Niagara Falls Air Reserve Station, New York

Final

Unrestricted Use Characterization Quality Assurance Project Plan for Sites DS001, DS003, ST009, ST011, TU956, and TU962

August 2013

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13. SUPPLEMENTARY NOTES

14. ABSTRACT
This Final Quality Assurance Project Plan is for performance-based remediation performed on sites DS001, DS003, ST009, ST011, TU956, and TU962 at Niagara Falls Air Reserve Station.

15. SUBJECT TERMS
Uniform Federal Policy-Quality Assurance Project Plans, Standard Operating Procedures

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FINAL

Niagara Falls Air Reserve Station

Unrestricted Use Characterization Quality Assurance Project Plan for Sites DS001, DS003, ST009, ST011, TU956, and TU962

Contract No: FA8903-09-D-8588
Task Order 0006

Prepared for:
Air Force Civil Engineer Center
2261 Hughes Avenue, Ste 155
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Acronyms and Abbreviations

°C	Degree(s) Celsius
°F	Degree(s) Fahrenheit
µg/L	Microgram(s) per liter
%D	Percent difference
%R	Percent recovery
A	Analytical
AES	Atomic Emission Spectrometry
AFB	Air Force Base
AFCEC	Air Force Civil Engineer Center
AFRES	Air Force Reserves
ARS	Air Reserve Station
ASP	Analytical Services Protocol
AST	Aboveground Storage Tank
B.A.	Bachelor of Arts
BFB	Bromofluorobenzene
bgs	Below ground surface
B.S.	Bachelor of Science
BTEX	Benzene, toluene, ethylene, and total xylenes
BX	Base Exchange
CAS	Chemical Abstract Service
CCB	Continuing calibration blank
CCC	Continuing calibration criteria
CCV	Continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CIH	Certified Industrial Hygienist
CO	Contracting Officer
COC	Constituent of Concern
COR	Contracting Officer's Representative
CSP	Certified Safety Professional
CVAAS	Cold vapor atomic absorption
CVOC	Chlorinated volatile organic compound
DDT	Dichlorodiphenyltrichloroethane
DER	Division of Environmental Remediation
DFTPP	
DL	Detection limit
DoD	Department of Defense
DQI	Data quality indicator
DQO	Data quality objective
DUSR	Data usability summary report
EA	EA Engineering, Science, and Technology, Inc.
ECD	Electron capture detector
ELAP	Environmental Laboratory Analytical Program

EPA	U.S. Environmental Protection Agency
ERPIMS	Environmental Restoration Program Information Management System
FID	Flame ionization detector
FS	Feasibility study
ft	Foot (feet)
GC	Gas chromatograph
HCl	Hydrochloric acid
HDPE	High density polyethylene
HNO_3	Nitric acid
IC	Ion chromatography
ICAL	Initial calibration
ICB	Initial calibration blank
ICP	Inductively coupled plasma
ICS	Interfering element check standard
ICV	Initial calibration verification
ID	Identification
IRP	Installation Restoration Program
IS	Internal standard
L	Liter
LCD	Liquid crystal display
LCS	Laboratory control sample
LOD	Limit of detection
LOQ	Limit of quantitation
MDL	Method detection limit
mg/kg	Milligram(s) per kilogram
mg/L	Milligram(s) per Liter
mL	Milliliter
MOGAS	Motor vehicle gasoline
M.S.	Master of Science
MS	Matrix spike
MSD	Matrix spike duplicate
MTBE	Methyl tert-butyl ether
NA	Not applicable
NFA	No Further Action
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
OSHA	Occupational Safety and Health Administration
PAH	Polycyclic aromatic hydrocarbon
PAL	Project Action Limit
PBR	Performance-Based Remediation
PCB	Polychlorinated biphenyl

PE	Professional Engineer
PID	Photoionization Detector
PG	Professional Geologist
PMP	Project Management Professional
POC	Point of Contact
ppb	Parts per billion
ppm	Parts per million
QA	Quality assurance
QAPP	Quality Assurance Project Plan
QC	Quality control
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RI	Remedial investigation
RL	Reporting Limit
RPD	Relative percent difference
RPM	Remedial Project Manager
RRO	Residual range organic
RRT	Relative retention time
RSD	Relative standard deviation
RT	Retention time
S	Sampling
SC	Site Closeout
SCO	Soil Cleanup Objective
SIM	Selected ion monitoring
SOP	Standard operating procedure
STR	Senior Technical Review
SVE	Soil vapor extraction
SVOC	Semivolatile organic compound
TAL	Target analyte list
TCE	Trichloroethene
TCL	Target compound list
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TPH	Total petroleum hydrocarbon
UFP	Uniform Federal Policy
USAF	U.S. Air Force
USCS	Unified Soil Classification System
UST	Underground storage tank
UU	Unrestricted Use
VOA	Volatile organic analysis
VOC	Volatile organic compound

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EXECUTIVE SUMMARY

This Quality Assurance Project Plan (QAPP), which has been prepared for the United States Air Force (USAF) Civil Engineer Center (AFCEC), applies to the environmental investigations to be performed at six sites at the Niagara Falls Air Reserve Station (ARS), New York. This is a performance-based remediation project, which was awarded on 26 September 2012, by the AFCEC to Versar Inc. and teaming partner EA Engineering P.C. and its affiliate EA Science and Technology, as Task Order 0006 under Contract No. FA8903-09-D-8588.

The Statement of Objectives for this project includes obtaining Site Closeout (SC) for all six sites covered in this QAPP. SC signifies when the USAF has completed all active management and monitoring at an environmental cleanup site, no additional environmental cleanup funds will be expended at the site and the USAF has obtained regulatory concurrence. For practical purposes, SC occurs when cleanup goals have been achieved that allow unrestricted use of the site (i.e., no further management, including institutional controls, is required).

This QAPP has been prepared to encompass investigation activities at six sites at Niagara Falls ARS and will present project-specific information for the planned data collection activities at each site, including quality objectives; identification of project team members; and detailed field sampling techniques, sampling locations, number of samples to be collected, and proposed analytical methodology and performance criteria. The QAPP has been prepared to satisfy guidance provided in the New York State Department of Environmental Conservation (NYSDEC) Division of Environmental Remediation-10/Technical Guidance for Site Investigation and Remediation (NYSDEC 2010a).

This document meets the requirements and elements set forth in the Intergovernmental Data Quality Task Force Uniform Federal Policy (UFP) for QAPPs (U.S. Environmental Protection Agency [EPA] 2005). The UFP QAPP Manual integrates the EPA 7-Step data quality objectives process (EPA 2006), and the terminology in this QAPP is consistent with the UFP QAPP Manual (EPA 2005). The worksheets in this document follow the format of Revision 1 of the UFP QAPP Workbook (EPA 2012a).

This QAPP details activities to be conducted at the following sites:

- DS001–Site 14, Air Force Reserve (AFRES) Hazardous Waste Drum Storage
- DS003–Site 12, Bldg. 850 Drum Storage
- ST009–Site 4, Base Exchange Motor Vehicle Gas Leak
- ST011–Site 1, JP-4 Pipeline Leak
- TU956–Underground Storage Tank (UST) 304
- TU962–UST 600.

Purpose and Regulatory Framework

This QAPP provides instruction and guidance associated with the collection, analysis, and reporting of data to ensure that the data collected are scientifically valid, meet the established quality control objectives, are legally defensible, and support project objectives. Project objectives include characterizing soil from the six sites at Niagara Falls ARS in respect to NYSDEC Unrestricted Use (UU) soil cleanup objectives as set forth in 6 New York Code of Rules and Regulations (NYCRR) Subpart 375-6.3 and provided in the associated Table 375-6.8(a). The UU soil cleanup objectives in Table 375-6.8(a) represent the lowest of the three values for protection of groundwater, ecological resources, and public

health developed as set forth in Environmental Conservation Law 27-1415(6) and represent the concentration of a contaminant in soil which, when achieved at a site, will require no use restrictions on the site for the protection of public health, groundwater and ecological resources due to the presence of contaminants in the soil. Additionally, based on previous detections of contaminants in groundwater, two of the sites (ST009 and ST011) will have groundwater characterized and compared to groundwater standards provided in Table 1 of 6 NYCRR Part 703.5 for Class GA water (6 NYCRR Part 703.5 Water Quality Regulations, as presented in the Division of Water Technical and Operational Guidance Series 1.1.1, 1998, as amended). Class GA waters are those considered a source of potable water.

In the absence of institutional or administrative controls, SC involves demonstrating that affected site media meet the UU criteria. While the contaminants associated with the subject sites are primarily petroleum-related volatile organic compounds and semivolatile organic compounds, a subset of environmental samples need to be analyzed for target compound list organics (volatile organic compounds, semivolatile organic compounds, pesticides, and polychlorinated biphenyls) and target analyte list inorganic constituents.

Based on the findings of the UU characterizations, limited remedial action (e.g., excavation) may be necessary to achieve SC. Any activities associated with remedial actions at these sites, if required based on the results for the proposed site characterization, will be detailed in a separate QAPP.

All of the sites covered in this QAPP are regulated under two programs, Resource Conservation and Recovery Act (RCRA) and the New York State Spills Program. Both programs are administered through the NYSDEC and managed under two Air Force remediation programs: the Installation Restoration Program, and the Compliance Restoration Program, respectively. The six sites covered in this QAPP are currently listed as closed under the Spills Program (TU956 and TU962) or have received no further action/no further response action planned status under the RCRA program (DS001, DS003, ST009, and ST011) from the NYSDEC. No remediation or monitoring is occurring at any of the six sites (Table 1-1).

Installation Background

Niagara Falls ARS is located in Niagara Falls, New York, approximately 15 mi north of Buffalo (Figure 1-1). The installation, which adjoins the Niagara Falls International Airport, encompasses approximately 550 acres. The ARS is located 3.5 mi northeast of the Niagara River, in a lowland area which separates Lake Ontario (to the north) from Lake Erie (to the south). The installation is generally flat, with surface elevations ranging from 585 ft above mean sea level at the northern boundary of the installation to 600 ft above mean sea level at the southern boundary.

Niagara Falls ARS was created in November 1942 and operated by the Aerospace Defense Command, with numerous groups active on the installation between 1946 and 1971. In 1971, the installation was transferred to the AFRES command, and the 914th Tactical Airlift Group (later called the 914th Tactical Airlift Wing) became the installation host.

Document Structure

Table 1-2 lists the 37 UFP QAPP worksheets and provides a cross walk to NYSDEC DER-10 requirements. References used in the preparation of this QAPP are provided following the QAPP worksheets. Appendixes include the following:

- **Appendix A—**Spectrum Analytical, Inc. (Spectrum) Certifications and Quality Assurance Manual

- **Appendix B**—Standard Operating Procedures for Field and Laboratory

- **Appendix C—**Health and Safety Plan Addendum

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TABLE 1-1
List of Sites at Niagara Falls Air Reserve Station covered under the
Unrestricted Use Characterization Quality Assurance Project Plan for Niagara Falls Air Reserve Station, New York

Site ID	Site Name	Regulatory Program	Status	Date
DS001	Site 14, AFRES Hazardous Waste Drum Storage	RCRA	No Further Action	1991
DS003	Site 12, Bldg. 850 Drum Storage	RCRA	No Further Action	1992
ST009	Site 4, BX MOGAS Leak	RCRA	No Further Response Action Planned	1999
ST011	Site 1, JP-4 Pipeline Leak	RCRA	No Further Response Action Planned	1999
TU956	UST 304	Spills	Closed	1997
TU962	UST 600	Spills	Closed	1998

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TABLE 1-2
QAPP Worksheets – DER 10 Crosswalk
Unrestricted Use Characterization Quality Assurance Project Plan for Niagara Falls Air Reserve Station, New York

