calibration ± 0.05 pH units of true value
	Continuing	150.1 SM4500H-B	One buffer check every 10 samples ± 5% of true value	9040B 9045C	N/A
	Other	150.1 SM4500H-B	Third point check	9040B 9045C	Third point check
	Ending	150.1 SM4500H-B	One buffer check ± 5% of true value	9040B 9045C	N/A

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Phenolics	Initial	420.1	5 levels plus a blank "r" [3] ≥ 0.995	9065 9066	5 levels plus a blank "r" [3] 0.995
	Continuing	420.1	One mid-level every 10 samples ± 10% true value	9065 9066	One mid-level ± 15% true value
	Ending	420.1	One mid-level ± 10% true value	9065 9066	One mid-level ± 15% true value
Phosphate	Initial	SM4500P-E	N/A	9056	3 levels plus a blank
	Continuing	SM4500P-E	N/A	9056	One per batch of 20 samples ± 15% of true value
	Ending	SM4500P-E	N/A	9056	N/A
Settleable Solids	Initial	160.5	N/A	--	N/A
	Continuing	160.5	N/A	--	N/A
	Ending	160.5	N/A	--	N/A
Specific Conductance	Initial	120.1 SM2510B	Standardize meter with 0.01 M KCl	9050A	N/A
	Continuing	120.1 SM2510B	One level every 10 samples ± 10% of true value	9050A	N/A
	Ending	120.1 SM2510B	One level ± 10% of true value	9050A	N/A
Sulfate	Initial	300.0A 375.4	Method 300.0A: 5 levels plus blank "r" [3] ≥ 0.995 Method 375.4: 3 levels plus blank "r" [3] ≥ 0.995	9038 9056A	Method 9038: 3 levels plus a blank for every hour of continuous sample analysis. Method 9056: 3 levels plus a blank

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Sulfate (Con'd)	Continuing	300.0A 375.4	Method 300.0A: One mid-level after every 10 samples ± 10% of true value Method 375.4: One level every 3 or 4 samples ± 10% of true value	9038 9056A	Method 9038: Independent-prepared check standard every 15 samples Method 9056: 1 per batch of 20 samples ± 10% of true value
	Ending	300.0A 375.4	± 10% of true value	9038 9056A	N/A
Sulfide	Initial	376.1	Method 376.1: This is a titration method. Therefore, calibrations are not applicable.	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
	Continuing	376.1	Method 376.1: N/A	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
	Ending	376.1	Method 376.1: N/A	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
Total Dissolved Solids	Initial	160.1 SM2540E	This is a gravimetric determination. Calibrate balance prior to analysis	--	N/A
	Continuing	160.1 SM2540E		--	N/A
	Ending	160.1 SM2540E		--	N/A

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Total Kjeldahl Nitrogen (TKN)	Initial	351.3 SM4500NO_3	Method 351.3: Titrimetric: Standardize titrant Colorimetric: 7 levels plus blank	--	N/A
	Continuing	351.3 SM4500NO_3	Method 351.3: N/A	--	N/A
	Ending	351.3 SM4500NO_3	Method 351.3: N/A	--	N/A
Total Organic Carbon (TOC)	Initial	415.1 SM5310D	3 levels plus blank	9060 Walkley Black	3 levels plus blank "r" [3] ≥ 0.995
	Continuing	415.1 SM5310D	1 mid-level every 10 samples ± 15% of true value	9060 Walkley Black	1 mid-level every 10 samples ± 15% of true value
	Ending	415.1 SM5310D	± 15% of true value	9060 Walkley Black	± 15% of true value
Total Organic Halides (TOX)	Initial	450.1	Method 450.1: Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration standard) Verify with independently-prepared check standard	9020B 9023 (EOX)	Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration standard) Verify with independently-prepared check standard –ICV ± 10% SOP CORP-WC-0001

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Total Organic Halides (TOX) (cont'd)	Continuing	450.1	± 10% of true value	9020B 9023 (EOX)	CCV ± 10% of true value SOP CORP-WC-0001
	Ending	450.1	± 10% of true value	9020B 9023 (EOX)	CCV ± 10% of true value SOP CORP-WC-0001
Total Solids	Initial	160.3	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.3		--	N/A
	Ending	160.3		--	N/A
Total Suspended Solids (Nonfilterable)	Initial	160.2 SM2540E	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.2 SM2540E		--	N/A
	Ending	160.2 SM2540E		--	N/A
Turbidity	Initial	180.1	Minimum of 1 level in each instrument range. Follow manufacturer's instructions	--	N/A
	Continuing	180.1	N/A	--	N/A
	Ending	180.1	N/A	--	N/A
Volatile Solids	Initial	160.4	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.4		--	N/A
	Ending	160.4		--	N/A

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
ICP & ICP/MS Metals (excludes Hg)	Initial	200.7	One level and blank. ICV RSD <3% from replicate	6010B	One level and blank. ICV RSD <5% from replicate
	Initial	200.8	One level and blank	6020	One level and blank
	Continuing	200.7	Every 10 samples ±10% of true value CCV RSD < 5% from replicate	6010B	Mid-level calibration standard Every 10 samples ± 10% of true value CCV RSD < 5% from replicate
	Continuing	200.8	N/A	6020	N/A
	Ending	200.7	±10% of true value CCV RSD < 5% from replicate	6010B	Mid-level calibration standard ± 10% of true value CCV RSD < 5% from replicate
	Ending	200.8	N/A	6020	N/A
	Other	200.7	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits	6010B	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits
	Other	200.8	N/A	6020	N/A

TABLE 8.5-5
SUMMARY OF INORGANIC METHOD CALIBRATIONS
(Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Mercury by CVAA & CVAFS	Initial	245.1 1631E	5 levels plus blank ICV ±10% of true value "r" [3] ≥ 0.995	7470A 7471A	5 levels plus blank ICV ± 10% of true value "r" [3] ≥ 0.995
	Continuing	245.1* 1631E	Daily or every 10 samples, whichever is more frequent ±20% of true value	7470A 7471A	Every 10 samples ±20% of true value
	Ending	245.1 1631E	±20% of true value	7470A 7471A	±20% of original prepared standard
	Other	245.1 1631E	Annually: MDL	7470A 7471A	Annually: MDL

* 245.1 continuing – Initial CCV ±5% of true value

Footnotes

1 National Pollutant Discharge Elimination System.

2 Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December, 1996).

3 "r" = correlation coefficient.

TABLE 8.5-6
SUMMARY OF ORGANIC METHOD CALIBRATIONS

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Aromatic Volatiles by GC	Initial	602	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.
	Continuing	602	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 injections or 12 hrs whichever is greater, % D ≤ 15%, gases 20% D. Evaluate per SOP
	Ending	602	Run closer per NELAC requirement, but no criteria	8021B	Mid-level calibration standard, % D ≤ 15%. Evaluate per SOP, except as noted above.
	Other	602	N/A	8021B	N/A
Herbicides by GC	Initial	615 [9]	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8151A	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	615 [9]	One or more calibration standards analyzed daily % D ± 15% of predicted response	8151A	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	615 [9]	N/A	8151A	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	615 [9]	N/A	8151A	N/A

TABLE 8.5-6
SUMMARY OF ORGANIC METHOD CALIBRATIONS (Cont'd)

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Pesticides/ PCBs by GC	Initial	608	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8081A 8082	Minimum of 5 levels. If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. (See SOP CORP-GC-0001)
	Continuing	608	One or more calibration standards analyzed daily. % D ± 15% of predicted response	8081A 8082	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	608	N/A	8081A 8082	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	608	N/A	8081A 8082	N/A

TABLE 8.5-6
SUMMARY OF ORGANIC METHOD CALIBRATIONS (Cont'd)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Petroleum Hydrocarbons /Oil and Grease	Initial	1664A	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be ± 10% at 2 mg and ± 0.5% at 1000 mg or recalibrate balance	9071B	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be ± 10% at 2 mg and ± 0.5% at 1000 mg or recalibrate balance
	Continuing	1664A	N/A	9071B	N/A
	Ending	1664A	N/A	9071B	N/A
Purgeable Halocarbons by GC	Initial	601	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.
	Continuing	601	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 injections, or 12 hrs, whichever is greater. % D < 15%, gases 20% D Evaluate per SOP
	Ending	601	Run per NELAC, but no requirement to meet	8021B	Mid-level calibration standard, % D <15%. Evaluate per SOP
	Other	601	N/A	8021B	N/A

TABLE 8.5-6
SUMMARY OF ORGANIC METHOD CALIBRATIONS (Cont'd)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Halogenated Volatiles by GC	Initial	--	N/A	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	--	N/A	8021B	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	--	N/A	8021B	Mid-level calibration standard % D < 15% of predicted response for any analyte quantitated and reported.
	Other	--	N/A	8021B	N/A
Semivolatiles	Initial	625	Minimum of 3 levels, lowest near but above MDL. If % RSD ≤ 35%, use avg RF. Otherwise calibration curve employed.	8270C	Minimum of 5 levels, % RSD for RF for CCCs[4] < 30% SPCCs[5]; RF > 0.050
	Continuing	625	One level every 24 hours. Acceptance criteria are found in the method and SOP.	8270C	Mid-level standard every 12 hours (after tuning) %D for CCCs[4] < 20 % between RF from standard and avg RF from initial SPCCs[5]; RF > 0.050.
	Ending	625	N/A	8270C	N/A
	Other	625	DFTPP[7] tuning every 24 hours before standard or sample runs.	8270C	DFTPP[7] tuning at the beginning of every 12 hour shift.

TABLE 8.5-6
SUMMARY OF ORGANIC METHOD CALIBRATIONS (Cont'd)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Volatiles	Initial	624	Minimum of 3 levels, lowest near but above MDL. If % RSD ≤ 35%, use avg RF. Otherwise, calibration curve employed.	8260B	Minimum of 5 levels, %RSD for RF for CCCs[4] < 30.0% SPCCs[5]:RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform
	Continuing	624	1 level every 24 hours Acceptance criteria are found in the method and SOP	8260B	Mid-level standard every 12 hours (after tuning) *%Drift for* CCCs[(4)] < 20.0% between RF from standard and avg RF from initial SPCCs[(5)]: RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform
	Ending	624	N/A	8260B	N/A
	Other	624	BFB[(6)] tuning at the beginning of every 24 hour shift.	8260B	BFB[(6)] tuning at the beginning of every 12 hour shift.

TABLE 8.5-6
SUMMARY OF ORGANIC METHOD CALIBRATIONS (Cont'd)

Footnotes

[1] National Pollutant Discharge Elimination System.

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[3] TCDD - 2,3,7,8-Tetrachlorodibenzo-p-dioxin.

[4] CCC - Continuing Calibration Compounds.

[5] SPCC - System Performance Check Compound.

[6] BFB – Bromofluorobenzene.

[7] DFTPP – Decafluorotriphenylphosphine.

[8] Footnote deleted.

[9] Method not listed in 40 CFR Part 136.

TABLE 8.6-1
PRECISION AND ACCURACY MEASUREMENTS

Measurement	Definition
Accuracy	The degree of agreement of a measurement with an accepted reference or true value. The only true or known values in the laboratory are spiked samples. Expressed as laboratory control sample (LCS) percent recovery (% R): $$LCS\ \%\ Recovery = \frac{X}{t} x\ 100$$ where: X = observed concentration t = concentration of spike added Expressed as matrix spike/matrix spike duplicate (MS/MSD) sample percent recovery (% R): $$MS / MSD\ \%\ Recovery = \frac{X_s - X}{t} x\ 100$$ where: X_s = observed concentration in spiked sample X = observed concentration in unspiked sample t = concentration of spike added
Precision	The measure of analytical reproducibility of two values. Expressed as the relative percent difference (RPD) of two values. $$RPD = \left[\frac{\lvert X_1 - X_2 \rvert}{\left(\frac{X_1 + X_2}{2} \right)} \right] x\ 100$$ where: X_1 = first observed concentration X_2 = second observed concentration

TABLE 8.6-1
PRECISION AND ACCURACY MEASUREMENTS (Cont'd)

Measurement	Definition
Arithmetic mean	The average of a set of values. $\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$ where: $\bar{x}$ = the mean x_i = the i^{th} data value n = number of data values
Standard Deviation	A measure of the random (probable) error associated with a single measurement within a data set. $s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$ where: s = sample standard deviation $\bar{x}$ = the mean x_i = the i^{th} data value n = number of data values
Quality Control Chart	A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.
ACCURACY	
Upper Control Limit (UCL)	$UCL = \bar{x} + 3s$
Upper Warning Limit (UWL)	$UWL = \bar{x} + 2s$
Lower Warning Limit (LWL)	$LWL = \bar{x} - 2s$
Lower Control Limit (LCL)	$LCL = \bar{x} - 3s$
PRECISION	
RPD	Zero to (mean RPD + 3s)

TABLE 8.11-1
INSTRUMENT MAINTENANCE SCHEDULE
ION CHROMATOGRAPH [1]

As Needed	Daily	Weekly	Monthly
Clean micro-membrane suppressor when decreases in sensitivity are observed.	Check plumbing/leaks.	Check pump heads for leaks.	Check all air and liquid lines for discoloration and crimping, if indicated.
Check fuses when power problems occur.	Check gases.	Check filter (inlet)	Check/change bed supports guard and analytical columns, if indicated.
Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.	Check pump pressure.		
De-gas pump head when flow is erratic.	Check conductivity meter.		