QAPP Worksheet No.	QAPP Worksheet Title	NYSDEC DER-10 Guidance
1 and 2	Title Page	Not Applicable
3 and 5	Project Organization and Quality Assurance Project Plan Distribution	Not Applicable
4,7, and 8	Personnel Qualifications and Sign-off Sheet	Chapter 1-Section 1.5
6	Communication Pathways	Not Applicable
9	Project Planning Session Summary	Not Applicable
10	Conceptual Site Model	Chapter 1 - Sections 1.8, 1.12, and 1.13
11	Project/Data Quality Objectives	Chapter 2, and Chapter 3 - Section 3.1
12	Measurement Performance Criteria	Chapter 2
13	Secondary Data Uses and Limitations	Chapter 1 – Sections 1.8 and 1.12
14 and 16	Project Tasks and Schedule	Not Applicable
15	Project Action Limits and Laboratory-Specific Detection/Quantitation Limits	Chapter 1 – Section 1.13
17	Sampling Design and Rationale	Chapter 3 – Sections 3.1 through 3.9
18	Sampling Locations and Methods	Chapter 3 – Sections 3.1 through 3.9
19 and 30	Sample Containers, Preservation, and Hold Times	Chapter 2 – Section 2.3
20	Field Quality Control Summary	Chapter 2 – Section 2.4
21	Field Standard Operating Procedures	Chapter 3 – Sections 3.4 through 3.9
22	Field Equipment Calibration, Maintenance, Testing, and Inspection	Chapter 3 – Sections 3.4 through 3.9
23	Analytical Standard Operating Procedures	Chapter 2 – Section 2.1
24	Analytical Instrument Calibration	Chapter 2 – Section 2.1
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection	Chapter 2 – Section 2.1
26 and 27	Sample Handling, Custody, and Disposal	Chapter 2
28	Analytical Quality Control and Corrective Action	Chapter 2
29	Project Documents and Records	Chapter 2 and Chapter 3
31,32, and 33	Assessments and Corrective Action	Chapter 2
34	Data Verification and Validation Input	Chapter 2 – Section 2.3
35	Data Verification Procedures	Chapter 2 – Section 2.3
36	Data Validation Procedures	Chapter 2 – Section 2.3
37	Data Usability Assessment	Chapter 2 – Section 2.3

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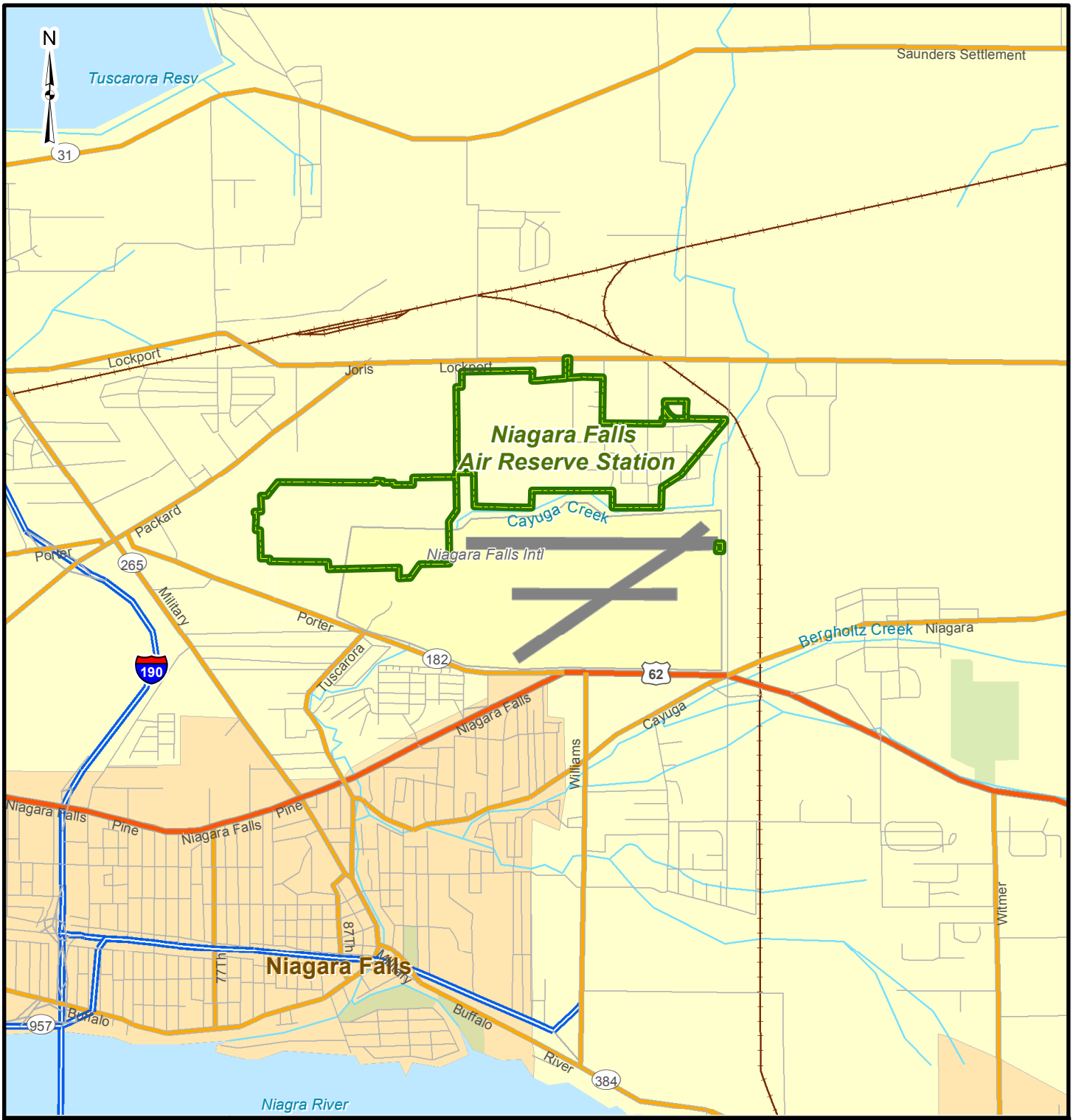

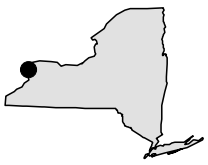

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Miles

EA®	Unrestricted Use Characterization Quality Assurance Project Plan Niagara Falls Air Reserve Station Niagara Falls, New York	FIGURE ES-1 Site Location Map

PROJECT MGR:	DESIGNED BY:	CREATED BY:	CHECKED BY:	SCALE:	DATE:	PROJECT NO:	FILE NO:
BY	FDJR	FDJR	BY	AS SHOWN	AUGUST 2013	6242107	G:\Projects\Federal\DOD\6265401

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2.0 References

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New York State Department of Environmental Conservation (NYSDEC). 1992. No Further Action at IRP Sites 6, 11, and 12. Administrative Record File Number 148, July 1992.

NYSDEC. 1996. Spill Number 9500124, Buildings 304, 306, and 308. Niagara Falls, Niagara County. 29 October.

NYSDEC. 1998. Spill Number 9711822, Building 600 UST. Niagara Falls, Niagara County. 1 October.

NYSDEC. 2010a. Program Policy DER-10. Technical Guidance for Site Investigation and Remediation. May.

NYSDEC. 2010b. Program Policy CP-51. Soil Cleanup Guidance. October.

QAPP WORKSHEETS #1 AND 2

Title Page

Site Name/Project Name: Niagara Falls Air Reserve Station (ARS), Performance-Based Remediation (PBR)

Site Location/Number: Niagara Falls ARS, New York

Contract/Work Assignment:

Document Title: Unrestricted Use (UU) Characterization Quality Assurance Project Plan (QAPP) for Sites DS001, DS003, ST009, ST011, TU956, and TU962

Lead Organization: Air Force Civil Engineering Center (AFCEC)

Preparation Date (Month/Year): August 2013

The QAPP is (select one): ___ Generic ___X_ Site-Specific

List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date
Not applicable (NA)	NA

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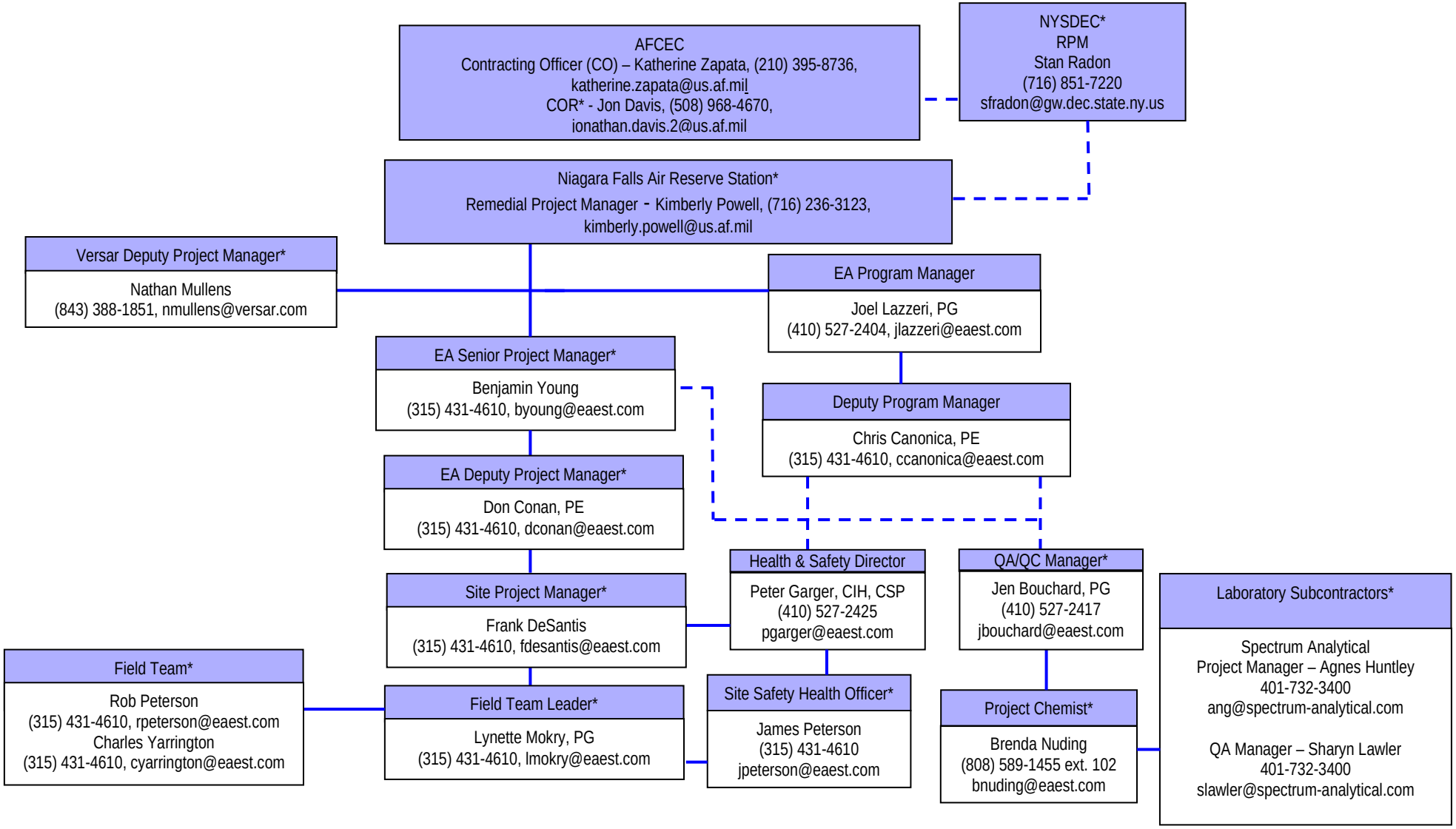
QAPP WORKSHEETS #3 AND 5

Project Organization and Quality Assurance Project Plan Distribution



*QAPP Recipient Lines of Authority Lines of Communication

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QAPP WORKSHEETS #4, 7, AND 8

Personnel Qualifications and Sign-Off Sheet

The qualifications of AFCEC and Niagara Falls ARS personnel are under the purview of the Department of Defense (DoD) and will not be outlined in this QAPP. In addition, state and Federal stakeholders' qualifications are under the purview of their respective agencies and will not be presented in this QAPP. The table below summarizes the responsibilities and provides a space for the signatures of key personnel to the sites covered in this QAPP. Signatures below indicate personnel have read and agree to implement this QAPP as written.