TABLE 8.11-2
INSTRUMENT MAINTENANCE SCHEDULE
TOTAL ORGANIC HALIDE ANALYZER [1]

Daily	As Needed
Check electrodes for damage; polish the electrodes.	Examine and clean or replace pyrolysis tube.
Replace dehydrating fluid and electrolyte fluid.	Clean titration cell.
Clean quartz boat.	Observe gas flow.
Observe check valves during use for backfeed.	Replace reference electrode fluid.
At end of each day of use, wash out absorption module, empty electrolyte and fill cell with DI water. Empty dehydrator tube	Change quartz wool.
Perform cell performance check.	Replace O-rings and seals.

TABLE 8.11-3
INSTRUMENT MAINTENANCE SCHEDULE
HIGH PRESSURE LIQUID CHROMATOGRAPH [1]

Daily	As Needed
Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent.	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.
Check gas supply.	Oil autosampler slides when sample does not advance.
Flush with an appropriate solvent to remove all bubbles.	Rinse flow cell with 1N nitric acid if sensitivity low.
Pre-filter all samples.	Change pump seals when flow becomes inconsistent.
	Repack front end of column Backflush column.

TABLE 8.11-4
INSTRUMENT MAINTENANCE SCHEDULE
ICP AND ICP/MS [1]

Daily	Monthly or As Needed	Semi-Annually	Annually
Check vacuum pump gage. (<10 millitorr)	Clean plasma torch assembly to remove accumulated deposits.	Change vacuum pump oil.	Notify manufacturer service engineer for scheduled preventive maintenance service.
Check that cooling water supply system is full and drain bottle is not full. Also that drain tubing is clear, tight fitting and has few bends.	Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance.	Replace coolant water filter. (may require more or less frequently depending on the quality of water)	
Check that nebulizer is not clogged.	Clean filters on back of power unit to remove dust.		
Check that capillary tubing is clean and in good condition.	Replace when needed: peristaltic pump tubing sample capillary tubing autosampler sipper probe		
Check that peristaltic pump windings are secure.	Check yttrium position. Check O-rings Clean/lubricate pump rollers.		
Check that high voltage switch is on.			
Check that torch, glassware, aerosol injector tube, bonnet are clean			

TABLE 8.11-5
INSTRUMENT MAINTENANCE SCHEDULE
CVAS AND CVAFS

Daily	As Needed	Annually
Change drying tube	Change pump tubing	Change Hg lamp
Check pump tubing/drain tubing	Check/change Hg lamp	
Check gas pressure	Clean optical cell	
Check aperture reading Check tubing	Lubricate pump	

TABLE 8.11-6
INSTRUMENT MAINTENANCE SCHEDULE
GAS CHROMATOGRAPH [1]

Daily *	As Needed
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance, or when column performance (e.g., peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Quarterly FID: clean detector, only as needed—not quarterly/or semi-annually.
Check temperatures of injectors and detectors. Verify temperature programs by RT shift.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Clean injector port weekly for TPH for 8015B, when breakdown fails; otherwise, when RT shift or bad samples run.	Annually FID: replace flame tip, only as needed. Only as needed: ECD--detector cleaning and re-foiling, whenever loss of sensitivity, erratic response, or failing resolution is observed
Check baseline level during analysis of run—not maintenance.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Watched weekly: check reactor temperature of electrolytic conductivity detector. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks, when analyzing pesticides; part of analysis—not maintenance. Clip column leader when chromatography looks bad—not daily.	Replace or repair flow controller if constant gas flow cannot be maintained. Replace fuse. Reactivate external carrier gas dryers. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. ECD: follow manufacturer's suggested maintenance schedule. Reactivate flow controller filter dryers when presence of moisture is suspected. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.

*No daily maintenance done on any instrument/method. Weekly change IPL on TPH instrument. Everything else is on an "as needed" basis.

TABLE 8.11-7
INSTRUMENT MAINTENANCE SCHEDULE
MASS SPECTROMETER [1]

Daily	Weekly	As Needed [2]	Quarterly	Semi-Annually	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43)	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between maintenance.	Check ion source and analyzer (clean, replace parts as needed)		Replace the exhaust filters on the mechanical rough pump every 1-2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	Check vacuum, relays, gas pressures and flows		
Check inlets, septa.		Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.	Change oil in the mechanical rough pump.		
Check baseline level.		Repair/replace jet separator.			
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates need for replacement.			

TABLE 8.11-8
INSTRUMENT MAINTENANCE SCHEDULE
TRAACS AUTO ANALYZER [1]

As Needed	Daily
Replaces air filter when progressive loss of air pressure is observed.	Check air pressure gauge (22 ± 2 psi)
Replace air valve tubing when occlusion in tubing is observed	Use recommended washout procedure (at end of analysis operations)
Change all pump tubes (or after 200 hours of pumping time, or after 1000 hours of pumping time)	
Clean sample probe shaft	
Replace pump platens	
Lightly lubricate the linear sample rails (use semi-fluid lubricant)	
Reaplce colorimeter lamp (or after 2500 hours of use)	

TABLE 8.11-9
INSTRUMENT MAINTENANCE SCHEDULE
ANALYTICAL/TOP LOADING BALANCES [1]

Daily	Annually
Check using Class 1-verified weights once daily or before use Clean pan and weighing compartment	Manufacturer cleaning and calibration

TABLE 8.11-10
INSTRUMENT MAINTENANCE SCHEDULE
REFRIGERATORS/WALK-IN COOLERS [1]

Daily	As Needed
Temperatures checked and logged	Refrigerant system and electronics serviced

TABLE 8.11-11
INSTRUMENT MAINTENANCE SCHEDULE
OVENS [1]

Daily	As Needed
Temperatures checked and logged	Electronics serviced

TABLE 8.11-12
Instrument Maintenance Schedule
Specific Digital Ion Analyzer [1]

Daily	As Needed
Daily when used: • Calibrate with check standards • Inspect electrode daily, clean as needed • Inspect electrode proper levels of filling solutions daily; fill as needed • Clean probe after each use	Electronics serviced

TABLE 8.11-13
INSTRUMENT MAINTENANCE SCHEDULE
TURBIDIMETER [1]

Daily	Monthly	As Needed
Daily when used: • Adjust linearity on varying levels of NTU standards. Standardize with NTU standards • Inspect cells	Clean instrument housing	Electronics serviced

TABLE 8.11-14
INSTRUMENT MAINTENANCE SCHEDULE
DISSOLVED OXYGEN METER [1]

Daily	As Needed
Daily when used: • Calibrate with saturated air • Check probe membrane for deterioration • Clean and replace membrane with HCl solution	• Electronics serviced • Clean and replace membrane with HCl solution

TABLE 8.11-15
INSTRUMENT MAINTENANCE SCHEDULE
CONDUCTANCE METER [1]

Daily	As Needed
Daily when used: • Check probe and cables • Inspect conductivity cell	Electronics serviced

TABLE 8.11-16
INSTRUMENT MAINTENANCE SCHEDULE
CHEMICAL OXYGEN DEMAND (COD) REACTOR [1]

Daily	As Needed
Daily when used: • Calibrate with check standards	Electronics serviced

TABLE 8.11-17
INSTRUMENT MAINTENANCE SCHEDULE
SPECTROPHOTOMETER [1]

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens	Check the zero % adjustment	Clean windows	Check instrument manual
	Clean sample compartment		Perform wavelength calibration
	Clean cuvettes		Replace lamp annually or when erratic response is observed
			Clean and align optics

TABLE 8.11-18
INSTRUMENT MAINTENANCE SCHEDULE
pH METER [1]

As Needed	Daily
Clean electrode	Inspect electrode. Verify electrodes are properly connected and filled
Refill reference electrode	Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer

TABLE 8.11-19
INSTRUMENT MAINTENANCE SCHEDULE
TOTAL ORGANIC CARBON ANALYZER [1]

Daily	As Needed	Weekly	Monthly
Check: • Oxygen supply • Persulfate supply • Acid supply • Carrier gas flow rate (~ 150 cc/min) • IR millivolts for stability (after 30 min. warm-up) • Reagent reservoirs	Check injection port septum after 50-200 runs Tube end-fitting connections after 100 hours or use Indicating drying tube NDIR zero, after 100 hours of use Sample pump, after 2000 hours for use Digestion vessel/condensation chamber, after 2000 hours of use Permeation tube, after 2000 hours of use NDIR cell, after 2000 hours of use Change pump tubing	Check liquid-flow-rate-pump-tubing conditions on autosampler Check injection port septum	Clean digestion vessel Clean condenser column Do the leak test

TABLE 8.11-20
Instrument Maintenance Schedule
Digestion Block [1]

Annually
Check temperature with NIST thermometer

TABLE 8.11-21
Instrument Maintenance Schedule
Flash Point Tester [1]

Daily
Check tubing Clean sample cup each use
Check gas
Clean flash assembly
Check stirrer

Footnote:

[1] Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.

TABLE 9.4-1
PROFICIENCY TESTING PROGRAMS

PT Sample Program Description	Analysis Performed	Frequency of Participation
NELAC Water Pollution (WP)	Per NELAC Fields of Testing list and individual state requirements	Semi-annual
NELAC Solid	Per NELAC Fields of Testing list and individual state requirements	Semi-annual
STL Corporate Double Blind	As requested	Annual
STL North Canton Internal PEs – Single or Double Blinds	As needed	As a follow-up to unacceptable PTs from other programs or internal/external audits
Non NELAC water pollution (WP)	As needed per state regulations	Per state requirements

Note: Various client and agency single and double blind PTs are introduced in the laboratory throughout the year.

Acronyms and Initialisms

ANSI	American National Standards Institute
ASQ	American Society for Quality
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
BOD	Biochemical Oxygen Demand
CCC	Calibration Check Compound
CF	Calibration Factor
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	Chain-of-Custody
COD	Chemical Oxygen Demand
CRDL	Contract Required Detection Limit
CRF	Cooler Receipt Form
CRQL	Contract Required Quantitation Limit
CSM	Customer Service Manager
CVAA	Cold Vapor Atomic Absorption (Spectroscopy)
DFTPP	Decafluorotriphenylphosphine
DOC	Dissolved Organic Carbon
DQO	Data Quality Objective
EH&S	Environmental Health and Safety
EPA	(U. S.) Environmental Protection Agency
FAS	Field Analytical Services
GC	Gas Chromatograph(y)
GC/MS	Gas Chromatography/Mass Spectrometry
HPLC	High Performance Liquid Chromatography
ICP	Inductively Coupled Argon Plasma (Spectroscopy)
ICP/MS	Inductively Coupled Argon Plasma/Mass Spectrometry
ICS	Interference Check Sample
IDL	Instrument Detection Limit
IS	Information Systems
IS	Internal Standard
ISO	International Organization for Standardization

Acronyms and Initialisms (cont'd)

IT	Information Technology
KRI	Key Result Indicator
LAN	Local Area Network
LCL	Lower Control Limit
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LWL	Lower Warning Limit
MDL	Method Detection Limit
MS	Matrix Spike
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NCM	Nonconformance Memo
NIST	National Institute of Standards Technology
NPDES	National Pollutant Discharge Elimination System
PAH	Polynuclear Aromatic Hydrocarbons (or PNA)
PC	Personal Computer
PCB	Polychlorinated Biphenyls
PDS	Post Digestion Spike
PT	Performance Test Sample
PM	Project Manager
PQL	Practical Quantitation Limit
PSRL	Project-Specific Reporting Limit
QA	Quality Assurance
QMP	Quality Management Plan
QAPP	Quality Assurance Project Plan or Quality Assurance Program Plan
QC	Quality Control
QuantIMS	TestAmerica North Canton Laboratory Information Management System

Acronyms and Initialisms (cont'd)

RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RFP	Request for Proposal
RL	Reporting Limit
RPD	Relative Percent Difference
RRF	Relative Response Factor
RSD	Relative Standard Deviation
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SOW	Statement of Work
SPCC	System Performance Check Compounds
SPLP	Synthetic Precipitation Leaching Procedure
SRM	Standard Reference Material
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
TOX	Total Organic Halides
UCL	Upper Control Limit
UPS	Uninterruptable Power Supply
USEPA	United States Environmental Protection Agency
UWL	Upper Warning Limit
VOA	Volatile Organic Analysis
WAN	Wide Area Network
WP	Water Pollution

Glossary

acceptance limits
Data quality limits specified for analytical method performance.

accuracy
Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100).

aliquot, aliquant
A measured portion of a sample taken for analysis.

analytical spike
A sample created by spiking target analytes into a prepared portion of a sample just prior to analysis. (Also see matrix spike.)

anomaly
See nonconformance.

areas needing improvement
Represent isolated instances of noncompliance or issues that are judged to have a less immediate impact on data quality. Laboratory management must correct the situation or otherwise ensure that the condition does not recur. This term replaces the previous term used "Observations."

arithmetic mean
The arithmetic mean ($\bar{x}$) is the average of a set of values. It is equal to the sum of the observed values divided by the number of observations. Also called "average".

where: $\bar{x}$ = the mean
x_i = the i^{th} data value
n = number of data values

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

Glossary (cont'd)

assessment
The evaluation process used to measure the performance or effectiveness of a system and its elements. Assessment is used as an all-inclusive term to denote any of the following: *performance*, systems, data and compliance audits, management systems reviews, peer reviews, inspections, or spot assessments.

associate
Employee.

audit
A planned and documented investigative evaluation of an item or process to determine its adequacy and effectiveness as well as compliance with established procedures, instructions, drawings, quality management plans, and other applicable documents.

benchmarking
A step-by-step method of improving performance by identifying and studying best practices and comparing them to industry practices.

bias
A systematic (consistent) error in test results. Bias is expressed as the difference between the population mean and the true or reference value, or as estimated from sample statistics, the difference between the sample average and the reference value.

blind performance evaluation sample
A sample either submitted to the laboratory or prepared in the laboratory whereby the concentrations of parameters of concern are known by the preparer and not by the laboratory.

calibration
Establishment of a relationship between various calibration standards and the measurements of them obtained by a measurement system, or portions thereof. The levels of the calibration standard should bracket the range of levels at which actual measurements are to be made. Calibration is also the act of making a scheduled comparison of instrument performance against national standards for instruments which measure physical parameters such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.

Glossary (cont'd)

calibration curve
The graphical relationship between the known values for a series of calibration standards and instrument responses.

calibration factor (CF)
The ratio of the instrument response of an analyte to the amount injected. CFs are used in external standard calibrations.

$$CF = \frac{Total\ Area\ of\ Peak}{Mass\ Injected}$$

calibration standard
A standard used to quantitate the relationship between the output of a sensor and a property to be measured. Calibration standards should be traceable to standard reference materials (provided by NIST, or other recognized standards agencies) or a primary standard.

certified reference material
A reference material accompanied by a certificate issued by an organization certifying the contents and concentration(s) of the material. (See also standard reference material.)

chain-of-custody (COC)
A system of documentation demonstrating the physical custody and traceability of samples.

check standard analyses
A standard (often a midpoint standard) analyzed at a frequency specified in the method or in a SOP to verify the continuing calibration of the standard curve.

client
Any individual or organization for whom items or services are furnished or work is performed in response to defined requirements and expectations.

Glossary (cont'd)

client sample
The material or collection media submitted to the laboratory for analysis. Field QC samples are considered client samples but laboratory QC samples are not counted as client samples when counting samples for QC batches.

coefficient of variation (relative standard deviation)
A measure of precision (relative dispersion). It is equal to the standard deviation (*s*) divided by the mean ($\bar{x}$) and multiplied by 100 to give a percentage value.

$$CV\ (RSD) = \left(\frac{s}{\bar{x}}\right) x\ 100$$

collocated samples
Independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. The results will indicate sampling as well as analytical variability.

comparability
Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

completeness
Completeness is a measure of the percentage of measurements that are judged to be valid measurements. At a minimum, the objective for completeness of data is 90% for each constituent analyzed. It is usually expressed as a percentage:

$$\%\ Completeness = \frac{V}{n} x\ 100$$

where: V = number of measurements judged valid
n = total number of measurements

Glossary (cont'd)

composite
A sample composed of two or more increments.

control chart
A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.

control table
A tabular presentation of test results with respect to time or sequence of measurement, together with limits within which the results are expected to lie when the analytical process is in a state of control.

controlled document
A document for which the distribution is known. Updates of the document are sent to the original recipients, unless the copy distributed is an uncontrolled copy.

corrective action
A measure taken to rectify conditions adverse to quality and, where necessary, to preclude their recurrence.

correlation coefficient
The correlation coefficient (r) is a determination of how closely data "fits" a straight line. It is a number between -1 and 1 that indicates the degree of linear relationship between two sets of numbers. A correlation coefficient of +1 (usually calculated to three decimal places or 1.000) means the data falls exactly on a straight line with positive slope. A correlation coefficient of -1 (or -1.000) means the data falls exactly on a straight line with negative slope.

customer
See client.

Glossary (cont'd)

data quality objective (DQO)
Data quality objectives (DQOs) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application (EPA 1994). Typically, DQOs are identified during project scope and development of sampling and analysis plans. In this QA manual, however, we refer to only the analytical DQOs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory. EPA has established six primary analytical DQOs for environmental studies: precision, accuracy, representativeness, completeness, comparability, and detectability.

The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. TestAmerica incorporates numerous QA and QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA and QC samples and their applications, described in Section 8.4 and are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples are received from the client as unknown samples. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses may include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), surrogate spikes, and yield tracers.

data validation
See validation - data.

data verification
See verification - data.

deficiency
See nonconformance or finding.

degrees of freedom
The number of independent deviations used in calculating an estimate of the standard deviation.

double blind performance evaluation sample
A sample that contains select parameters at defined levels. The levels are unknown to the laboratory. The laboratory is also unaware that the sample is a performance evaluation sample.

Glossary (cont'd)

duplicate sample analyses
Different aliquots of the same sample are analyzed to evaluate the precision of an analysis.

error
The difference between an observed or measured value and its true value.

field blank
A blank that is prepared and handled in the field and analyzed in the same manner as its corresponding client samples.

field matrix spike
A sample created by spiking target analytes into a sample in the field at the point of sample acquisition.

finding
Noncompliant practices or policies which have significant adverse impact on data quality, technical defensibility, or regulatory acceptance of data. Findings require immediate attention by the laboratory management and must be resolved to comply with TestAmerica quality documents and laboratory-established procedures often called deficiencies by auditors.

geometric mean
The n^{th} root of the product of all values in a set of n values or the antilogarithm of the arithmetic mean of the logarithms of all the values of a set of n values. The geometric mean is generally used when the logarithms of a set of values are nearly normally (Gaussian) distributed, such as is the case of much population data.

initial calibration
Analysis of a series of analytical standards at different specified concentrations; used to define the linearity and dynamic range of the response of an instrument to the target compounds prior to the analysis of samples.

inspection
Examination or measurement of an item or activity to verify conformance to specific requirements.

instrument detection limit (IDL)
IDL is a calculated estimate of instrument detectability defined by SW846 Method 6020 and used in SW846 Method 6010B.

Glossary (cont'd)

internal standard (IS)
A compound added to every standard, QC sample, client sample, or sample extract at a known concentration prior to analysis for the purpose of quantitation. For example, internal standards are used as the basis for quantitation of the target compounds by GC/MS.

linear regression
A statistical method for finding a straight line that best fits a set of two or more data points, thus providing a relationship between two or more variables.

matrix
The component or substrate which contains the analyte(s) of interest. Examples of matrices are water, soil or sediment, and air. Matrix is not synonymous with phase (liquid or solid).

matrix effect
An interference in the measurement of analyte(s) in a sample that is caused by materials in the sample. Matrix effects may cause elevated reporting limits or may prevent the acquisition of acceptable results.

matrix spike (MS)
An aliquot of a matrix fortified (spiked) with known quantities of specific compounds and subjected to an entire analytical procedure in order to indicate the appropriateness of the method for a particular matrix. The percent recovery for the respective compound(s) is then calculated.

matrix spike duplicate (MSD)
A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

may
Denotes permission but not a requirement.

mean
See arithmetic mean.

Glossary (cont'd)

measurement
The process or operation of ascertaining the extent, degree, quantity, dimensions, or capability with respect to a standard.

median
The middle value of a set of data when the data set is ranked in increasing or decreasing order.

method
An assemblage of techniques.

method blank (MB)
An analytical control consisting of all reagents, which may include internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination. Examples of method blanks are a volume of deionized or distilled laboratory water for water samples, a purified solid matrix for soil/sediment samples, or a generated zero air.

method detection limit (MDL)
The minimum concentration of an analyte that, in a given matrix and with a specific method, can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is operationally defined as:

$$MDL = st_{(n-1,\ =0.99)}$$

where:

s = the standard deviation of a number of measurements of a blind or sample matrix containing the analyte at a concentration near the lowest standard recommended in the method and

$t_{(n-1,\ =0.99)}$ = the student's value for a one-sided t-statistic appropriate for the number of samples used to determine (s), at the 99% confidence level and n-1 degrees of freedom.

modified method
A standard or reference method which has been changed to meet project or matrix requirements.

must
Denotes a requirement is mandatory and has to be met.

Glossary (cont'd)

notable practices
Laboratory practices that increase effectiveness and quality and represent improvements with respect to conventional laboratory operations.

nonconformance
An unplanned deviation from an established protocol or plan. The deviation may be the result of TestAmerica actions, then termed a deficiency. If the deviation is the result of events beyond the control of TestAmerica, it is termed an anomaly.

operational calibration
Routinely performed as part of instrument usage, such as the development of a standard calibration curve. Operational calibration is generally performed for instrument systems.

outlier
A result excluded from the statistical calculations due to being deemed "suspicious" when applying the "Grubbs Test" (or equivalent).

parameter
A constant or coefficient that describes some characteristic of a population (e.g., standard deviation, mean, regression coefficients). Also, a chemical being measured, i.e., an analyte.

percent difference
When two independent measurements of the same characteristics are available, it is possible to use the percent difference instead of the coefficient of variation to measure precision.

$$\%D = \left| \frac{X_1 - X_2}{X_1} \right| x\ 100\%$$

where: $\%D$ = percent difference
X_1 = first value
X_2 = second value

Glossary (cont'd)

percent recovery
A measure of accuracy determined from the comparison of a reported spike value to its true spike concentration.

$$\%R = \frac{observed\ conc.\ -\ sample\ conc.}{true\ spike\ conc.} \times 100\%$$

performance audit
See performance evaluation.

performance evaluation (PE)
A type of audit in which a known or characterized value is compared to the result obtained through the routine analysis of the sample in the laboratory to evaluate the proficiency of an analyst or laboratory.

performance test (PT)
See performance evaluation (PE)

periodic calibration
A calibration that is performed at prescribed intervals for equipment such as balances, thermometers, and balance weights. In general, they are performed on equipment that are distinct, singular purpose units, and are relatively stable in performance.

population
A generic term denoting any finite or infinite collection of individual things, objects, or events.

practical quantitation limit (PQL)
The lowest concentration a method can reliably achieve within limits of precision and accuracy and is derived from empirical, matrix-free method performance studies.

Glossary (cont'd)

precision
Precision is an estimate of variability, that is, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. The precision of a measurement system is affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent difference (RPD) for duplicate measurements.

preventive maintenance
An organized program within TestAmerica laboratories of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use.

primary standard
A material having a known, stable property that can be accurately measured or derived from established physical or chemical constants. It is readily reproducible and can be accepted (within stated limits) and used to establish the same value of another substance or item.

procedure
Detailed instructions to permit replication of a method. (See standard operating procedure.)

proficiency testing
A series of planned tests which will determine the ability of field technicians or laboratory analysts to perform routine analyses. The results from this testing may be used for comparison against established criteria or for relative comparisons among the data from a group of technicians or analysts.

project-specific reporting limit (PSRL)
See reporting limit.

protocol
Methodology specified in regulatory, authoritative, or contractual situations.

QC batch
The QC batch consists of a set of up to 20 field samples that behave similarly (i.e., same matrix) and are processed using the same procedures, reagents, and standards within the same time period.

Glossary (cont'd)

QC check sample
A reference matrix containing known concentrations of parameters of interest. If prepared in the laboratory, it is made using stock standard solutions independent of those used for calibration. If the results of these parameters do not meet acceptance criteria, corrective actions are taken.

qualification (personnel)
The characteristics of abilities gained through education, training, or experience, as measured against established requirements, such as standards or tests, that qualify an individual to perform a required function.

quality
The sum of features and properties/characteristics of a process, item, or service that bears on its ability to meet the stated needs of the user. TestAmerica has defined quality as meeting the needs of our clients, both internal and external.

quality assurance (QA)
An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the customer.