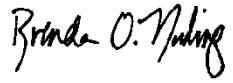
Organization: EA

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Joel Lazzeri	Program Manager	Bachelor of Science (B.S.) Geology, University of Delaware (1977) Master of Science (M.S.) Geology, University of New Orleans (1979) Mr. Lazzeri has 22 years of experience serving as a Program Manager and Project Manager. Mr. Lazzeri has managed underground storage tank (UST)/aboveground storage tank (AST), Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), and Resource Conservation and Recovery Act (RCRA) remedial projects involving a wide range of contaminants and diverse geological settings. He has successfully managed or provided quality assurance (QA)/senior technical review (STR) for environmental projects in more than 35 states and territories within all U.S. Environmental Protection Agency (EPA) regions.	• Professional Geologist • EA Project Managers Training • 8-Hour Health and Safety Supervisors Training • 40-Hour Hazardous Waste Operations Health and Safety Training	16 August 2013
Chris Canonica	Deputy Program Manager	B.S. Civil Engineering, Syracuse University (1989) Mr. Canonica is a Professional Engineer with over 22 years of experience performing project management, program management, and client management for commercial, industrial, and federal entities. His current responsibility include program management, senior review, and engineering staff management for all of EA's engineering work in New York State.	• Professional Engineer • EA Project Managers Training • 8-Hour Health and Safety Supervisors Training • 40-Hour Hazardous Waste Operations Health and Safety Training	16 August 2013

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Benjamin Young	Senior Project Manager	B.S. Civil Engineering, University of Texas-Arlington, (1983) Mr. Young is a Registered PE with over 28 years of experience in performing and managing multidiscipline environmental engineering and consulting work. He has extensive regulatory experience in RCRA; CERCLA; solid waste; and other regulatory programs. He has effectively managed a broad range of environmental projects including assessments, audits, investigation, design, implementation, and operation and maintenance for federal, state, and private clients. Mr. Young has effectively managed the projects maintaining control of budget, schedule, staffing, subcontractors, and deliverable quality.	• Registered Professional Engineer – GA (1989) • 8-Hour Health and Safety Supervisors Training • 40-Hour Hazardous Waste Operations Health and Safety Training	16 August 2013 

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Donald Conan	Deputy Project Manager	B.S. Civil & Environmental Engineering, Clarkson University (1987) Mr. Conan is a Registered PE with more than 23 years of extensive experience in environmental and civil engineering. As a Senior Engineer in EA's Syracuse, New York office, Mr. Conan is responsible for managing the engineering staff in the Syracuse office, and the technical oversight of environmental restoration projects under various federal and state programs. His broad experience includes conducting site assessments related to hazardous waste and petroleum contamination and the remediation activities associated with these projects.	• Professional Engineer—FL, ME, NY • Occupational Safety and Health Administration (OSHA) 30-Hour Construction Safety and Health; 2011 • U.S. Army Corps of Engineers; Construction Quality Management for Contractors; 2011 • OSHA 8-Hour Hazardous Waste Operations Supervisory; 1999 • OSHA 40-Hour Hazardous Waste Operations Safety Training; 1992 • OSHA 8-Hour Hazardous Waste Operations Refresher; Annual • University of Wisconsin, Designing Air-Based In Situ Soil and Groundwater Remediation Systems; 1995 • U.S. Navy Northern Division, Data Quality Objectives/Assessment Workshop; 1997 • American Public Works Association, Public Works Construction Inspection; 1990 • Ductile Iron Pipe Research Association, Basic Corrosion Short Course; 1989 • NYSDEC Soil Vapor Intrusion Training; 2005	16 August 2013 

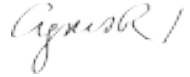
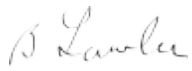
Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Jennifer Bouchard	QA/Quality Control (QC) Manager	M.S.; Utah State University; Geology; 2000 Bachelor of Arts (B.A.); Franklin and Marshall College; Geology; 1996 Ms. Martin Bouchard, Professional Geologist (PG), Project Management Professional (PMP), has 14 years of experience in the planning and management of hydrogeologic investigation and remediation projects. She has been responsible for reviewing audit results, project status, technical issues and quality of deliverables with various levels of management and the technical team.	• Registered Professional Geologist TN (No. 4971); 2003 • PMP 2009 • OSHA 40-Hour HAZWOPER Training & 8-Hour Refresher Training	16 August 2013 
Pete Garger	Health and Safety Director	Sc. M. Hygiene and Public Health, Johns Hopkins University (1981) B.A. Chemistry, Hofstra University (1978) Mr. Garger oversees health and safety activities for EA, including all health and safety activities in support of multiple Army, U.S. Air Force (USAF), Navy, and EPA contracts. Maintains EA's Corporate health and safety Program Manual, coordinates safety training activities, and develops and maintains medical surveillance programs. He has 30+ years of experience managing and conducting industrial hygiene services, including oversight of environmental remediation projects. Manages OSHA reporting requirements, helping EA maintain an OSHA recordable incident rate of 0.95 over the last 5 years.	• Certified Safety Professional (CSP) • Certified Industrial Hygienist (CIH) • OSHA Construction Outreach Trainer for 10- and 30-Hour Construction Safety Training • Department of Transportation Hazmat Training • U.S. Army Corps of Engineers – Unexploded Ordnance Training	16 August 2013 

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Brenda Nuding	Program Chemist	B.A. Chemistry, University of Oregon, (1987) Ms. Nuding is an environmental professional and chemist with more than 21 years of wide-ranging experience in the planning, technical support, management, and execution of multidisciplinary environmental consulting projects. Her technical experience includes over 11 years in an environmental analytical laboratory in various positions, including organic and inorganic quantitative analysis, methods development, training of analysts, QC, and management. Current responsibilities include QA oversight and technical support for chemical analysis for a variety of DoD projects. Her primary duties include preparing and reviewing technical reports; maintaining QA/QC; managing the collection of environmental samples; and acquiring, managing, validating, and assessing environmental data. Areas of specialization include preparation of statistically defensible sampling and analysis plans for remedial activities designed for regulatory review and the statistical evaluation of environmental data. Her extensive experience includes successful performance on large- and small-scale environmental programs for the USAF and other federal and private clients.	• AFCEC and the Environment ERPTools/X Training • Air Shipment of Hazardous Goods • U.S. Department of Transportation Hazardous Materials Regulations Training • U.S. Army Corps of Engineers Construction Quality Management for Contractors Course • Data Quality Objectives Training: Managing Uncertainty with Systematic Planning for Environmental Decision Making • EA Project Manager Training • 40-Hour Hazardous Waste Operations Health and Safety Training • 30-Hour Construction Safety Training	16 August 2013 

Organization: Versar

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Nathan Mullens	Senior Project Manager	B.S. Geology, Virginia Tech (2000) Mr. Mullens has 13 years of experience in a broad range of environmental and engineering projects including management of Geophysical Surveys, Time Critical Removal Actions, and other MEC restoration projects. Mr. Mullens also has experience managing soil, surface water, wastewater and groundwater related projects at commercial, industrial, and government facilities. He has experience in site assessments, environmental compliance assistance and remedial action implementation. He has managed RCRA sampling analysis reports and pollution prevention plans in addition to RCRA Part B Permits and Integrated Solid Waste Management Plans.	• Registered Environmental Manager #12322, National Registry of Environmental Professionals, United States • OSHA 40-Hour HAZWOPER Training & 8-Hour Refresher Training • RCRA Hazardous Waste Regulations Training • Uniform Federal Policy (UFP) QAPP Course	16 August 2013 

Organization: Spectrum Analytical Inc.

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Agnes Huntley	Laboratory Project Manager	B.S. Chemistry and Math, Simmons College. Ms. Huntley has over 16 years experience in Environmental Laboratory processes and management.		16 August 2013 
Sharyn Lawler	Laboratory QA Manager	B.S. Coastal Plant Ecology, UMASS Amherst Ms. Lawler has over 30 years of environmental laboratory experience.		16 August 2013 

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QAPP WORKSHEET #6

Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Modifications to Contract	AFCEC CO	Katherine Zapata	(210) 395-8736	Coordinate with COR to execute contract modifications.
Modifications to Program	AFCEC COR	Jon Davis	(508) 968-4670	Primary point-of-contact for AFCEC. Programmatic information, coordination issues, and draft and final reports. Coordinate with CO and Senior Project Manager.
Modifications to Contractor Program	Senior Project Manager	Ben Young	(315) 431-4610	Coordinate field work, programmatic direction, and issue resolution.
Modifications to Contractor Program	Deputy Project Manager	Don Conan	(315) 431-4610	Coordinate field work, programmatic direction, and issue resolution. Issue resolution with Senior Project Manager.
Significant Corrective Actions	Site Project Manager	Frank DeSantis	(315) 431-4610	Coordinate field work, programmatic direction and issue resolution. Notification of issues to Senior Project Manager, AFCEC, and Niagara Falls ARS.
	Niagara Falls ARS RPM	Kim Powell	(716) 236-3123	Coordination and resolution of issues between USAF/EPA/NYSDEC RPMs.
Modifications to Technical Direction or Modification of Approved Documents	Site Project Manager	Frank DeSantis	(315) 431-4610	The Site Project Manager will direct the Field Team Leader/Site Health and Safety Officer in the implementation of the field activities.
Modifications to QAPP Once in Execution	Field Team Leader	Lynette Mokry	(315) 431-4610	Notify the Site Project Manager of field-related problems by phone, email, or fax by close of business the next business day.
Project QA/QC Responsibilities	Project QA/QC Manager	Jennifer Bouchard	(315) 431-4610	Report on the adequacy, status, and effectiveness of the QA program by phone or email during the regular project review meetings and as needed.
Analytical Corrective Actions	Project Chemist	Brenda Nuding	(808) 589-1455, Ext. 102	Report on the adequacy, status, and effectiveness of the QA program to Site Project Manager, Senior Project Manager, and QA/QC Manager.
Approval of Amendments to QAPP	Site Project Manager	Frank DeSantis	(315) 431-4610	Notify Field Team Leader, Site Health and Safety Officer, and Project Chemist of QAPP amendments by phone, email, or fax by close of business the next business day.
Modifications to analytical laboratory responsibilities	Project Chemist	Brenda Nuding	(808) 589-1455	Notify Site Project Manager within 1 week of performance problems encountered by the contracted analytical laboratories by phone, email, or fax.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Modifications to analytical responsibilities	Laboratory Project Manager – Spectrum Analytical, Inc.	Agnes Huntley	(401) 732-3400	Report project nonconformance issues within 1 week to the Project Chemist by phone, email, or fax.
Modifications to analytical QA responsibilities	Laboratory QA Manager – Spectrum Analytical, Inc.	Sharyn Lawler	(401) 732-3400	Report project nonconformance issues within 1 week to the Laboratory Project Manager in person or by phone, email, or fax.

QAPP WORKSHEET #9

Project Planning Session Summary

Site Name/Project Name:	Multiple Sites/Niagara Falls ARS
Site Location:	Base-wide
Projected Date(s) of Sampling:	September 2013
Site Specific Project Manager:	Ben Young
Date of Session:	17 April 2013
Scoping Session Purpose:	General introduction of PBR contract to NYSDEC

Name	Organization	Project Role	Phone	E-mail Address
Kim Powell	914th MSG/CEV	Niagara Falls ARS RPM	716-236-3123	kimberly.powell@us.af.mil
Andy Salisbury	914th MSG/CEV	Niagara Falls ARS	716-236-3125	andrew.salisbury.1@us.af.mil
Ellen Marien	914th MSG/CEV	Niagara Falls ARS, Chief Environmental and Planning	716-236-3126	ellen.marien@us.af.mil
Ben Young	EA	EA Project Manager	315-430-4610	byoung@eaest.com
Frank DeSantis	EA	EA Site Manager	315-430-4610	fdesantis@eaest.com
Don Conan, PE	EA	EA Deputy Project Manager	315-430-4610	dconan@eaest.com
Jaime Hojdila, PG, PMP	Versar	Versar Deputy Project Manager	480-838-5352	jhojdila@versar.com
Dennis Weiss	NYSDEC – Region 9	Regulator	716-236-3135	drweiss@gw.dec.state.ny.us
Stan Radon	NYSDEC – Region 9	Regulator	716-851-7220	sfradon@gw.dec.state.ny.us

Comments:

Conference call was held with NYSDEC to discuss overall PBR contract and general discussion of sites. NYSDEC wanted to confirm that activities described in QAPP will be based on guidance from NYSDEC Division of Environmental Remediation (DER)-10 (NYSDEC 2010a). A consensus was reached that due to the number of sites, in an effort to streamline review of documents, sites would be grouped as appropriate in associated QAPPs.

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QAPP WORKSHEET #10

Conceptual Site Model

This worksheet describes the conceptual site model as applicable to the objectives of this QAPP for the following sites:

- DS001–Site 14, Air Force Reserve (AFRES) Hazardous Waste Drum Storage
- DS003–Site 12, Bldg. 850 Drum Storage
- ST009–Site 4, Base Exchange (BX) Motor Vehicle Gas (MOGAS) Leak
- ST011–Site 1, JP-4 Pipeline Leak
- TU956–UST 304
- TU962–UST 600.

Environmental Setting

The Niagara Falls ARS is underlain by 3–18 ft of unconsolidated deposits (overburden), which are in turn underlain by sedimentary bedrock (late-Silurian Period Lockport Dolostone). The stratigraphy at the Niagara Falls ARS generally consists of (from the ground surface down):

- 1–3 ft of reworked soil/fill
- 5–10 ft of lacustrine silt and clay
- 5–10 ft of till
- Lockport Dolostone.