Quality Assurance Directive
QA directives are memos issued by the QA Director (or the QA Managers for their facility) to clarify policies, Procedures, and the QMP; or to give direction for an immediate action to ensure or maintain quality.

Quality Management Plan (QMP)
The Quality Management Plan for Environmental Analyses (QMP) is a formal document that describes quality systems in terms of organizational structure, functional responsibilities of management, and staff, and lines of authority. The QMP documents the QMS and describes both the organizational and project-specific principles, goals, controls, and tools of the QMS. The QMP provides the criteria and specifications for the generation of environmental analytical data.

Glossary (cont'd)

Quality Assurance Project or Program Plan (QAPP)
A formal document describing in comprehensive detail the necessary QA, QC, and other technical activities that must be implemented to ensure the results of the work performed will satisfy the stated performance criteria.

quality control (QC)
The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that it meets the stated requirements established by the client or by TestAmerica.

quality improvement
The process of improving the quality of operations. This process encourages worker recommendations for improvement of work processes and requires timely management evaluation and feedback or implementation.

quality management
That aspect of the overall management system of the organization that determines and implements the quality policy. Quality management includes strategic planning, allocation of resources, and other systematic activities (e.g., planning, implementation, and assessment) pertaining to the quality management system.

quality management system (QMS)
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, and implementation plan of an organization for ensuring quality in its work processes, products, and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

random error
Variations of repeated measurements that are random in nature and individually not predictable.

range
The difference between the largest and smallest numbers in a set of numbers.

Glossary (cont'd)

raw data
All documentation associated with the original recording of analytical results pertinent to a specific sample or set of samples. This may include laboratory worksheets, calculation forms, instrument-generated output, analyst notes, etc., from sample receipt through final reporting.

reagent water
Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. The reagent water's purity and acceptability is verified by analysis with each set of samples.

recovery
See percent recovery.

reference method
A method of known and demonstrated accuracy.

regression coefficients
The quantities describing the slope and intercept of a regression line.

relative error
An error expressed as a percentage of the true value or accepted reference value.

relative percent different (RPD)
Statistic for evaluating the precision of a replicate set. For replicate results:

$$RPD = \left[\frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2} \right)} \right] x\ 100$$

where: X_1 = first observed concentration
X_2 = second observed concentration

Glossary (cont'd)

relative response factor (RRF)
A measure of the relative mass spectral response of a compound compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. Because a RRF is the comparison of two responses, it is a unitless number. RRFs are determined by the following equation:
where: A = area of the characteristic ion measured

$$RRF = \frac{A_x}{A_{IS}} x \frac{C_{IS}}{C_x}$$

C = concentration
IS = internal standard
x = analyte of interest

relative standard deviation (RSD)
See coefficient of variation.

reporting limit (RL)
One of two types of reporting limit conventions within TestAmerica. The Reporting Limit (RL) is based on an evaluation of the PQLs at TestAmerica Laboratories and the expected method performance in routine water and soil matrices. Project Specific Reporting Limits (PSRLs) are reporting limits that are defined by project requirements.

representative sample
A sample taken to represent a lot or population as accurately and precisely as possible.

representativeness
Representativeness is the degree to which data accurately and precisely represent a characteristic of a *population, a variation in a physical or chemical property at a sampling point,* or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result (concentration) is representative of the constituent concentration in the sample matrix. At each TestAmerica laboratory, every effort must

Glossary (cont'd)

be made to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before subsampling.

reproducibility
The precision, usually expressed as a standard deviation, measuring the variability among results of measurements of the same sample at different laboratories.

response factor (RF)
A factor derived from the calibration of a compound that is used in the quantitation calculation of sample analytes. A response factor may be derived from an external standard calibration (then called a Calibration Factor) or from an internal standard calibration (then called a Relative Response Factor).

secondary standard
A material having a property that is calibrated against a primary standard.

self assessment
Assessments of work conducted by individuals, groups, or organizations directly responsible for overseeing or performing the work.

shall
Denotes a requirement that is mandatory and has to be met.

should
Denotes a guideline or recommendation.

standard addition
The procedure of adding known increments of the analyte of interest to a sample to cause increases in detection response to subsequently establish, by extrapolation of the plotted responses, the level of the analyte of interest present in the original sample.

Glossary (cont'd)

standard deviation
A measure of the dispersion about the mean of the elements in a population. The square root of the variance of a set of values:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$

where: s = standard deviation
$\exists$ = sum of
X = observed values
n = number of observations

standardization
The establishment of the value of a potential standard with respect to an established or known standard.

standard method
A method of known and demonstrated precision issued by an organization generally recognized as competent to do so.

standard operating procedure (SOP)
A written document that details an operation, analysis, or action, with prescribed techniques and steps, that is officially approved as the method for performing certain routine or repetitive tasks.

standard reference material (SRM)
A material produced in quantity, of which certain properties have been certified by the National Institute of Standards and Technology (NIST), formerly NBS, or other agencies to the extent possible to satisfy its intended use.

standard verification
Standard is checked by TestAmerica or the vendor versus a known specification.

Glossary (cont'd)

statistic
A constant or coefficient that describes some characteristic of a sample. Statistics are used to estimate parameters of populations.

stock solution
A concentrated solution of analyte(s) or reagent(s) prepared and verified by prescribed procedure(s), and used for preparing working standards or standard solutions.

subsample
A portion taken from a sample. A laboratory sample may be a subsample of a gross sample; similarly, a test portion may be a subsample of a laboratory sample.

supplier
See vendor.

surrogate (surrogate standard)
Compounds, when required by a method, that are used added to every blank, sample, LCS, matrix spike, matrix spike duplicate, and standard. They are used to evaluate analytical efficiency by measuring recovery. Surrogates include brominated, fluorinated, or isotopically-labeled compounds that are not expected to be detected in environmental media.

systematic error
The condition of a consistent deviation of the results of a measurement process from the reference or known level.

systems audit or evaluation
A systematic on-site qualitative review of facilities, procedures, equipment, training, record keeping, data verification, and reporting aspects of a quality assurance system to arrive at a measure of the capability of the system. Within TestAmerica, system audits or evaluations are performed on a periodic basis under the direction of the TestAmerica Corporate Director of Quality Assurance.

technique
Physical or chemical principle for characterizing materials of chemical systems.

Glossary (cont'd)

traceability of data
The entire documented chain of acquired data from the original acquisition effort through to the final tabulation, synthesis, reduction, and storage activities. The documentation will allow complete reconstruction of the data.

traceability of samples
During all environmental monitoring field efforts, acquired samples will be assigned specific and unique identification numbers. These sample numbers shall be accompanied by documentation (chain-of-custody form) which clearly identifies all parameters associated with sample acquisition. All additional sample numbering systems applied to the sample must be clearly cross-referenced to the field sample number to provide for traceability of samples from acquisition to reporting of sample results.

traceability of standards
The ability of an analytical standard material used for calibration purposes to be traced to its source. The standards used by TestAmerica must be traceable via written documentation to sources which produce or sell verified or certified standards, i.e., National Institute for Standards and Technology, or vendors preparing standards from those sources which they have certified.

validation - computer software
The process of establishing documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting predetermined specifications and quality attributes. This process demonstrates and documents that the software performs correctly and meets all specified requirements.

validation - data
The process of a second party performing a systematic review of the raw and final data produced by a laboratory using predetermined criteria to ascertain the validity of the data with respect to the criteria (e.g., HAZWRAP data validation).

vendor
Any individual or organization furnishing items or services or performing work according to a procurement document. This is an all-inclusive term used in place of any of the following: supplier, seller, contractor, subcontractor, or consultant.

Glossary (cont'd)

verification - computer software
The process of checking the accuracy of manually entered or automatically (electronically) calculated information.

verification - data
The process of reviewing data to ensure that data reduction has been correctly performed and that analytical results to be reported correspond to the data acquired and processed.

APPENDIX G
Responsibilities of Owner and Remedial Party

Responsibilities

The responsibilities for implementing the Site Management Plan ("SMP") for the Former Monarch Chemicals Facility (the "site"), number VCA No. V00119-8, are divided between the site owner(s) and a Remedial Party, as defined below. The owner and tenant is/are currently listed as:

Site owner:
FNLR Compounds Matter Too LLC
c/o Fortress Investment Group LLC
1345 Avenue of the Americas, 46th Floor
New York, New York 10105
Attn: William Turner and Chase Romney

Site tenant:
Univar
9370 Pratolino Villa Drive
Dublin, Ohio 43016
Attn: Jack Spicuzza

Solely for the purposes of this document and based upon the facts related to a particular site and the remedial program being carried out, the term Remedial Party ("RP") refers to any of the following: certificate of completion holder, volunteer, applicant, responsible party, and, in the event the New York State Department of Environmental Conservation ("NYSDEC") is carrying out remediation or site management, the NYSDEC and/or an agent acting on its behalf. The RP is:

H.B. Fuller Company
1200 Willow Lake Blvd
PO Box 64683
St. Paul, Minnesota 55164
Attn: Jonathan Blaine

Nothing on this page shall supersede the provisions of an Environmental Easement, Consent Order, Consent Decree, agreement, or other legally binding document that affects rights and obligations relating to the site.

Site Owner's Responsibilities:

1) The owner shall follow the provisions of the SMP as they relate to future construction and excavation at the site.

2) In accordance with a periodic time frame determined by the NYSDEC, the owner shall periodically certify, in writing, that all Institutional Controls set forth in a(n) Deed Restriction remain in place and continue to be complied with. The owner shall provide a written certification to the RP, upon the RP's request, in order to allow the RP to include the certification in the site's Periodic Review Report (PRR) certification to the NYSDEC.

3) In the event the site is delisted, the owner remains bound by the Deed Restriction and shall submit, upon request by the NYSDEC, a written certification that the Deed Restriction is still in place and has been complied with.

4) The owner shall grant access to the site to the RP and the NYSDEC and its agents for the purposes of performing activities required under the SMP and assuring compliance with the SMP.

5) The owner is responsible for assuring the security of the remedial components located on its property to the best of its ability. If damage to the remedial components or vandalism is evident, the owner shall notify the site's RP and the NYSDEC in accordance with the timeframes indicated in Section 1.3 - Notifications.

6) If some action or inaction by the owner adversely impacts the site, the owner must notify the site's RP and the NYSDEC in accordance with the time frame indicated in Section 1.3 - Notifications and coordinate the performance of necessary corrective actions with the RP.

7) The owner must notify the RP and the NYSDEC of any change in ownership of the site property (identifying the tax map numbers in any correspondence) and provide contact information for the new owner of the site property. 6 NYCRR Part contains notification requirements applicable to any construction or activity changes and changes in ownership. Among the notification requirements is the following: Sixty days prior written notification must be made to the NYSDEC. Notification is to be submitted to the NYSDEC Division of Environmental Remediation's Site Control Section. Notification requirements for a change in

use are detailed in Section 1.3 of the SMP. A change of use includes, but is not limited to, any activity that may increase direct human or environmental exposure (e.g., day care, school or park). A 60-Day Advance Notification Form and Instructions are found at http://www.dec.ny.gov/chemical/76250.html.

8) In accordance with the tenant notification law, within 15 days of receipt, the owner must supply a copy of any vapor intrusion data, that is produced with respect to structures and that exceeds NYSDOH or OSHA guidelines on the site, whether produced by the NYSDEC, RP, or owner, to the tenants on the property. The owner must otherwise comply with the tenant and occupant notification provisions of Environmental Conservation Law Article 27, Title 24.

Remedial Party Responsibilities

1) The RP must follow the SMP provisions regarding any construction and/or excavation it undertakes at the site.

2) The RP shall report to the NYSDEC all activities required for remediation, operation, maintenance, monitoring, and reporting. Such reporting includes, but is not limited to, periodic review reports and certifications, electronic data deliverables, corrective action work plans and reports, and updated SMPs.

3) Before accessing the site property to undertake a specific activity, the RP shall provide the owner advance notification that shall include an explanation of the work expected to be completed. The RP shall provide to (i) the owner, upon the owner's request, (ii) the NYSDEC, and (iii) other entities, if required by the SMP, a copy of any data generated during the site visit and/or any final report produced.

4) If the NYSDEC determines that an update of the SMP is necessary, the RP shall update the SMP and obtain final approval from the NYSDEC. Within 5 business days after NYSDEC approval, the RP shall submit a copy of the approved SMP to the owner(s).

5) The RP shall notify the NYSDEC and the owner of any changes in RP ownership and/or control and of any changes in the party/entity responsible for the operation, maintenance, and monitoring of and reporting with respect to any remedial system (Engineering Controls). The RP shall provide contact information for the new party/entity. Such activity constitutes a Change of Use pursuant to 375-1.11(d) and requires 60-days prior notice to the NYSDEC. A 60-Day Advance Notification Form and Instructions are found at http://www.dec.ny.gov/chemical/76250.html .

6) The RP shall notify the NYSDEC of any damage to or modification of the systems as required under Section 1.3 - Notifications of the SMP.

7) Prior to a change in use that impacts the remedial system or requirements and/or responsibilities for implementing the SMP, the RP shall submit to the NYSDEC for approval an amended SMP.

8) Any change in use, change in ownership, change in site classification (*e.g.*, delisting), reduction or expansion of remediation, and other significant changes related to the site may result in a change in responsibilities and, therefore, necessitate an update to the SMP and/or updated legal documents. The RP shall contact the NYSDEC project manager to discuss the need to update such documents.

Change in RP ownership and/or control and/or site ownership does not affect the RP's obligations with respect to the site unless a legally binding document executed by the NYSDEC releases the RP of its obligations.

Future site owners and RPs and their successors and assigns are required to carry out the activities set forth above.

APPENDIX H
Boring Logs and Well Construction Logs

DELTA ENVIRONMENTAL CONSULTANTS

TEST BORING LOG

BORING NO.: MW-114

PROJECT: Geneva, NY									Sheet 1 of 1
CLIENT: HB Fuller									
DELTA PROJECT NO: V004210-1									
DRILLING METHOD: H.S.A.					SAMPLER	BIT SIZE	CORE	CASING	
DRILLING RIG: CME 55					Split-Spoon	NA	NA	NA	DATE: 7-14-05
DRILLER: Lyon Drilling									
DEPTH IN FT.	SAMPLE NO.	BLOWS PER 6"	REC. (ft.)	PID (ppm)	SOIL DESCRIPTION				REMARKS
	1	NA	3.0	2.0	Brn, Silt and Clay with little gravel and cinder fill, moist				
1.0									
2.0									
									Soil sample
3.0									MW-114A
									at 2' to 4'
4.0								4.0'	
	2		4.0	1.0	Brn, Silt and Clay, damp				
5.0								5.2'	
6.0				1.0	Brn, F. Sand, moist to vy moist				
								6.7'	
7.0									
				2.0	Brn, Vy F. Sand with little silt, moist to vy moist				
8.0									
	3		4.0	3.0	same as above, wet				Soil sample
9.0									MW-114B
									at 8' to 12'
10.0									
11.0									
12.0								12.0'	
	4		4.0	2.0	Brn, F. Sand, wet				
13.0									
14.0									
15.0								14.9'	
				0.0	Brn, Clay, wet				
16.0								16.0'	
					Boring terminated at 16'				
17.0					Monitoring well set at 14' BG. Well screen 4' to 14', sand pack				
					2.7' to 14', bentonite seal 1.2' to 2.7', grout 0' to 1.2', flush mount				
18.0					casing				

DELTA ENVIRONMENTAL CONSULTANTS

TEST BORING LOG

BORING NO.: MW-115

PROJECT: Geneva, NY									Sheet 1 of 1
CLIENT: HB Fuller									
DELTA PROJECT NO: V004210-1									
DRILLING METHOD: H.S.A.					SAMPLER	BIT SIZE	CORE	CASING	
DRILLING RIG: CME-55					Split-Spoon	NA	NA	NA	DATE: 7-12-05
DRILLER: Lyon Drilling									
DEPTH IN FT.	SAMPLE NO.	BLOWS PER 6"	REC. (ft.)	PID (ppm)	SOIL DESCRIPTION				REMARKS
	1	NA	3.5	0.0	Gravel Fill, moist				
1.0								0.9'	
				0.0	Brn, Silt, moist				
2.0									
								2.5'	Soil sample
3.0				0.0	Brn, Vy F. Sand with little silt, vy moist				MW-115A
									at 1' to 4'
4.0									
	2		4.0	0.0	same as above				
5.0									
6.0									
7.0									
8.0									
	3		4.0	0.0	same as above				Soil sample
9.0								8.9'	MW-115B
				0.0	Brn, Vy F. Sand, wet				at 8' to 12'
10.0									
11.0								11.0'	
				0.0	Brn, Clay, stiff, moist				
12.0									
	4		4.0	0.0	same as above				
13.0									
14.0									
15.0									
16.0								16.0'	
					Boring terminated at 16'				
17.0					Monitoring well set at 14' BG. Well screen 4' to 14', sand pack				
					2.7' to 14', bentonite seal 1.2' to 2.7', grout 0' to 1.2', flush mount				
18.0					casing				

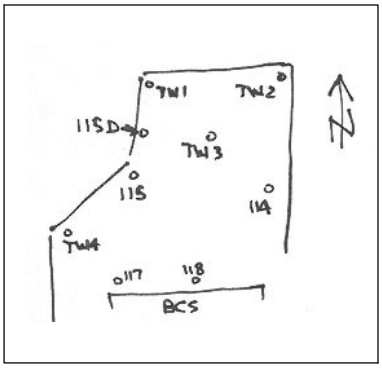
ECOR Solutions, Inc.
3191 Copper Oaks Place
Woodbury, MN
612.382.3763
kbrapp@comcast.net

Lithologic Log

Boring No.: MW-115D

Well No.: MW-115D

location sketch



Project Name:	**Former Monarch Chem**	**Drilling Co:**	**Nothnagle**
Address:	**61 Gates Ave**	**Driller:**	**Kevin Busch**
	Geneva, NY	**Method:**	**HSA**
Geologist:	**R. A. Wojciak**	**Diameter:**	**6 1/4" ID**
Start Date:	**11/10/2010**	**Total Depth:**	**24'**
End Date:	**11/12/2010**	**Note:**	**background PID 0.1 - 0.5 ppm**
Comment:	**flush w/ grade well, 2" PVC riser, 2" x 5' SS screen, 10 slot**		

Depth (ft)	Well Constr	Sampler Interval	Percent Recovery	Chemistry Sample	PID (ppm)	Description
0					0.2	Fill, parking lot/driveway, black silt w/ sand and gravel
2			75		0.0	brown, silty, very fine grained san
4					0.4	brown, very fine grained sand
6			100		0.5	brown, sandy silt
8					0.5	brown clay, 1" thick sandy clay seam at 7 1/2' depth, moist
					0.1	brown, very fine grained sand
					0.2	brown, fine grained sand, wet
10			100		0.1	brown sandy clay, moist
					0.1	brown, very fine grained sand, wet
					0.1	brown, clayey silt
12					0.1	brown, sandy clay

ECOR Solutions, Inc.
3191 Copper Oaks Place
Woodbury, MN
612.382.3763
kbrapp@comcast.net

Lithologic Log

Boring No.: MW-115D

Well No.: MW-115D

Depth (ft)	Well Constr	Sampler Interval	Percent Recovery	Chemistry Sample	PID (ppm)	Description
14			100		0.1	grayish green, very fine grained sand, wet
					0.1	grayish green silty clay, moist
					0.1	grayish green, clayey, fine grained sand, moist
16					0.1	grayish green, silty clay, moist
					0.1	gray silty clay, moist to wet
					0.1	grayish green, clayey silt, wet
18			100		0.1	grayish green sandy clay, moist
					0.1	gray, clayey, very fine grained sand, wet
20					0.1	grayish green, fine grained sand, wet
					0.1	grayish green, silty, very fine grained sand, wet
22			50		0.1	
					0.1	grayish green, very fine grained sandy silt, wet
24					0.1	grayish green, very fine grained sand, wet

DELTA ENVIRONMENTAL CONSULTANTS

TEST BORING LOG

BORING NO.: MW-116

PROJECT: Geneva, NY									Sheet 1 of 1
CLIENT: HB Fuller									
DELTA PROJECT NO: V004210-1									
DRILLING METHOD: H.S.A.					SAMPLER	BIT SIZE	CORE	CASING	
DRILLING RIG: CME-55					Split-Spoon	NA	NA	NA	DATE: 7-12-05
DRILLER: Lyon Drilling									
DEPTH IN FT.	SAMPLE NO.	BLOWS PER 6"	REC. (ft.)	PID (ppm)	SOIL DESCRIPTION				REMARKS
	1	NA	1.4	0.0	Gravel Fill and Silt, moist				
1.0									
2.0									
									Soil sample MW-116A at 2' to 4'
3.0									
4.0								4.0'	
	2		4.0	0.0	Brn, Silt and Clay, moist				
5.0									
6.0									
								6.5'	
7.0				0.0	Brn, Vy F. Sand and Silt, wet				
								7.6'	
8.0				0.0	Brn, Clay, stiff, moist			8.0'	
	3		4.0	0.0	Brn, Silt with little vy f. sand, vy moist				Soil sample MW-116B at 8' to 14'
9.0									
10.0									
11.0									
12.0									
	4		4.0	0.0	same as above				
13.0									
14.0									
15.0									
16.0								16.0'	
					Boring terminated at 16'				
17.0					Monitoring well set at 14' BG. Well screen 4' to 14', sand pack				
					2.7' to 14', bentonite seal 1.2' to 2.7', grout 0' to 1.2', flush mount				
18.0					casing				

DELTA ENVIRONMENTAL CONSULTANTS

TEST BORING LOG

BORING NO.: MW-117

PROJECT: HB Fuller, Geneva, NY								Sheet 1 of 1
CLIENT: HB Fuller								
DELTA PROJECT NO: V0042101								
DRILLING METHOD: 4.25" H.S.A					SAMPLER	BIT SIZE	CORE	CASING
DRILLING RIG: CME 55					Macro Core	NA	NA	NA
DRILLER: Lyon Drilling					INSPECTOR: Mark J Schumacher			DATE: 7-12-05
DEPTH IN FT.	SAMPLE NO.	BLOWS PER 6"	REC. (ft.)	PID (ppm)	SOIL DESCRIPTION			REMARKS
	1	NA	1.3	0	Brn., Silt and F. Gravel, damp, no odors			
1.0								
2.0								
3.0								
4.0							4.0'	
	2		2.5	138	Black, F. Sand, moist, strong petroleum odor, black staining			
5.0								
6.0								
				138	wet at 6.0 feet			Soil sample MW-117A from depth of 6.0' - 10.0'
7.0								
8.0								
	3		4.0	138	same as above			
9.0								
							9.8'	
10.0								
				68	Brn., F. Sand, wet, light petroleum odor			
11.0							11.2'	
				48	Brn., Silt and Clay, wet, light petroleum odor			
12.0							12.0'	
	4		4.0	26	Brn., Clay, wet, trace petroleum odor			Soil sample MW-117B from depth of 12.0' - 16.0'
13.0								
14.0								
				0	no odors			
15.0								
16.0							16.0'	
					Boring Terminated at 16.0'			
17.0					Well set to 14' with 10' of screen.			
					Sand 2.6' to 14', Bentonite Seal 1.1' to 2.6', Grout 0 - 1.1'			
18.0								

DELTA ENVIRONMENTAL CONSULTANTS

TEST BORING LOG

BORING NO.: MW-118

PROJECT: Geneva, NY									Sheet 1 of 1
CLIENT: HB Fuller									
DELTA PROJECT NO: V004210-1									
DRILLING METHOD: H.S.A.					SAMPLER	BIT SIZE	CORE	CASING	
DRILLING RIG: CME-55					Split-Spoon	NA	NA	NA	DATE: 7-12-05
DRILLER: Lyon Drilling									
DEPTH IN FT.	SAMPLE NO.	BLOWS PER 6"	REC. (ft.)	PID (ppm)	SOIL DESCRIPTION				REMARKS
	1	NA	1.7	1.8	Gravel Fill with trace silt, moist				
1.0									
2.0									
									Soil sample MW-118A at 2' to 4'
3.0									
4.0								4.0'	
	2		4.0	1.8	Brn, Silt and Clay, stiff, moist				
5.0									
6.0								6.1'	
				8.2	Brn, Vy F. Sand, wet				Soil sample MW-118B at 6' to 11'
7.0									
8.0									
	3		4.0	8.2	same as above				
9.0									
10.0									
11.0								11.1'	
				0.0	Brn, Silt and Clay, vy moist				
12.0									
	4		4.0	0.0	same as above				
13.0									
14.0									
15.0									
16.0								16.0'	
					Boring terminated at 16'				
17.0					Monitoring well set at 14' BG. Well screen 4' to 14', sand pack 2.7' to 14', bentonite seal 1.2' to 2.7', grout 0' to 1.2', flush mount casing				
18.0									