At the six sites of concern, the thickness of overburden ranges from approximately 10 to 12 ft, with the water table located at approximately 4–7 ft below ground surface (bgs).

Groundwater exists within the overburden and the upper portion of the Lockport Dolostone, which is generally closely fractured in the upper 10–15 ft. No confining unit is present between the overburden and shallow bedrock; therefore, these units appear to have a direct hydraulic connection. Due to the low-permeability overburden material, the groundwater velocity is relatively slow. Most groundwater flow within the Lockport Dolostone is within secondary porosity features (i.e., along horizontal bedding planes, vertical fractures, and joints), particularly in the upper 5–15 ft where these openings may be weathered and/or expanded by dissolution. Shallow groundwater flows toward nearby streams and drainages, which may be to the east, southeast, south, or southwest. Groundwater movement may also be locally influenced by anthropogenic features, such as pumping wells, sumps, building foundations, sewer lines, etc.

Surface water primarily flows through a system of ditches and underground storm drains that discharge into Cayuga Creek. A drainage ditch is present along the eastern side of DS003. Cayuga Creek, which is the only major surface water body on the installation, enters at the northeastern boundary and flows along the eastern and southern boundaries before exiting the installation and discharging to Little River, a tributary to Niagara River.

Potential ecological habitat areas at the subject sites consist primarily of open fields around office buildings, hangars, and runways. These developed areas, characterized by buildings, mowed lawns, and impervious roads and parking lots, provide limited to low quality habitat. This habitat is not considered of ecological importance, because it is suitable habitat for only a few individuals of common wildlife

species that are habituated to humans. Cayuga Creek provides some higher quality habitat, with riparian vegetation growing along the banks.

Fate and Transport

A number of fate and transport pathways govern the transfer of elevated concentrations of chemicals between different environmental media and different portions of the Niagara Falls ARS sites (Figures 10-1 through 10-3). Chemicals released to soil at these sites may be transported by erosion and re-deposited in other portions of the sites. Constituents of concern (COC) have the potential to adsorb onto soil particles. Volatile chemicals can enter the vapor phase via partitioning from water or volatilization from particles with adsorbed contaminants present. Bioaccumulation, where an organism accumulates toxic chemicals from the environment in food, water, and the incidental ingestion of soil, may also be a relevant transport pathway. Dependent upon the chemical and the organism, these chemicals may accumulate in tissue.

Project Background

The location and surrounding features for each site are shown on Figure 10-4. A summary of the project background for each site is presented in the following paragraphs.

DS001 (Site 14)

DS001 is a 0.07-acre site located off of Otis Drive consisting of a fenced, unroofed, unbermed, asphalt storage pad which was used beginning in 1979 as a storage area for hazardous waste drums (Figure 10-5). No spills have been documented at DS001 and no corrective actions have occurred at the site.

Sampling conducted in 1989 included collection of wipe samples from the asphalt pad, composited surface soil samples from areas adjacent to the pad, and a composite sample from surface soil collected from cracks in the asphalt. The wipe and soil samples were analyzed for volatile organic compounds (VOCs), petroleum hydrocarbons, and sulfates. All wipe samples contained sulfates and No. 6 Fuel Oil (a component of asphalt), which was not considered a RCRA waste. No additional sampling was completed at the site and based on results of the sampling, no further action (NFA) status was recommended in 1990; NYSDEC concurred according to the Final Sampling/Monitoring Report (E&E 1996).

Site soils, both shallow subsurface (0–2 ft. interval) and subsurface soil from the groundwater/soil interface, require characterization to verify that current conditions meet UU criteria. The potential COCs at DS001 are VOCs and semivolatile organic compounds (SVOCs), based on past use as a drum storage area.

DS003 (Site 12)

DS003 is a 0.28-acre open grassy area west of Building 850 that was used during the 1950s and early-1960s as an accumulation point for drums of waste oil and hazardous waste from a nearby hangar (Figure 10-6). No spills have been documented at DS003 and no corrective actions have occurred at the site.

Phase I and II site assessments were completed in the 1980s. Six soil samples and one duplicate sample were collected from two locations (three different intervals at two borings extended to a total depth of 9 and 12 ft bgs) were analyzed for metals (EPA Method SW3050/6010), VOCs (EPA Method SW8240), and SVOCs (EPA Method SW3550/8270). Soil analytical results indicated the presence of oil and

grease, and total organic halogens. Trichlorethene (TCE) was detected in one soil sample (4.5–6.0 ft bgs) at an estimated concentration (0.55 mg/kg) which exceeds the current NYSDEC UU Soil Cleanup Objectives (SCOs) of 0.47 mg/kg.. Acetone was detected in four of the six soil boring samples greater than the UU SCO (0.05 mg/kg); however, similar detections were reported in two of three method blanks and attributed to laboratory contamination. Based on results of the sampling and the results of a human health baseline risk assessment, NFA status was recommended in 1992; and, NYSDEC, EPA, and the Air Force concurred according to the NYSDEC's NFA Memorandum (NYSDEC 1992).

Soil Boring	Boring Location	Sample Depth	Acetone	TCE
B-12-1-5	12SB-1	4.5–6	**0.084**	ND
B-12-1-7		9–10.5	**0.14**	ND
B-12-1-8		10.5–12	ND	ND
B-12-2-1	12SB-2	0–1.5	0.03	ND
B-12-2-4		4.5–6	0.032	**0.55**
B-12-2-6		7.5–9	**0.085**	0.046

Note: ND = Non-detect
All concentrations are in mg/kg.
Values in **bold** exceed soil cleanup objectives

Site soils, both shallow subsurface (0–2 ft. interval) and subsurface soils at the soil/groundwater interface, require additional characterization to verify that current conditions meet UU criteria. The potential COCs at DS003 are VOCs and SVOCs, based on past use as a drum storage area and previous sampling results.

ST009 (Site 4)

ST009 is a 0.35-acre open field located on the corner of Kinross and Olmstead Streets (Figure 10-7). Depth to groundwater averages 7 ft bgs and generally flows to the south/southwest. The average thickness of the overburden at ST009 is 12 ft. In 1982, a pipe leading to a MOGAS UST ruptured at the former BX Gas Station (Building 405). The tank was in direct contact with the overburden groundwater and groundwater entered the tank, displacing the fuel into the surrounding soil. Phase I/II and remedial investigation (RI)/feasibility study (FS) investigations were performed from 1983 through 1994, which included the installation and sampling of four monitoring wells (two upgradient [MW4-1 and MW4-2] and two downgradient [MW4-3 and MW4-4]) in 1984. Benzene (23 µg/L) was detected in groundwater above NYSDEC Class GA standards (1 µg/L) at MW4-3. Total petroleum hydrocarbons (TPH) and petroleum-related VOCs (specifically benzene, ethylbenzene, and xylenes) were detected in soil samples collected during the investigations.

The USTs and associated piping were removed in 1990. In 1992, a groundwater and soil vapor extraction (SVE) system, consisting of two groundwater extraction wells and two SVE wells, was installed and operated over a 6 month period. After the six month remediation period, only benzene (12 µg/L) was detected in groundwater above NYSDEC Class GA standards (1 µg/L).

Post-remedial soil samples were composited from two depth intervals (4–6 and 10 ft bgs and 6-8 ft bgs and 8–10 ft bgs) at two borings. These two soil samples were submitted for analysis by TCLP for VOCs and PAHs. Total BTEX concentrations were detected in each sample, but were one to two orders of magnitude less than pre-excavation concentrations. In March 1993, the NYSDEC acknowledged that additional remedial action was not required and listed the site as inactive. However, semiannual groundwater sampling was required for at least 2 years because of the residual contaminant concentrations in subsurface soil. Concentrations of BTEX in groundwater were reported in samples from MW4-3in 1996 and 1997.

Seven groundwater sampling events were conducted at monitoring well MW4-3. Total BTEX concentrations decreased to non-detect in November 1996, but increased to 170 µg/L during the following sampling event. Subsequent sampling through March 1998 indicated a decrease in BTEX concentrations (to approximately 40 µg/L) and only benzene (8.3 µg/L) was reported at a concentration greater than the NYSDEC Class GA standard (1 µg/L) during the last sampling even in September 1998. Additionally, by 1999, no evidence of migration had been observed, as noted by the lack of petroleum-related COCs detected in the furthest downgradient well (MW4-4). Based on the lack of migration and the decline in contaminant concentrations at MW4-3, NFA was approved by the NYSDEC for ST009 in 2001. Subsequently, monitoring wells MW4-3 and MW4-4 were abandoned in 2002. In order to verify that the site meets UU criteria, subsurface soils and groundwater at ST009 require additional characterization. The potential COCs at ST009 are associated with the tank rupture and include petroleum-related VOCs (specifically BTEX constituents and methyl tert-butyl ether [MTBE]) and SVOCs (naphthalene).

ST011 (Site 1)

ST011 encompasses 0.09-acres between McGuire Street and Building 600 (Figure 10-8). Depth to groundwater is approximately 5 ft bgs with flow generally to the southwest. Bedrock at the site is approximately 10 ft bgs. In 1969, an undetermined volume of JP-4 (jet fuel) leaked from a hydrant system, saturating soil. The leaking pipeline section was immediately drained, capped at both ends, and abandoned in place. No record of associated soil excavation exists. In 1986, the area was capped with asphalt and is currently used as a roadway and parking area. An additional potential source of petroleum-related contaminants at ST011 includes a former diesel fuel service area (and associated UST) located to the south according to the Limited RI/FS Report (E&E 1994). However, no documentation of a contaminant release was found in the administrative record for the fuel service area.

As part of a 1991 Installation Restoration Program (IRP) RI/FS, two subsurface soil samples were collected from a single soil boring near the southwest side of Building 600. TPH concentrations were 18,000 mg/kg and 1,100 mg/kg in the two soil samples. Additional soil samples were collected as part of the 1994 Limited RI/FS (E&E) from locations near the 1991 RI/FS sampling location and immediately south of a former fuel service area. TPH was detected in those soil samples at concentrations of 150 mg/kg and 440 mg/kg, respectively.

Groundwater monitoring in 10 of 11 overburden and bedrock wells installed at the site showed that there were no contaminants present above NYSDEC Class GA standards and guidance values. Therefore, NYSDEC concurred with Niagara Falls ARS recommendation that NFA was required for the JP-4 Pipeline Leak according to the No Further Response Action Planned Decision Document (E&E 1999). With the exception of MW1-3DA (one of the two bedrock monitoring wells at the site), the remaining on-site monitoring wells were abandoned by 1997. MW1-3DA was retained to monitor detections of chlorinated volatile organic compounds (CVOCs), which were not attributed to the JP-4 leak. MW1-3DA was last sampled 2008 and only *cis*-1,2-DCA remained at a concentration greater than the standard (7.1 µg/L) in groundwater. Based on the decreasing trend in CVOC concentrations, MW1-3DA was removed from the base-wide groundwater monitoring program and from the active monitoring requirements of the RCRA Permit in 2011.

COPC Detected in GW at SB01-504-GW	Result (µg/L)	NYSDEC Class GA Standards (µg/L)
ST011 (Site 1)		
1,2,4-Trimethylbenzene	29	**5**
1,3,5-Trimethylbenzene	5.2	**5**
Ethylbenzene	12	5
m- and p-Xylene	6.1	5
Naphthalene	26	5
n-Propylbenzene	7.9	**5**

In 2009, a petroleum odor was encountered when soil was excavated during installation of a water line west of the Building 600 loading dock. An additional site investigation was conducted, which included installation of 15 soil borings (SB01-501 through 514) and collection of 6 groundwater samples. Petroleum-related COCs (including ethylbenzene, xylenes, and naphthalene) were detected at concentrations greater than NYSDEC Class GA groundwater standards in one *in situ* groundwater sample from SB01-504 (Figure 10-8). No additional monitoring wells were installed. None of the compounds that were detected in the associated soil samples exceeded the UU criteria.

No corrective actions have occurred at ST011. Subsurface soils and groundwater require additional characterization to determine if the site meets UU criteria. The potential COCs at ST011 include petroleum-related VOCs (specifically BTEX constituents) and SVOCs (polycyclic aromatic hydrocarbons [PAHs]).