DELTA ENVIRONMENTAL CONSULTANTS

TEST BORING LOG

BORING NO.: MW-119

PROJECT: Geneva, NY									Sheet 1 of 1
CLIENT: HB Fuller									
DELTA PROJECT NO: V004210-1									
DRILLING METHOD: H.S.A.					SAMPLER	BIT SIZE	CORE	CASING	
DRILLING RIG: CME-55					Split-Spoon	NA	NA	NA	DATE: 7-11-05
DRILLER: Lyon Drilling									
DEPTH IN FT.	SAMPLE NO.	BLOWS PER 6"	REC. (ft.)	PID (ppm)	SOIL DESCRIPTION				REMARKS
	1	NA	2.5	0.0	Gravel Fill, moist				
1.0									
2.0								2.1'	
				8.7	Brn, Silt with some gravel fill, moist				Soil sample MW-119A at 2' to 4'
3.0									
4.0								4.0'	
	2		4.0	1.5	Brn, Silt and Clay, stiff				
5.0									
6.0					water noted in borehole				
7.0									
8.0									
	3		4.0	1.5	same as above			8.8'	
9.0									
				3.7	Brn, vy f. sand, wet				Soil sample MW-119B at 8' to 12'
10.0									
11.0									
								11.4'	
12.0				3.7	Brn, Silt and Clay, wet			12.0'	
	4		4.0	1.0	Brn, Silt and F.Sand (interbedded), wet				
13.0									
14.0								14.1'	
				1.0	Gray, Clay, moist				
15.0									
16.0								16.0'	
					Boring terminated at 16'				
17.0					Monitoring well set at 14' BG. Well screen 4' to 14', sand pack 2.7' to 14', bentonite seal 1.2' to 2.7', grout 0' to 1.2', flush mount casing				
18.0									

DELTA ENVIRONMENTAL CONSULTANTS

TEST BORING LOG

BORING NO.: MW-120

PROJECT: Geneva, NY									Sheet 1 of 1
CLIENT: HB Fuller									
DELTA PROJECT NO: V004210-1									
DRILLING METHOD: H.S.A.					SAMPLER	BIT SIZE	CORE	CASING	
DRILLING RIG: CME-55					Split-Spoon	NA	NA	NA	DATE: 7-11-05
DRILLER: Lyon Drilling									
DEPTH IN FT.	SAMPLE NO.	BLOWS PER 6"	REC. (ft.)	PID (ppm)	SOIL DESCRIPTION				REMARKS
	1	NA	3.1	0.0	Gravel Fill with little silt, moist				
1.0								1.0'	
				3.6	Brn, Silt with trace gravel fill, moist				
2.0									
									Soil sample MW-120A at 2' to 4'
3.0									
4.0								4.0'	
	2		3.5	2.2	Brn, F. Sand, moist				
5.0									
6.0					wet @ 6.0'				
7.0									
8.0									
	3		4.0	2.2	same as above			8.7'	
9.0									
				2.8	Brn, Silt and Clay, trace f. sand, wet				Soil sample MW-120B at 8' to 12'
10.0									
11.0									
12.0								12.0'	
	4		4.0	0.0	Brn, F. Sand and Silt (interbedded), wet				
13.0									
14.0									
15.0									
16.0								16.0'	
					Boring terminated at 16'				
17.0					Monitoring well set at 14' BG. Well screen 4' to 14', sand pack 2.7' to 14', bentonite seal 1.2' to 2.7', grout 0' to 1.2', flush mount casing				
18.0									

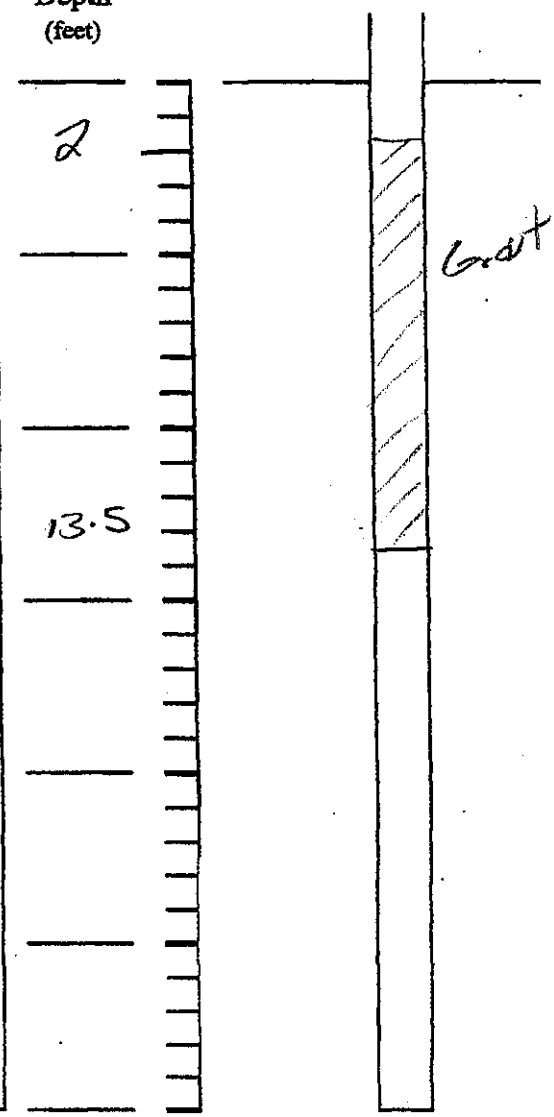
WELL DECOMMISSIONING RECORD

Site Name: H.B. Fuller	Well I.D.: MW-119
Site Location: Gates Ave. Geneva N.Y.	Driller: T. Mangefrida
Drilling Co.: Nothnagle Drilling	Inspector:
	Date: 8-9-16

DECOMMISSIONING DATA
(Fill in all that apply)

OVERDRILLING	
Interval Drilled	
Drilling Method(s)	
Borehole Dia. (in.)	
Temporary Casing Installed? (y/n)	
Depth temporary casing installed	
Casing type/dia. (in.)	
Method of installing	
CASING PULLING	
Method employed	
Casing retrieved (feet)	
Casing type/dia. (in)	
CASING PERFORATING	
Equipment used	
Number of perforations/foot	
Size of perforations	
Interval perforated	
GROUTING	
Interval grouted (FBLS)	13.5-2
# of batches prepared	1
For each batch record:	
Quantity of water used (gal.)	7.8
Quantity of cement used (lbs.)	94
Cement type	TYPE I
Quantity of bentonite used (lbs.)	3.9
Quantity of calcium chloride used (lbs.)	—
Volume of grout prepared (gal.)	10
Volume of grout used (gal.)	2

WELL SCHEMATIC*



COMMENTS: Removed Surface Completion and cut PVC 2FT. BGS Tremie grout abandoned in place

* Sketch in all relevant decommissioning data, including: interval overdrilled, interval grouted, casing left in hole, well stickup, etc.

Thomas H. Mangefrida
Drilling Contractor

Department Representative

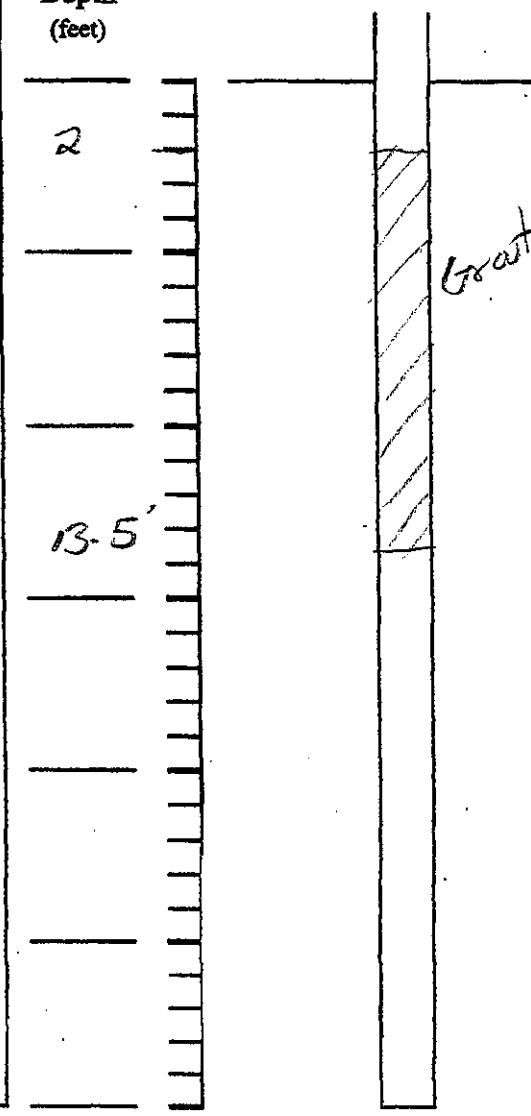
WELL DECOMMISSIONING RECORD

Site Name: H-B- Fuller	Well I.D.: MW-120
Site Location: Gates Ave Geneva N.Y.	Driller: T. Mangefrida
Drilling Co.: Nothnagle Drilling	Inspector:
	Date: 8-9-10

DECOMMISSIONING DATA
(Fill in all that apply)

OVERDRILLING	
Interval Drilled	
Drilling Method(s)	
Borehole Dia. (in.)	
Temporary Casing Installed? (y/n)	
Depth temporary casing installed	
Casing type/dia. (in.)	
Method of installing	
CASING PULLING	
Method employed	
Casing retrieved (feet)	
Casing type/dia. (in)	
CASING PERFORATING	
Equipment used	
Number of perforations/foot	
Size of perforations	
Interval perforated	
GROUTING	
Interval grouted (FBLS)	13.5 - 2
# of batches prepared	
For each batch record:	
Quantity of water used (gal.)	7.8
Quantity of cement used (lbs.)	94
Cement type	Type I
Quantity of bentonite used (lbs.)	3.9
Quantity of calcium chloride used (lbs.)	
Volume of grout prepared (gal.)	10
Volume of grout used (gal.)	9

WELL SCHEMATIC*



COMMENTS: Removed surface completion and cut PVC off 2ft BGS. Tremie grout abandoned in place

* Sketch in all relevant decommissioning data, including: interval overdrilled, interval grouted, casing left in hole, well stickup, etc.

Thomas H. Mangefrida
Drilling Contractor

Department Representative

DELTA ENVIRONMENTAL CONSULTANTS

TEST BORING LOG

BORING NO.: MW-121

PROJECT: Geneva, NY									Sheet 1 of 1
CLIENT: HB Fuller									
DELTA PROJECT NO: V004210-1									
DRILLING METHOD: H.S.A.					SAMPLER	BIT SIZE	CORE	CASING	
DRILLING RIG: LM-1					Split-Spoon	NA	NA	NA	DATE: 7-13-05
DRILLER: Lyon Drilling									
DEPTH IN FT.	SAMPLE NO.	BLOWS PER 6"	REC. (ft.)	PID (ppm)	SOIL DESCRIPTION				REMARKS
	1	NA	1.7	0.0	Railroad cinders				
1.0									1.1'
				0.0	Brn, F. Sand, dry				
2.0									
									Soil sample
3.0									3.1' MW-121A
				0.0	Brn, Silt, damp				at 2' to 4'
4.0									4.0'
	2		2.9	8.4	Brn, F. Sand with some silt, moist				
5.0									
					wet @ 5.5'				
6.0									
7.0									
8.0									8.0'
	3		4.0	9.5	Brn, Silt and Clay, wet				Soil sample
9.0									MW-121B
									at 8' to 12'
10.0									
11.0									
12.0									12.0'
	4		4.0	2.0	Brn, Silt, Clay and F. Sand (interbedded), moist to wet				
13.0									
14.0									
15.0									
16.0									16.0'
					Boring terminated at 16'				
17.0					Monitoring well set at 14' BG. Well screen 4' to 14', sand pack				
					2.7' to 14', bentonite seal 1.2' to 2.7', grout 0' to 1.2', flush mount				
18.0					casing				

DELTA ENVIRONMENTAL CONSULTANTS

TEST BORING LOG

BORING NO.: MW-122

PROJECT: Geneva, NY									Sheet 1 of 1
CLIENT: HB Fuller									
DELTA PROJECT NO: V004210-1									
DRILLING METHOD: H.S.A.					SAMPLER	BIT SIZE	CORE	CASING	
DRILLING RIG: LM-1					Split-Spoon	NA	NA	NA	DATE: 7-13-05
DRILLER: Lyon Drilling									
DEPTH IN FT.	SAMPLE NO.	BLOWS PER 6"	REC. (ft.)	PID (ppm)	SOIL DESCRIPTION				REMARKS
	1	NA	3.1	0.0	Brn, F. Sand, dry				
1.0									
					1.4'				
2.0				1.0	Brn, Silt and Clay, dry				
									Soil sample MW-122A at 2' to 4'
3.0									
4.0									
	2		4.0	1.0	same as above				
5.0									
6.0									
7.0									
					7.7'				
8.0									
	3		4.0	4.0	Brn, F. Sand, wet				Soil sample MW-122B at 8' to 12'
9.0									
10.0									
11.0									
12.0									
	4		4.0	2.0	same as above				
13.0									
14.0									
15.0									
16.0									
					16.0'				
					Boring terminated at 16'				
17.0					Monitoring well set at 14' BG. Well screen 4' to 14', sand pack 2.7' to 14', bentonite seal 1.2' to 2.7', grout 0' to 1.2', flush mount casing				
18.0									

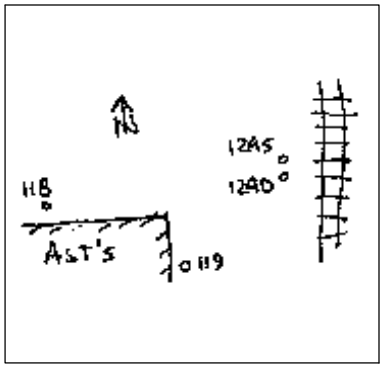
ECOR Solutions, Inc.
3191 Copper Oaks Place
Woodbury, MN
612.382.3763
kbrapp@comcast.net

Lithologic Log

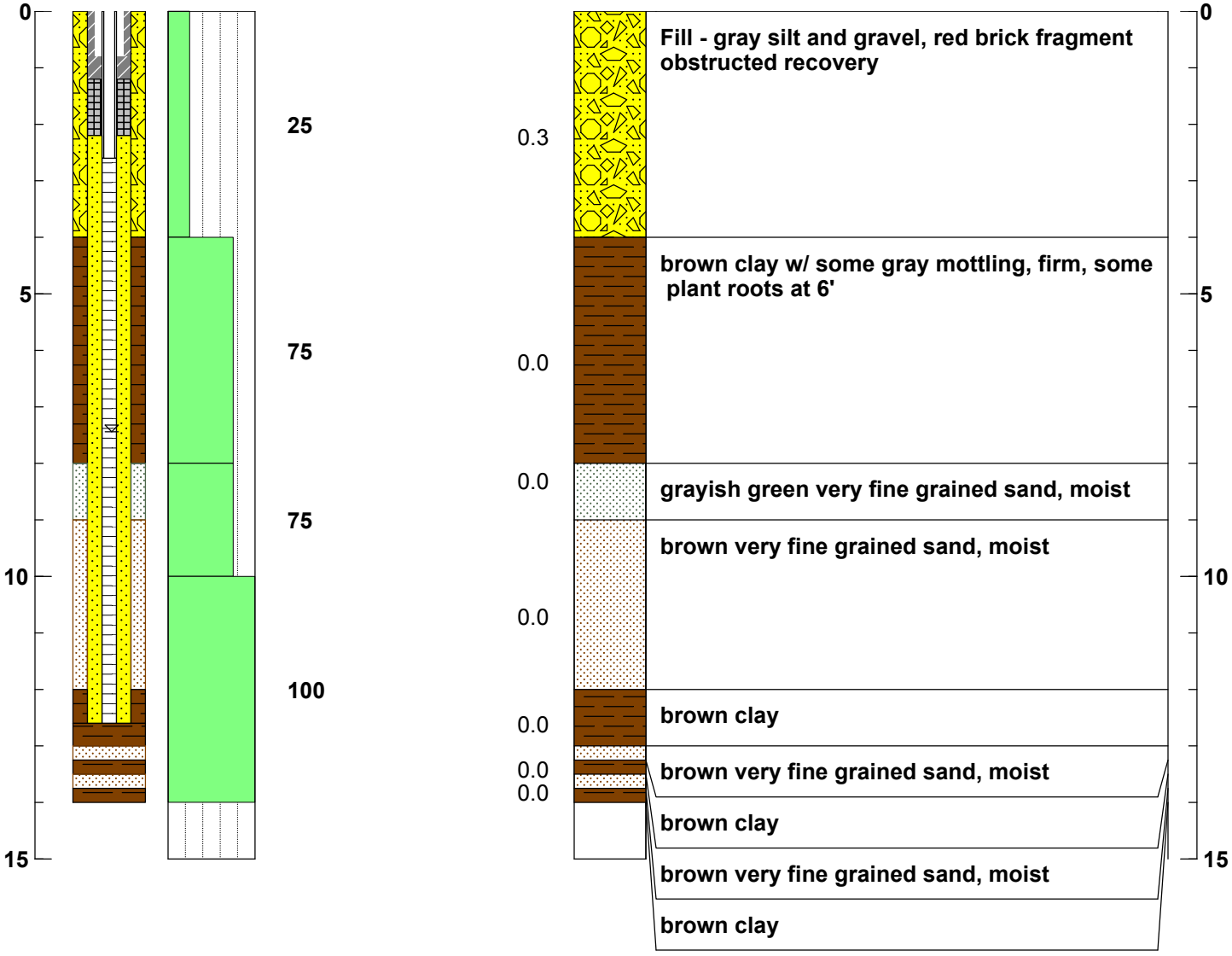
Boring No.: MW 124S

Well No.: MW 124S

location sketch



Project Name:	**Former Monarch Chem**	**Drilling Co:**	**Nothnagle**
Address:	**61 Gates Ave.**	**Driller:**	**Kevin Bush**
	Geneva, NY	**Method:**	**HSA**
Geologist:	**Bob Wojciak**	**Diameter:**	**6 1/4"**
Start Date:	**8/14/08**	**Total Depth:**	**14'**
End Date:	**8/15/08**		
Comment:	**used 4' geoprobe ahead of auger to sample**		



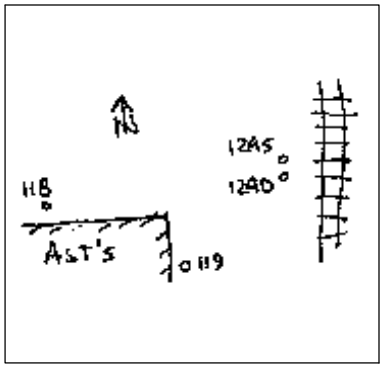
3191 Copper Oaks Place
Woodbury, MN
612.382.3763
kbrapp@comcast.net

Lithologic Log

Boring No.: MW 124D

Well No.: MW 124D

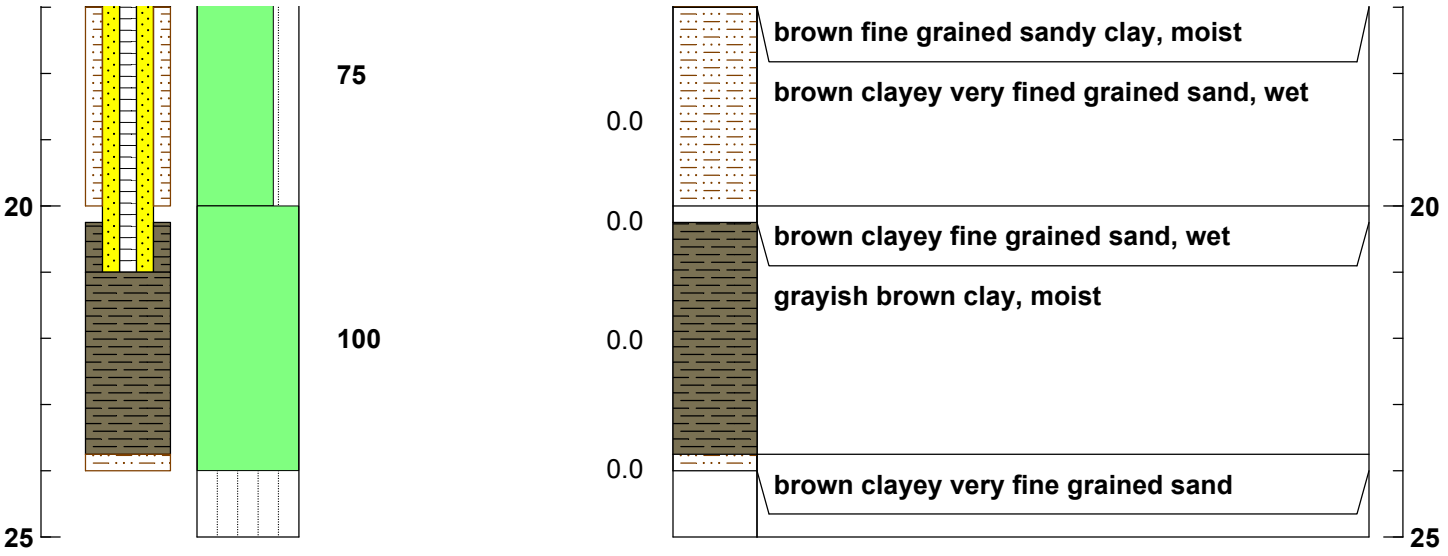
location sketch

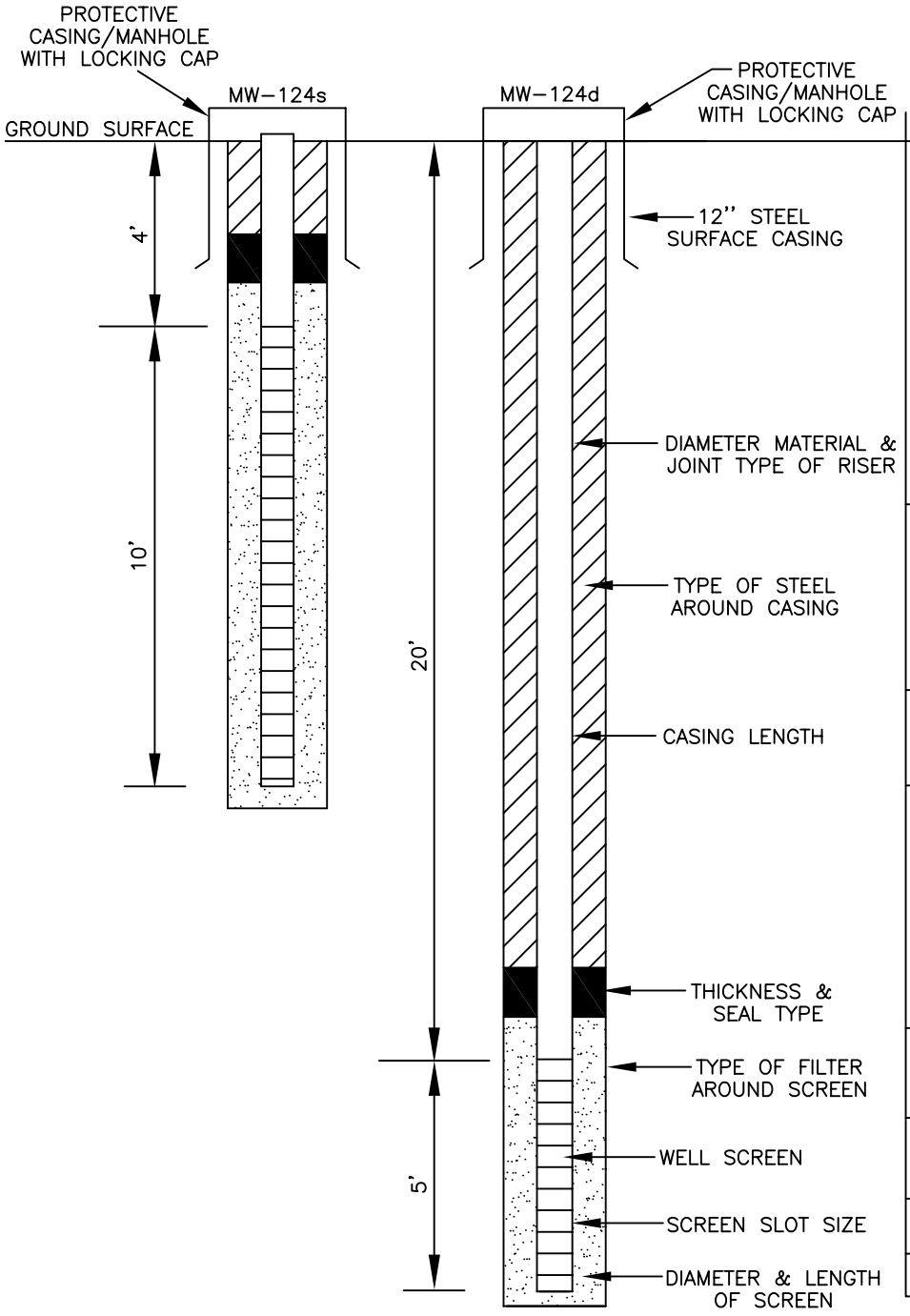


Project Name:	**Former Monarch Chem**	**Drilling Co:**	**Nothnagle**
Address:	**61 Gates Ave.**	**Driller:**	**Kevin Bush**
	Geneva, NY	**Method:**	**HSA**
Geologist:	**Bob Wojciak**	**Diameter:**	**6 1/4"**
Start Date:	**8/14/08**	**Total Depth:**	**24'**
End Date:	**8/15/08**		
Comment:	**24'**		