TU956 (UST 304)

TU956 is a 0.004-acre grassy area located south of Building 304 (Figure 10-9). A 550-gal steel No. 2 fuel oil UST installed in 1953 was removed in 1995 from TU-956. During tank removal, soil samples were collected per NYSDEC requirements: one composite samplethe bottom, and one from the piping areas. Soil sample results from tank removal activities were compared to the NYSDEC soil cleanup objectives in effect at that time. However, results reported in parts per billion (ppb) were erroneously compared to NYSDEC guidance values reported in parts per million (ppm). Because this indicated that some contaminant concentrations exceeded the criteria, NYSDEC requested that additional soil sampling be performed. In September 1996, one composite sample was collected from three equidistant locations from approximately 4.3 to 6.5 ft bgs within the footprint of the former UST. The sample was submitted for TCLP VOC (EPA Method SW8021) and SVOC base/neutral compound (EPA Method SW8270) analyses. Total xylenes were detected at a concentration (10 µg/L) greater than the TCLP-based Spill Technology and Remediation Series (STARS #1) August 1992 criteria (individual xylene isomers <5 µg/L each). NYSDEC classified Spill No. 9500124 as “inactive” on 29 October 1996, due to the relatively low concentrations observed. NYSDEC closed the spill on 6 June 1997.

Based on a comparison of the original soil data collected during the tank removal in 1995 to the current NYSDEC UU SCOs (NYSDEC 2010a), the site soil meets UU criteria. Although the original soil results do not exceed either past or current criteria, and TCLP analysis of soil samples is no longer included in the NYSDEC guidance for fuel oil sites, additional soil sampling (including the soil/groundwater interface) is required to show that impacts to soil did not remain following tank closure. Subsurface soils at TU956 require further evaluation to confirm that the site meets UU criteria and is protective of groundwater. The potential COCs for the site are petroleum-related compounds (specifically BTEX and PAHs).

TU962 (UST 600)

The TU962 site encompasses 0.002-acres located north of Building 600 (Figure 10-10). A 280-gal steel fuel oil UST was installed in 1957 and removed in 1997 from TU962. NYSDEC Spill No. 9711822 was closed on 30 September 1998.

During the tank removal in 1997, the extent of the excavation was 7 ft × 10 ft × 6 ft bgs. Visibly impacted soil was removed. The UST had been secured to a 10–12-in. thick concrete pad that was keyed into bedrock. This limited the vertical extent of the excavation. Composite soil samples were collected from the excavation bottom and sidewalls following the tank removal and analyzed for TCLP VOCs (EPA Method SW8270). The excavation was backfilled with clean soil and compacted crushed stone in 12 in. lifts.

Several VOCs (n-Butylbenzene, naphthalene, 1,2,4-trimethylbenzene, 1,3,5-trimethylbenzene, and total xylenes) were detected at concentrations exceeding their respective NYSDEC STARS guidance value. However, NYSDEC did not require further action following the tank removal because the detected concentrations were considered low; therefore, the site was classified as "inactive" (NYSDEC 1998). In addition to classification as inactive, the NYSDEC closure letter stipulated that "*…any soil removed from this area in the future must either be sampled and analyzed, to prove it is not contaminated, or [properly] disposed*."

Additional soil sampling, including from the soil/groundwater interface, is required to show that impacts to soil do not remain following the UST removal. Subsurface soils at TU962 require further evaluation to confirm the site meets UU criteria and is protective of groundwater. The potential COCs for the site are petroleum-related compounds (specifically BTEX and PAHs).

The Problem to be Addressed by the Project

The overall objective of the additional investigation efforts is to document that current site conditions meet UU criteria, which include NYSDEC Part 375 UU SCOs and NYSDEC Class GA groundwater standards and guidance values (6 NYCRR Part 703.5 Water Quality Regulations, as presented in the Division of Water Technical and Operational Guidance Series 1.1.1, 1998, as amended). Demonstrating that potentially affected site media meet the UU criteria for a full suite of analytes will allow for site closeout (SC), without reliance on institutional or administrative controls. While the COCs associated with the subject sites are primarily petroleum-related VOCs and SVOCs (specifically BTEX constituents and PAHs), to show the site meets UU criteria a subset of environmental samples need to be analyzed for target compound list (TCL) organics (VOCs, SVOCs, PCBs, pesticides, and herbicides) and target analyte list (TAL) inorganic constituents (NYSDEC 2010a). Additional analytical data are, therefore, required to determine the following:

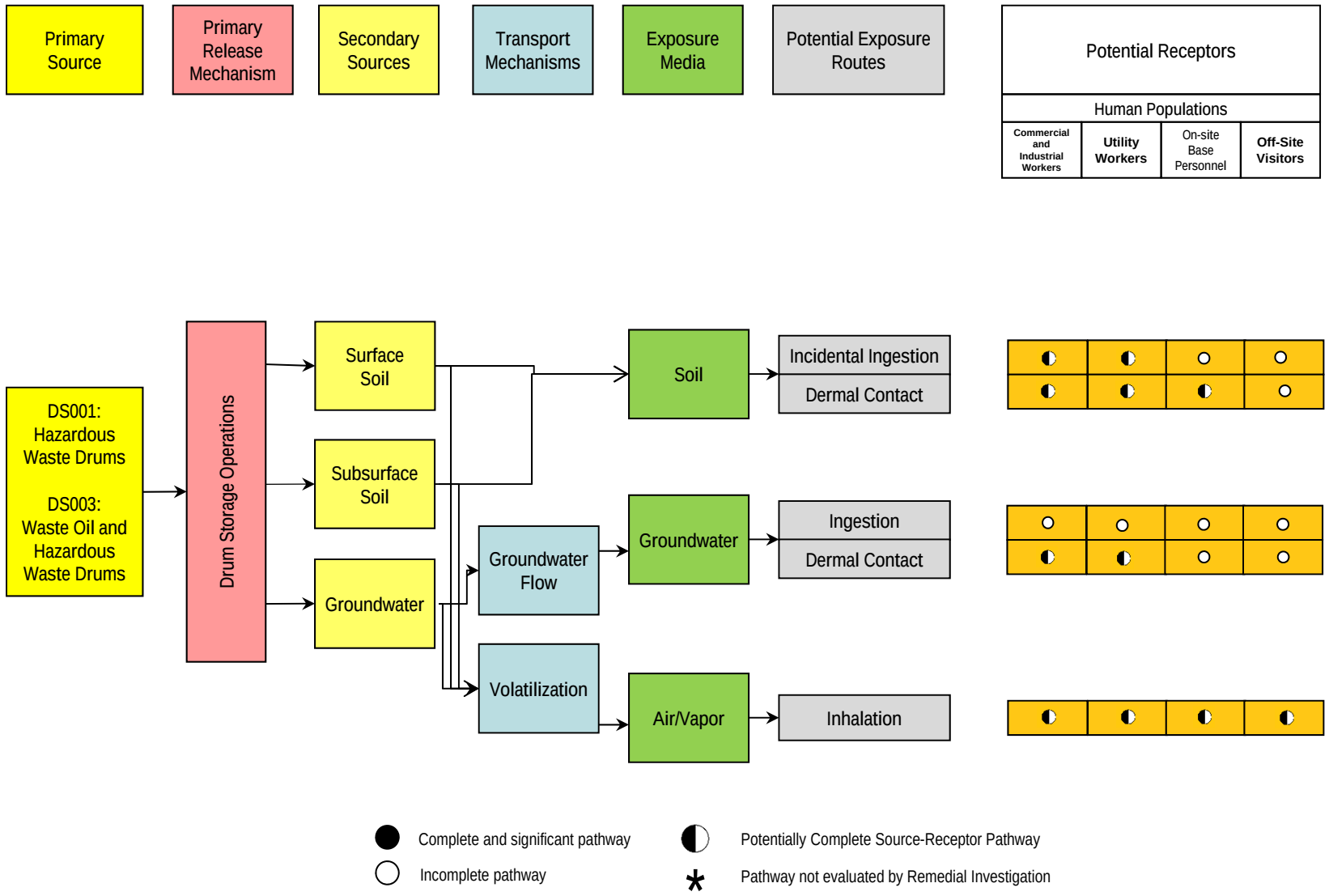
1. Determine whether current concentrations of COCs in potentially affected site media meet UU criteria.

2. Delineate the extent of UU criteria exceedances, if any.

3. Provide sufficient data to develop a remedial plan, if necessary, to meet UU criteria for site COCs in affected media.

4. Evaluate potential for impacts to soil at sites DS001, DS003, ST009, ST011, TU956, and TU962.

5. Evaluate potential groundwater impacts at sites ST009 and ST011.



EA®	Unrestricted Use Characterization Quality Assurance Project Plan Niagara Falls Air Reserve Station Niagara Falls, New York	FIGURE 10-1 DS001 and DS003 Conceptual Site Model		
		PROJECT MGR: BY	DATE: AUGUST 2013	EA PROJECT NO: 6265401

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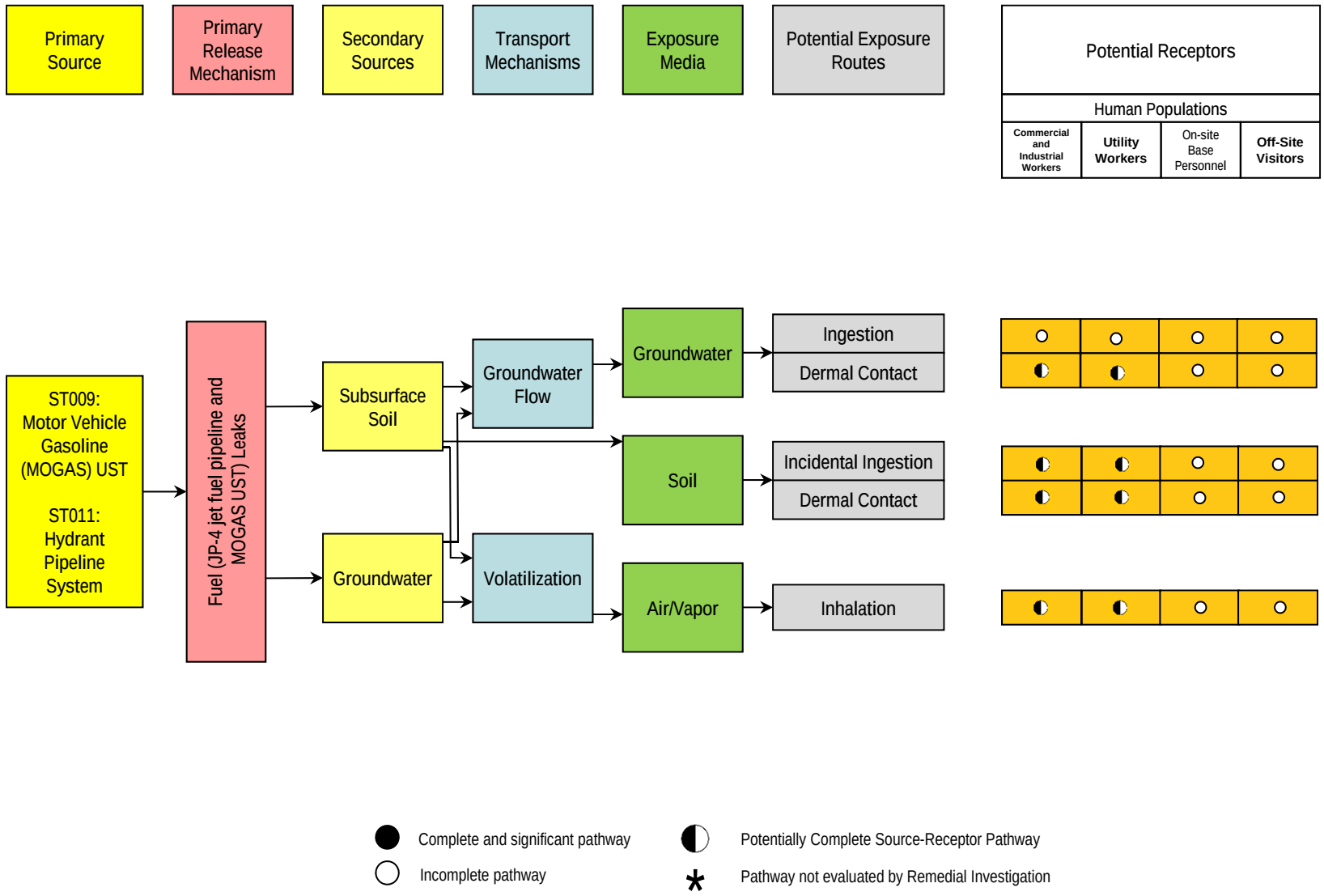

EA®	Unrestricted Use Characterization Quality Assurance Project Plan Niagara Falls Air Reserve Station Niagara Falls, New York	FIGURE 10-2 ST009 and ST011 Conceptual Site Model		
		PROJECT MGR: BY	DATE: AUGUST 2013	EA PROJECT NO: 6265401