Depth	Sample Interval	Percent Recovery	PID	Description
0			0.3	Fill - gray silt and gravel, red brick fragment obstructed recovery
5	25	75	0.0	brown clay w/ some gray mottling, firm, some plant roots at 6'
			0.0	grayish green very fine grained sand, moist
10	75		0.0	brown very fine grained sand, moist
			0.0	brown clay
			0.0	grayish brown clay
			0.0	brown clay
	100		0.0	brown, very fine grained sand, moist
15			0.0	brown clay, very soft, plastic, moist
			0.0	brown sandy clay, soft, wet
			0.0	brown clayey fine grained sand

Depth	Sample Interval	Percent Recovery	PID	Description





	MW-124s	MW-124d
DIAMETER MATERIAL & JOINT TYPE OF RISER	2" PVC THREADED	2" PVC THREADED
TYPE OF STEEL AROUND CASING	BENTONITE GROUT	BENTONITE GROUT
CASING LENGTH	4'	20'
THICKNESS & SEAL TYPE	BENTONITE CHIPS	BENTONITE CHIPS
TYPE OF FILTER AROUND SCREEN	#2 SAND PACK	#2 SAND PACK
WELL SCREEN	STAINLESS	STAINLESS
SCREEN SLOT SIZE	#10	#10
DIAMETER & LENGTH OF SCREEN	2"-10'	2"-5'

NOT TO SCALE

ECOR Solutions, Inc.
1075 Andrew Drive, Suite I
West Chester, PA 19380

File: WELL CONSTRUCTION.dwg Date: 7/23/08

Figure 2
Proposed Groundwater Monitoring Well Construction Detail

Former Monarch Chemicals Facility
61 Gates Avenue
Geneva, NY

APPENDIX I
Site Management Forms

INSPECTION LOG - GENEVA, NY SITE

Date: _____

Name: _____

	Yes Ok	No Not Ok
Cap\Soil Cover		
Vegetation		
No Trash or Debris		
Gates Locked		
Corrective Action		
Drainage		
Surface water drainage		
Ponding		
Railroad ballast contained		
Parking lot condition		
Drainage ditch		
Corrective Action		
Monitoring Wells\Piezometers		
Located - cover		
Manhole (secure)		
Water accumulation\damage		
Corrective Action		
Safety Equipment		
Fire Extinguishers		
Eye Wash		
Air Monitoring Equipment		
Adequate Supply of Tyvek Suits, Rubber Gloves, Hard Hats, Eye Protection, ect.		
Communication System - Two Way Radios, Air Horns, Telephone		
Corrective Action		
Sample Collection\Storage		
Samples Intact		
Refrigerated		
Corrective Action		

Comments:

Summary of Green Remediation Metrics for Site Management

Site Name: _____ Site Code: _____
Address: _____ City: _____
State: _____ Zip Code: _____ County: _____

Initial Report Period (Start Date of period covered by the Initial Report submittal)
Start Date: _____

Current Reporting Period
Reporting Period From: _____ To: _____

Contact Information
Preparer's Name: _____ Phone No.: _____
Preparer's Affiliation: _____

I. Energy Usage: Quantify the amount of energy used directly on-site and the portion of that derived from renewable energy sources.

	Current Reporting Period	**Total to Date**
Fuel Type 1 (e.g. natural gas (cf))		
Fuel Type 2 (e.g. fuel oil, propane (gals))		
Electricity (kWh)		
Of that Electric usage, provide quantity:		
Derived from renewable sources (e.g. solar, wind)		
Other energy sources (e.g. geothermal, solar thermal (Btu))		

Provide a description of all energy usage reduction programs for the site in the space provided on Page 3.

II. Solid Waste Generation: Quantify the management of solid waste generated on-site.

	Current Reporting Period (tons)	**Total to Date (tons)**
Total waste generated on-site		
OM&M generated waste		
Of that total amount, provide quantity:		
Transported off-site to landfills		
Transported off-site to other disposal facilities		
Transported off-site for recycling/reuse		
Reused on-site		

Provide a description of any implemented waste reduction programs for the site in the space provided on Page 3.

III. Transportation/Shipping: Quantify the distances travelled for delivery of supplies and lab-supplied bottles, shipping of laboratory samples, and the removal of waste.

	Current Reporting Period (miles)	**Total to Date (miles)**
Standby Engineer/Contractor		
Laboratory Courier/Delivery Service (bottle and sample delivery)		
Waste Removal/Hauling		

Provide a description of all mileage reduction programs for the site in the space provided on Page 3. Include specifically any local vendor/services utilized that are within 50 miles of the site.

IV. Water Usage: Quantify the volume of water used on-site from various sources.

	Current Reporting Period (gallons)	**Total to Date (gallons)**
Total quantity of water used on-site (not including treated water)		
Of that total amount, provide quantity:		
Public potable water supply usage		
Surface water usage		
On-site groundwater usage		
Collected or diverted storm water usage		

Provide a description of any implemented water consumption reduction programs for the site in the space provided on Page 3.

V. Land Use and Ecosystems: Quantify the amount of land and/or ecosystems disturbed and the area of land and/or ecosystems restored to a pre-development condition (i.e. Green Infrastructure).

	Current Reporting Period (acres)	**Total to Date (acres)**
Land disturbed		
Land restored		

Provide a description of any implemented land restoration/green infrastructure programs for the site in the space provided on Page 3.

Description of green remediation programs reported above (Attach additional sheets if needed)
Energy Usage:
Waste Generation:
Transportation/Shipping:
Water usage:
Land Use and Ecosystems:
Recommendations/Other:

CONTRACTOR CERTIFICATION
I, _____ (**Name**) do hereby certify that I am _____ (**Title**) of _____ (**Contractor Name**), which is responsible for the work documented on this form. According to my knowledge and belief, all of the information provided in this form is accurate and the site management program complies with the DER-10, DER-31, and CP-49 policies. _____ **Date** **Contractor**

APPENDIX J
Carbon Footprint Analysis

Back to Intro | Back to Summary | Help

EPA CENTER FOR CORPORATE CLIMATE LEADERSHIP
U.S. Environmental Protection Agency

Scope 1 Emissions from Mobile Sources

Guidance

(A) Enter annual data for each vehicle or group of vehicles (grouped by vehicle type, vehicle year, and fuel type) in ORANGE cells in **Table 1**. Example entry is shown in first row (GREEN *Italics*). Only enter vehicles owned or leased by your organization on this sheet. All other vehicle use such as employee commuting or business travel is considered a scope 3 emissions source and should be reported in the corresponding scope 3 sheets.

- *Note: The latest mobile combustion factors reflect year 2021 data. Therefore, for all vehicle model years 2022 onward, the 2021 year factor is used.*
- *Select "On-Road" or "Non-Road" from the drop down box to determine the Vehicle Types available. You **must make this selection before picking the vehicle type.***
- Select "Vehicle Type" from drop down box (closest type available).
- Enter "Fuel Usage" in appropriate units (units appear when vehicle type is selected).
  - If mileage or fuel usage is unknown, estimate using approximate fuel economy values from the manufacturer, www.fueleconomy.gov, or the Reference Table below.
  - Vehicle year and Miles traveled are not necessary for non-road equiment.

(B) When using biofuels, typically the biofuel (biodiesel or ethanol) is mixed with a petroleum fuel (diesel or gasoline) for use in vehicles. Enter the biodiesel and ethanol percentages of the fuel if known, or leave default values shown below.

Biodiesel Percent:	20	%
Ethanol Percent:	80	%

(C) Biomass CO_2 emissions from biodiesel and ethanol are not reported in the total emissions, but are reported separately at the bottom of the sheet.

Table 1. Mobile Source Fuel Combustion and Miles Traveled

Source ID	Source Description	On-Road or Non-Road?	Vehicle Type	Vehicle Year	Fuel Usage	Units	Miles Traveled
Fleet-012	*HQ Fleet*	*OnRoad*	*Passenger Cars - Gasoline*	*2019*	*500*	*gal*	*12,400*
Annual inspection	round trip - car	OnRoad	Passenger Cars - Gasoline	2024	5	gal	90
Biannual groundwater sampling	round trip - light duty truck	OnRoad	Light-Duty Trucks - Gasoline	2024	5	gal	90

Reference Table: Average Fuel Economy by Vehicle Type

Vehicle Type	Average Fuel Economy (mpg)
Passenger Cars	24.8
Other 2-Axle, 4-Tire Vehicles	18.1
Motorcycles	44.0
Single unit 2-Axle 6-Tire or More Trucks	7.9
Combination Trucks	6.9
Diesel Buses (Diesel Heavy-Duty Vehicles)	7.4

Average mpg values from the U.S. Department of Transportation, Federal Highway Administration, Highway Statistics 2022 (Updated February 2024), Table VM-1.

GHG Emissions

Total Organization-Wide Mobile Source Fuel Usage and CO_2 Emissions (On-Road and Off-Road Vehicles)

Fuel Type	Fuel Usage	Units	CO_2 (kg)	
Motor Gasoline	10	gallons	88	
Diesel Fuel	0	gallons	0	
Residual Fuel Oil	0	gallons	0	
Aviation Gasoline	0	gallons	0	
Kerosene-Type Jet Fuel	0	gallons	0	
Liquefied Petroleum Gases (LPG)	0	gallons	0	
Ethanol	0	gallons	0	*Note: emissions here are only for the gasoline portion of the fuel, biogenic CO_2 emissions are reported below (note different formula)*
Biodiesel	0	gallons	0	*Note: emissions here are only for the diesel portion of the fuel, biogenic CO_2 emissions are reported below (note different formula)*
Liquefied Natural Gas (LNG)	0	gallons	0	
Compressed Natural Gas (CNG)	0	scf	0	

Total Organization-Wide On-Road Gasoline Mobile Source Mileage and CH_4/N_2O Emissions

Total CO_2 Equivalent Emissions (metric tons) - Mobile Sources	**0.0879**
Total Biomass CO_2 Equivalent Emissions (metric tons) - Mobile Sources	**0.0**

APPENDIX K
Request to Import/Reuse Fill Material Form

NEW YORK STATE
DEPARTMENT OF ENVIRONMENTAL CONSERVATION

Request to Import/Reuse Fill or Soil

This form is based on the information required by DER-10, Section 5.4(e) and 6NYCRR Part 360.13. Use of this form is not a substitute for reading the applicable regulations and Technical Guidance document.

SECTION 1 – SITE BACKGROUND

Site Name:

Site Number:

The allowable site use is: Choose an item

Have Ecological Resources been identified? Choose an item

Is this soil originating from the site? Choose an item

How many cubic yards of soil will be imported/reused? Choose an item

If greater than 1000 cubic yards will be imported, enter volume to be imported:

SECTION 2 – MATERIAL OTHER THAN SOIL

Is the material to be imported gravel, rock or stone? Choose an item

Does it contain less than 10%, by weight, material that passes a size 100 sieve? Choose an item

Is this virgin material from a permitted mine or quarry? Choose an item

Is this material recycled concrete or brick from a DEC registered processing facility? Choose an item

SECTION 3 - SAMPLING

Provide a brief description of the number and type of samples collected in the space below:

Example Text: 5 discrete samples were collected and analyzed for VOCs. 2 composite samples were collected and analyzed for SVOCs, Inorganics & PCBs/Pesticides.

If the material meets requirements of DER-10 section 5.4(e)5 (other material), no chemical testing needed.

SECTION 3 CONT'D - SAMPLING

Provide a brief written summary of the sampling results or attach evaluation tables (compare to DER-10, Appendix 5):

Example Text: Arsenic was detected up to 17 ppm in 1 (of 5) samples; the allowable level is 16 ppm.

If Ecological Resources have been identified use the "If Ecological Resources are Present" column in Appendix 5.

SECTION 4 – SOURCE OF FILL

Name of person providing fill and relationship to the source:

Name and address of fill source:

Location where fill was obtained:

Identification of any state or local approvals as a fill source:

If no approvals are available, provide a brief history of the use of the property that is the fill source:

Provide a list of supporting documentation included with this request:

The information provided on this form is accurate and complete.

_____ _____

Signature Date

_____

Print Name

_____

Firm