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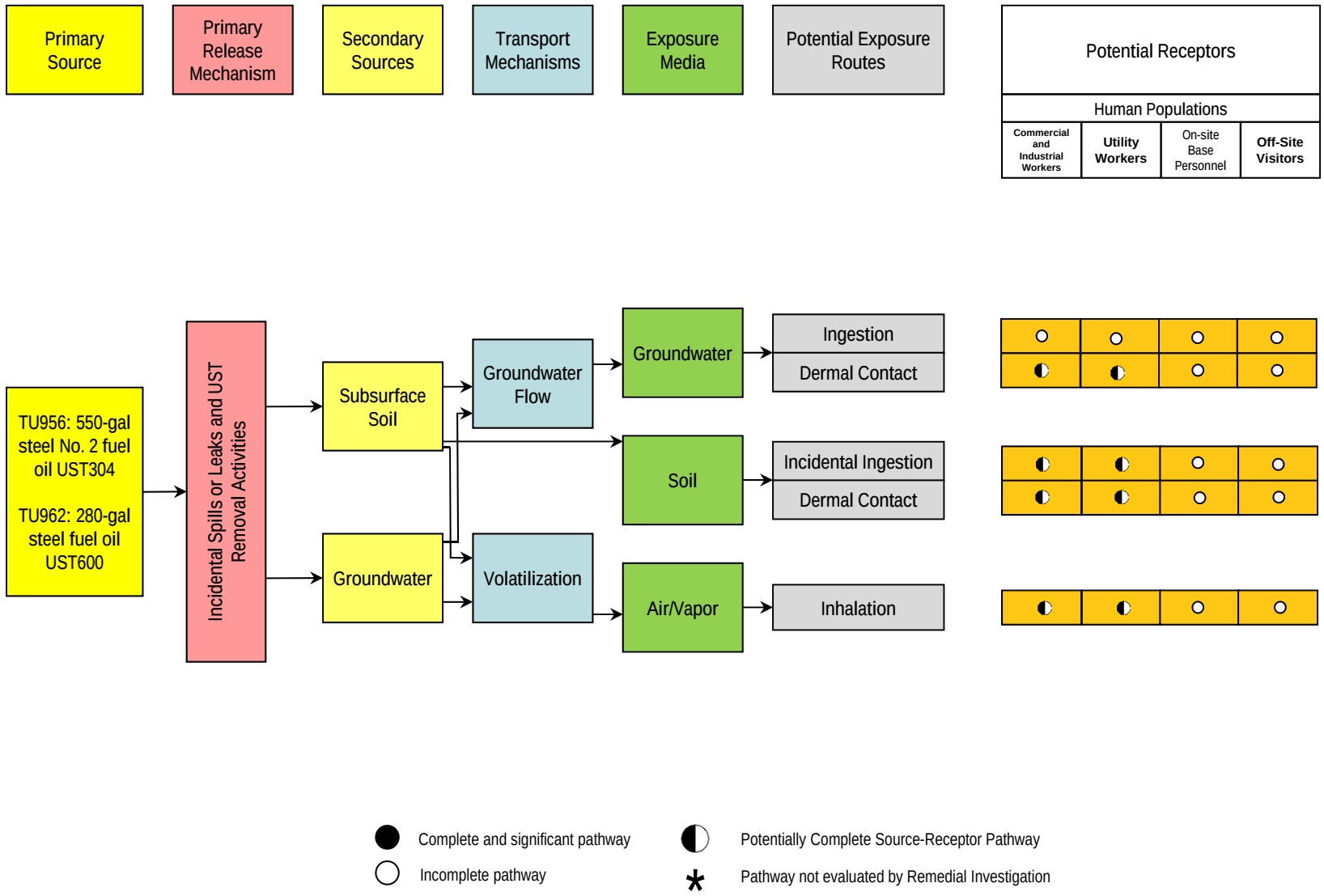

EA®	Unrestricted Use Characterization Quality Assurance Project Plan Niagara Falls Air Reserve Station Niagara Falls, New York	FIGURE 10-3 TU956 and TU962 Conceptual Site Model		
		PROJECT MGR: BY	DATE: AUGUST 2013	EA PROJECT NO: 6265401

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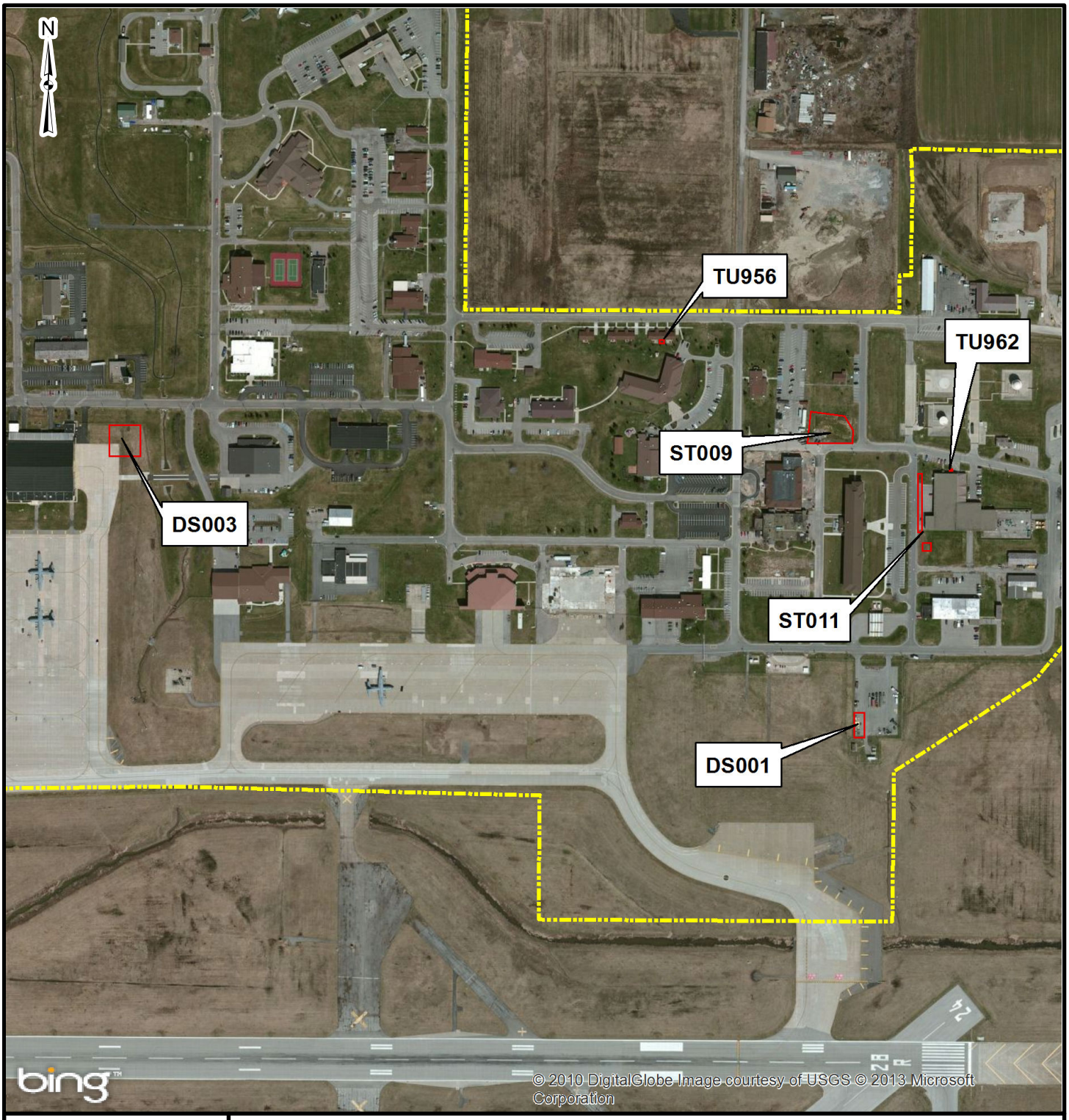

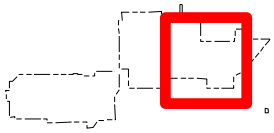

Installation Area

Approximate Site Boundaries

0 375 750

Feet

Unrestricted Use Characterization
Quality Assurance Project Plan
Niagara Falls Air Reserve Station
Niagara Falls, New York

FIGURE 10-4
Unrestricted Use Sites

PROJECT MGR:	DESIGNED BY:	CREATED BY:	CHECKED BY:	SCALE:	DATE:	PROJECT NO:	FILE NO:
BY	FD	FD	MH	AS SHOWN	AUGUST 2013	6265401	G:\Projects\Federal\DOD\6242107-Niagara Falls ARS

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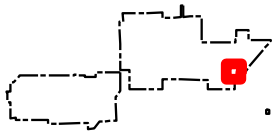

Approximate Site Boundary

Fence Line

0 300

Feet

Unrestricted Use Characterization
Quality Assurance Project Plan
Niagara Falls Air Reserve Station
Niagara Falls, New York

FIGURE 10-5
DS001 - Site 14
(Former AFRES Hazardous Waste Drum Storage)

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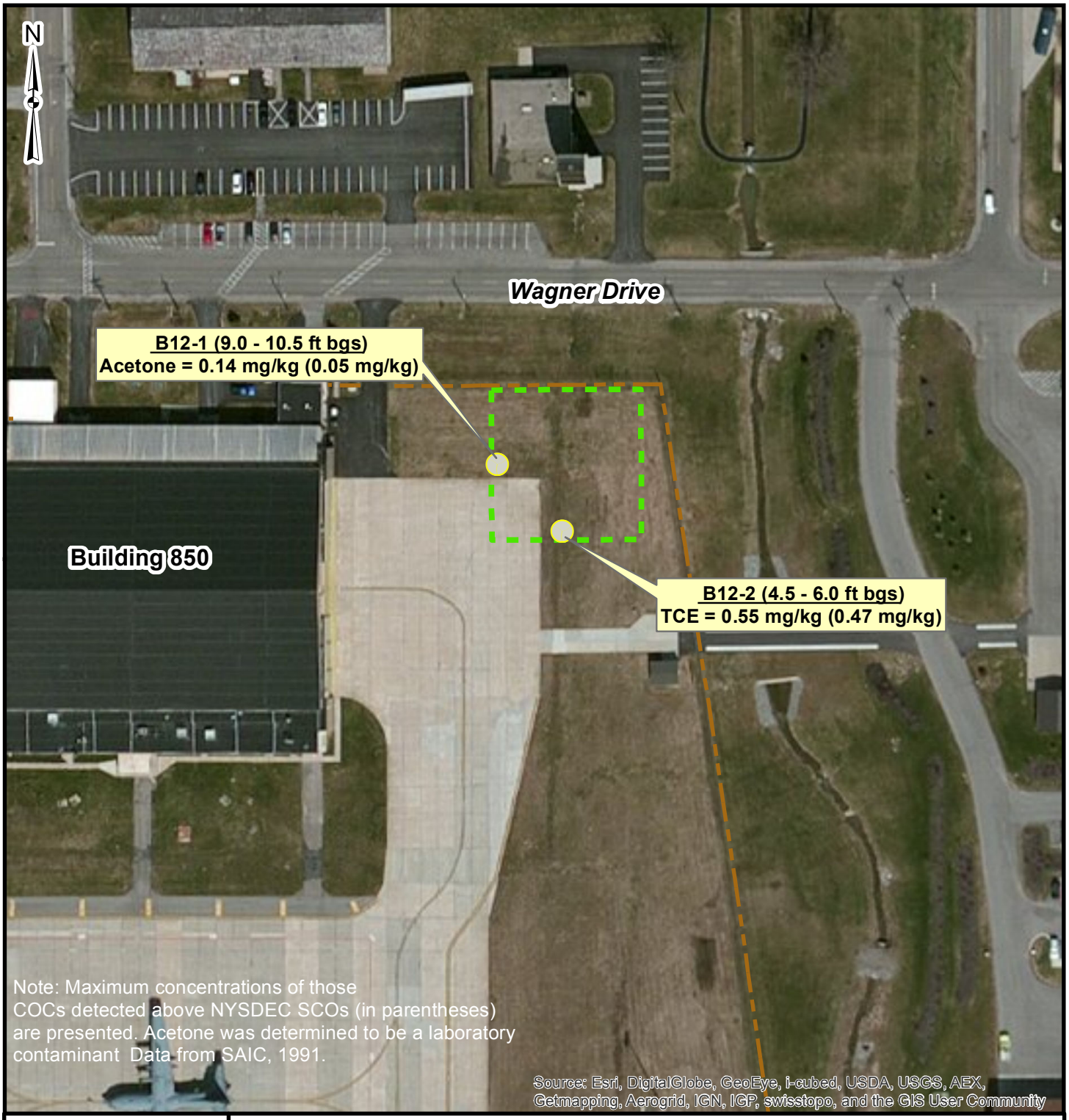

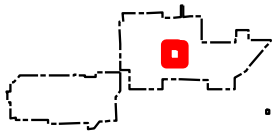

Approximate Site Boundary

Fence Line

Historical Subsurface Soil Sample (1989)

0 300

Feet

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Quality Assurance Project Plan
Niagara Falls Air Reserve Station
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FIGURE 10-6
DS003 - Site 12
(Former Building 850
Drum Storage Area)

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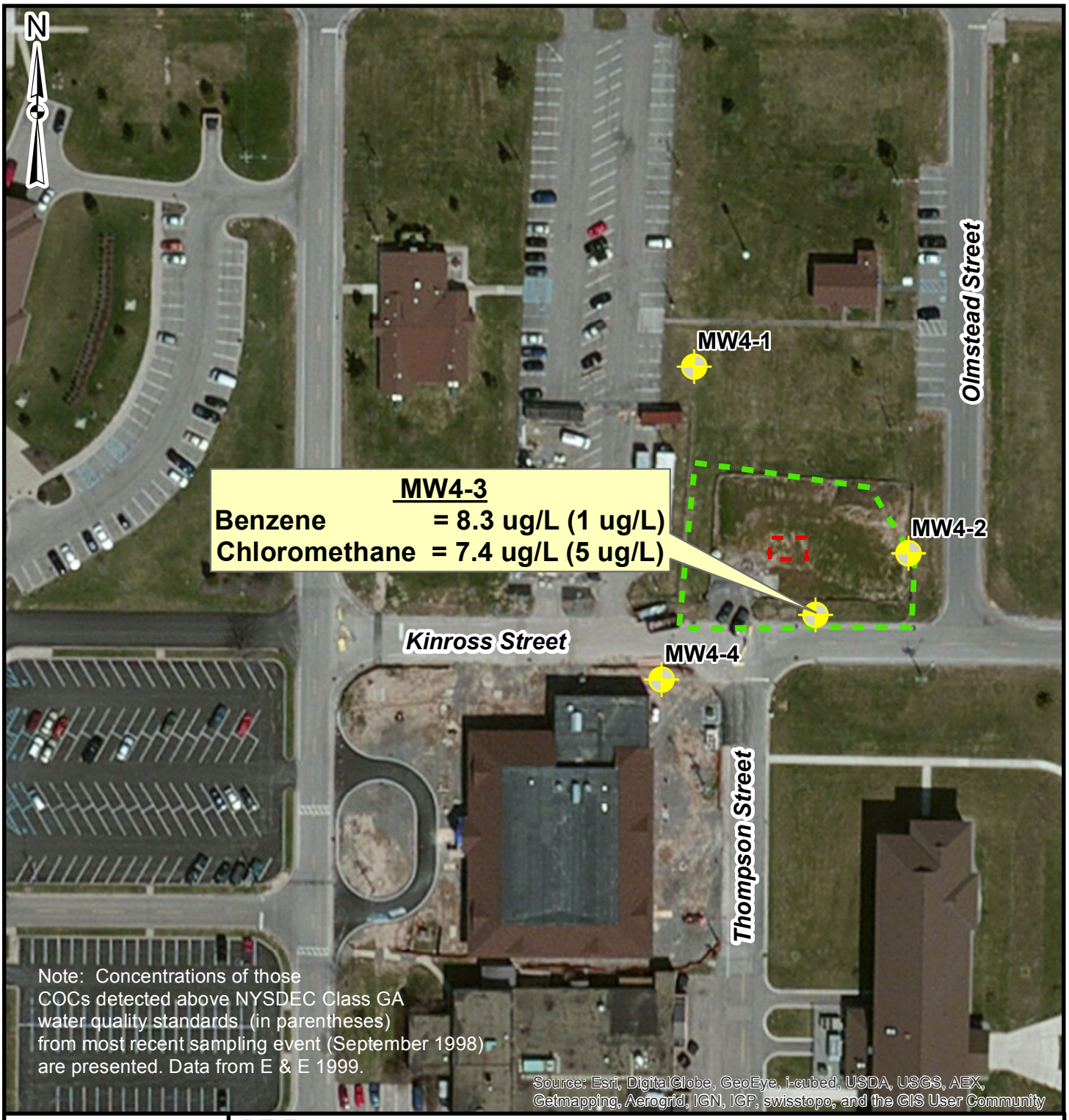

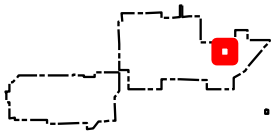

Approximate Site Boundary

Approximate Former Source Area

Former Monitoring Well

0 200

Feet

Unrestricted Use Characterization
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FIGURE 10-7
ST009 - Site 4
(Former MOGAS BX)

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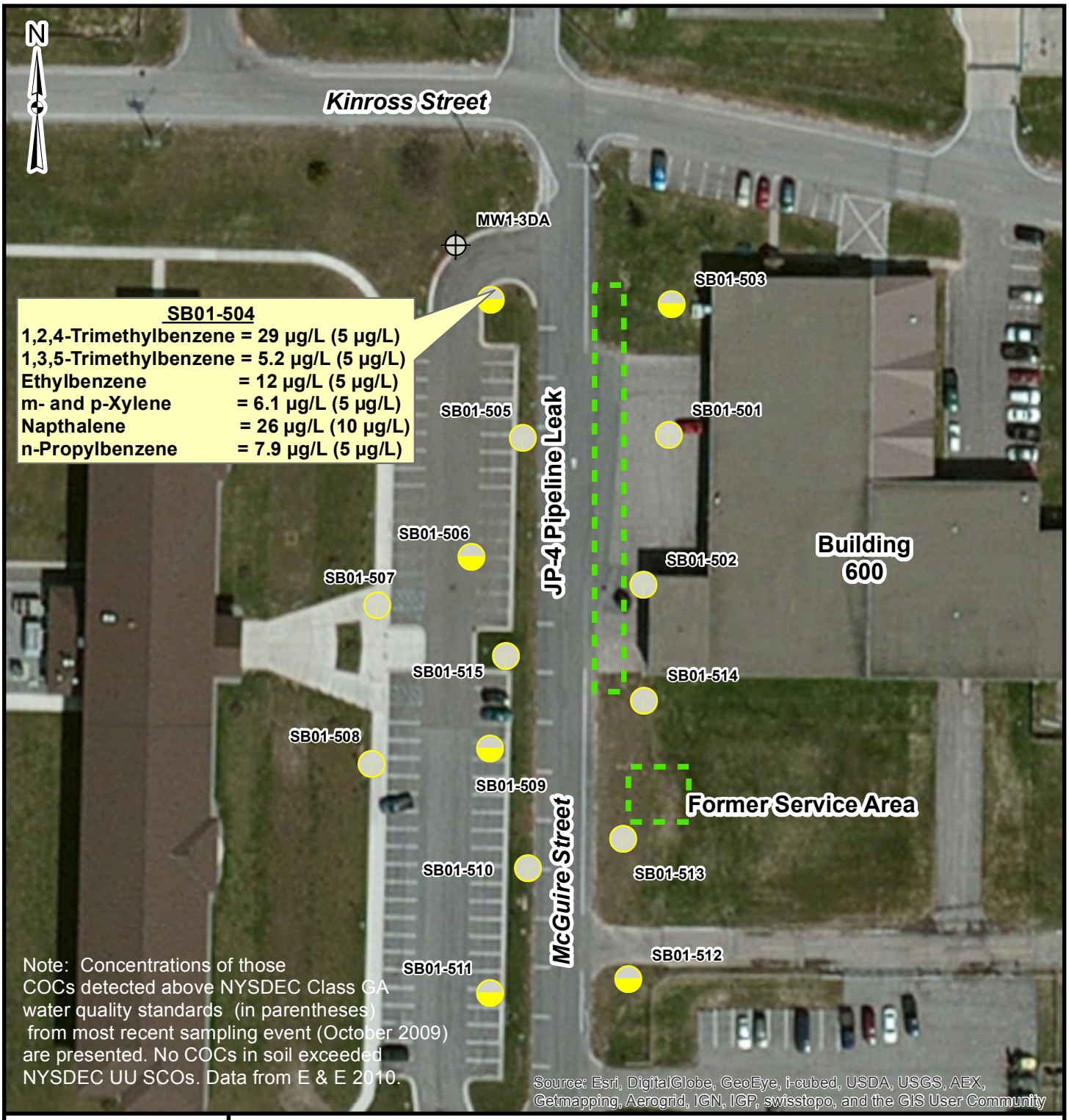

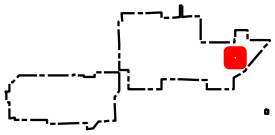

Approximate Former Source Area

Soil Sample Location (2009)

Soil and Groundwater Sample Location (2009)

0 100

Feet

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Niagara Falls Air Reserve Station
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FIGURE 10-8
ST011- Site 1 (Former JP-4 Leak)

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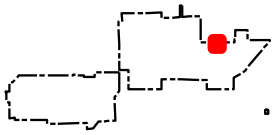

Approximate Area of UST Excavation

0 60

Feet

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FIGURE 10-9
TU956 (UST 304)

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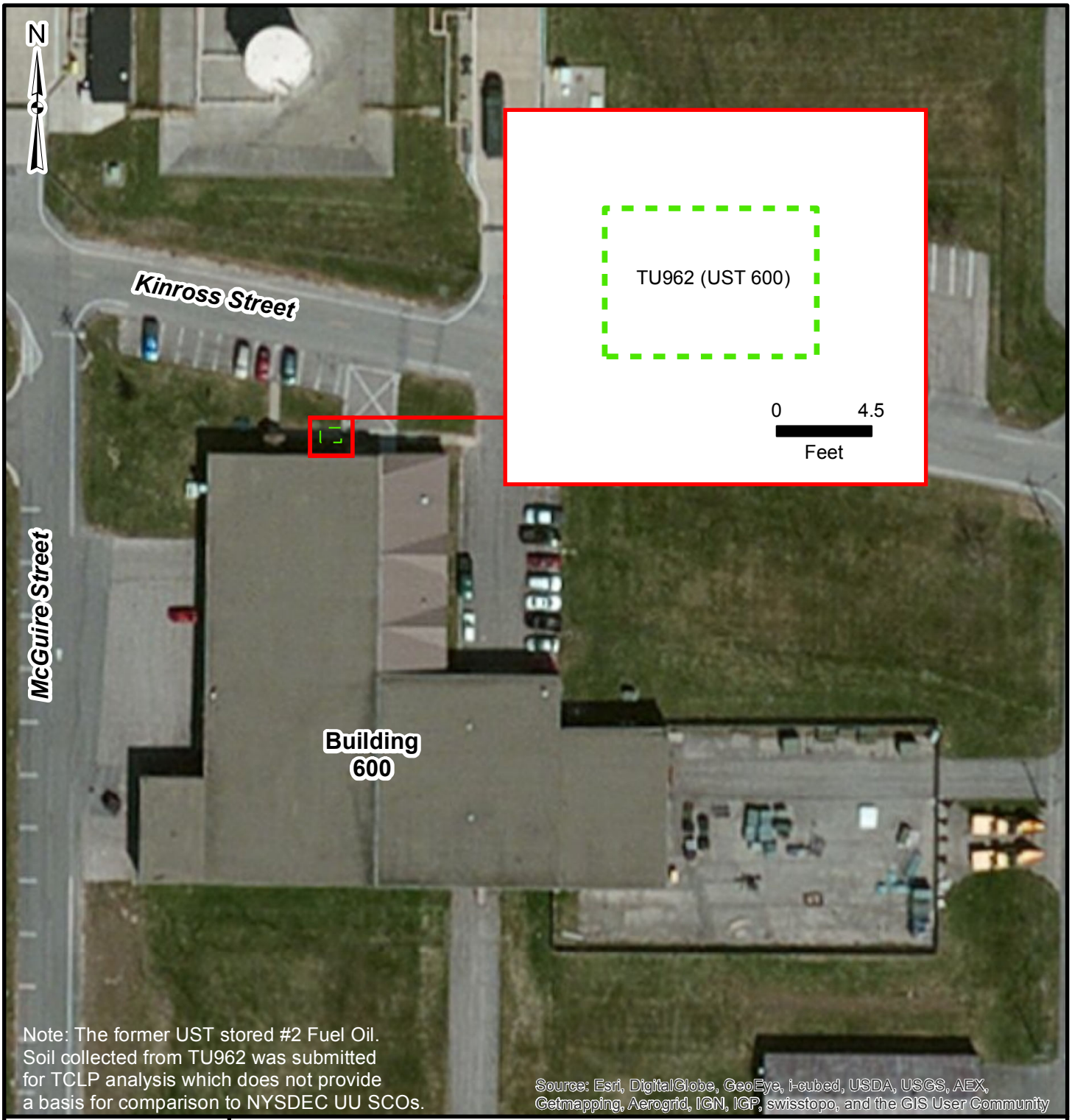

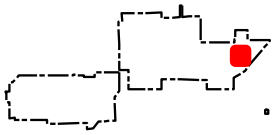

Approximate Area of UST Excavation

0 80
Feet

Unrestricted Use Characterization
Quality Assurance Project Plan
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Niagara Falls, New York

FIGURE 10-10
TU962 (UST 600)

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QAPP WORKSHEET #11

Project/Data Quality Objectives

This worksheet is used to develop and document project data quality objectives (DQOs) using a systematic planning process that follows the EPA DQO Process and documents the environmental decisions that need to be made and the level of data quality needed. The DQO process is outlined in the EPA 2006 guidance document entitled *Guidance on Systematic Planning Using the DQOs Process* (EPA/240/B-06/001, February 2006) (EPA 2006).

The seven steps are as follows: (1) State the Problem, (2) Identify the Goals of the Study, (3) Identify Information Inputs, (4) Define the Boundaries of the Study, (5) Develop the Analytic Approach, (6) Specify Performance or Acceptance Criteria, and (7) Develop a Detailed Plan for Data Collection. The specific QA/QC requirements developed for the site are consistent with those presented in the DoD Quality Systems Manual (QSM), Version 4.2 (DoD 2010).

1. State the Problem

Additional data are required to determine if potentially affected media for the sites meet UU criteria for a full suite of analytes because previous investigations did not fully characterize site media for all analytes. The potentially affected media at the sites is soil at all sites and groundwater at two sites. If soil samples meet the statewide UU SCOs, *"no action or study is warranted because of soil contamination"* per NYSDEC DER *Commissioner Policy on Soil Cleanup Guidance* (CP-51) (NYSDEC 2010b). In addition, per NYSDEC DER-10 (NYSDEC 2010a), because the UU SCOs incorporate the more restrictive criteria for protection of human health, ecological resources, or groundwater, achieving UU SCOs means that protection of groundwater and ecological resource requirements have already been met. Based on previous investigations, it is anticipated that the potential groundwater impacts are limited to sites: ST011 (JP-4 Pipeline Leak) and ST009 (BX MOGAS Leak). The following paragraphs summarize the site-specific considerations of this problem statement. The conceptual site model for each site is described in Worksheet #10 of this QAPP.

DS001 (Site 14) and DS003 (Site 12)

There have been no documented spills at DS001 or DS003. No corrective actions have been initiated at the sites. NFA status for DS001 and DS003 was approved by NYSDEC. Due to the lack of any documented releases at the sites, it is anticipated that DS001 and DS003 may meet UU criteria in soil and that no groundwater impacts have occurred. Additional soil characterization is needed to verify that the current soil concentrations meet the listed UU SCOs for the full suite of analytes and is protective of groundwater.

ST009 (Site 4)

A release occurred in 1982, due to a gasoline UST leak. Corrective actions, including the removal of the UST system, were completed at ST009. NYSDEC agreed that NFA would be required following groundwater monitoring that demonstrated a significant decline in benzene concentrations over time. No monitoring is currently required for the site under the existing RCRA Part 373 Permit. Based on the previous corrective actions completed at the site and the natural attenuation of benzene, it is anticipated that subsurface soil and groundwater concentrations have decreased to less than UU criteria. Additional soil and groundwater characterization is needed to verify that the current concentrations meet the listed UU criteria for the full suite of analytes.

ST011 (Site 1)

In 1969, an undetermined volume of JP-4 leaked from a hydrant system. Following spill response actions, including decommissioning and abandoning the JP-4 pipeline in place, and subsequent groundwater investigation and monitoring, NYSDEC agreed that NFA was required to address the JP-4 pipeline leak. In 2009 during a water line installation, soil with a petroleum odor was encountered; as a result, an additional site investigation was completed. The investigation included installation of 15 soil borings (SB01-501 through 514) and collection of 6 *in situ* groundwater samples. Petroleum-related COCs were detected at concentrations greater than NYSDEC Class GA groundwater standards in only one groundwater sample. Concentrations in soil samples did not exceed UU criteria. Additional soil and groundwater characterization is needed to verify that the current concentrations meet the listed UU criteria for the full suite of analyses.

TU956 (UST 304) and TU962 (UST 600)

Based upon review of historical information, it is anticipated that residual soil contamination at these sites, if present, is limited to near the former USTs. NYSDEC classified these former UST sites as "inactive" following tank removal although low-level detections of petroleum-related compounds remained based on post-excavation confirmatory samples. The low-level concentrations observed in the mid-1990s have likely decreased below UU criteria. Additional subsurface soil characterizations are needed at both sites to verify that the current concentrations meet the listed UU criteria for the full suite of analyses and that soil is protective of groundwater.

2. Identify the Goals of the Study

The data collected under this plan will be used to achieve the following objectives:

A. Determine if previous activities at the sites have impacted the subsurface. If contamination is identified, additional sampling or other remedial activities may be warranted, in which case a work plan would be developed.

    1. Collect soil samples for field-screening with a photoionization detector (PID) to evaluate the potential presence of VOCs.

    2. Collect soil samples for visual-manual classification, including documentation of indicators of potential contamination (e.g., staining, obvious odors).

    3. Collect soil samples for laboratory analysis of TCL VOCs and TCL SVOCs to determine if residual contamination remains and/or undocumented spills may have occurred.

    4. Collect soil samples to establish concentrations in soil for a full suite of analytes to evaluate whether UU criteria have been met.

    5. Collect in situ groundwater samples from ST009 and ST011 for a full suite of analytes to evaluate whether UU criteria have been met.

B. Verify that site media meet UU criteria. If the proposed actions described above indicate that site media do not meet UU criteria, additional site activities may be warranted, including additional groundwater investigation at sites where soil samples indicate the potential for impacts to groundwater. Per DER-10, soil concentrations above the statewide UU SCOs do not necessarily

represent a risk to human health and/or the environment (NYSDEC 2010a). An evaluation of any potential UU SCO exceedances would be completed to evaluate the potential source and risk associated with any exceedance.

3. Identify Information Inputs

To characterize the potentially-affected media to determine any residual contamination and evaluate current conditions with respect to UU criteria, the following data will be collected:

1. Soil borings will be installed (Figures 11-1 through 11-6) to collect soil samples for laboratory analysis.

2. A lithological description of the soil retrieved from the soil borings will be created.

3. At sites ST009 and ST011 in situ groundwater samples also will be collected and submitted for laboratory analysis.

Samples will be submitted for TCL VOCs and TCL SVOCs, with a percentage of samples submitted for laboratory analysis of a full suite of analytes as per DER-10 (NYSDEC 2010a). Sample design and rationale is discussed in Worksheet #17. Worksheet #18 summarizes the sampling program (including target analytes, analytical groups, and sample collection methods) that is proposed to satisfy the scope of the investigation.

4. Define Boundaries of the Study

Figures 11-1 through 11-6 present the proposed sampling locations for each site; thereby, defining the lateral boundaries for these media. Step 3 of the DQOs lists target analytes for soil and groundwater and will be detailed further in Worksheet #18. The vertical extent of the investigation will be as follows:

- Site DS001: 2 ft bgs based on the lack of a verified spill/incident.

- Site DS003: 2 ft bgs based on the lack of a verified spill/incident. An additional sample will be collected at the soil/groundwater interface based on a previous detection of TCE at 4.5–6 ft bgs.

- Site ST009: at the soil/groundwater interface or at bedrock (8–10 ft bgs).

- Site ST011: at the soil/groundwater interface or at bedrock (8–10 ft bgs).

- Site TU956: at the soil/groundwater interface or at bedrock (8–10 ft bgs).

- Site TU962: at the soil/groundwater interface or at bedrock (8–10 ft bgs).

The temporal extent of the field activities to be performed under this plan extends from August to October 2013.

5. Develop the Analytic Approach

Soil samples will be collected from borings located near former known and assumed source areas to evaluate any residual soil contamination.

DS001 (Site 14)

Surface soil (0–2 ft bgs) will be collected to evaluate the possibility of a historic surface release. Samples will be analyzed for TCL VOCs and SVOCs based on historic use. Additionally, for comparison to UU criteria, one soil sample will be analyzed for the full list of TCL organics (plus 10 VOC and 20 SVOC highest concentration tentatively identified compounds [TICs]) and TAL inorganic constituents, as per DER-10 (NYSDEC 2010a).

DS003 (Site 12)

Surface soil (0–2 ft bgs) will be collected to evaluate the possibility of a historic surface release. One additional subsurface soil sample will be collected in the vicinity of a previous detection of TCE at 4.5-6 ft bgs to confirm subsurface soil is protective of groundwater. Samples will be analyzed for TCL VOCs and SVOCs based on historic use. Additionally, for comparison to UU criteria, one soil sample will be analyzed for the full list of TCL organics (plus 10 VOC and 20 SVOC highest concentration TICs and TAL inorganic constituents) as per DER-10 (NYSDEC 2010a).

ST009 (Site 4)

Subsurface soil samples will be collected at the soil/groundwater interface to confirm that subsurface soil meets the UU criteria and remains protective of groundwater. Samples will be analyzed for TCL VOCs and SVOCs based on historic use. The analytes will encompass the suite of contaminants shown in the gasoline table (Table 3) of CP-51 (NYSDEC 2010b) because of a known gasoline release. Additionally, for comparison to UU criteria, one soil sample will be analyzed for the full list of TCL organics (plus 10 VOC and 20 SVOC highest concentration TICs and TAL inorganic constituents) as per DER-10 (NYSDEC 2010a).

ST011 (Site 12)

Subsurface soil samples will be collected at the soil/groundwater interface to confirm that subsurface soil meets the UU criteria and remains protective of groundwater. Samples will be analyzed for TCL VOCs and SVOCs based on historic use. The analytes will encompass the suite of contaminants shown in the fuel oil and gasoline tables (i.e., Tables 2 and 3) of CP-51 (NYSDEC 2010b) because of a known JP-4 release. Additionally, for comparison to UU criteria, one soil sample will be analyzed for the full list of TCL organics (plus 10 VOC and 20 SVOC highest concentration TICs and TAL inorganic constituents) as per DER-10 (NYSDEC 2010a).

TU956 (UST 304)

Subsurface soil samples will be collected at the soil/groundwater interface to confirm that subsurface soil meets the UU criteria and remains protective of groundwater. Samples will be analyzed for TCL VOCs and SVOCs based on historic use. The analytes will encompass the suite of contaminants shown in the fuel oil table (Table 3) of CP-51 (NYSDEC 2010b) because of the known UST. Additionally, for comparison to UU criteria, one soil sample will be analyzed for the full list of TCL organics (plus 10 VOC and 20 SVOC highest concentration TICs and TAL inorganic constituents) as per DER-10 (NYSDEC 2010a).

TU962 (UST 600)

Subsurface soil samples will be collected at the soil/groundwater interface to confirm that subsurface soil meets the UU criteria and remains protective of groundwater. Samples will be analyzed for TCL VOCs and SVOCs based on historic use. The analytes will encompass the suite of contaminants shown in the fuel oil table (Table 3) of CP-51 (NYSDEC 2010b) because of the known UST. Additionally, for comparison to UU criteria, one soil sample will be analyzed for the full list of TCL organics (plus 10 VOC and 20 SVOC highest concentration TICs and TAL inorganic constituents) as per DER-10 (NYSDEC 2010a).

Groundwater samples will be collected only at ST009 and ST011, where historical groundwater results indicated the presence of petroleum-related constituents (i.e., BTEX and naphthalene). In addition, one groundwater sample from each of these two areas will also be analyzed for full list of TCL organics (plus 10 VOC and plus 20 SVOC highest concentration TICs) and TAL inorganic constituents to determine whether groundwater concentrations have met NYSDEC Class GA groundwater standards and guidance values. For the remaining sites where only soil sampling is initially planned, additional *in situ* overburden groundwater samples may be collected if COPC concentrations in subsurface soil indicate the potential for impacts to groundwater.

6. Specify Performance or Acceptance Criteria

The analyte groups need to be sufficient to allow for comparison of the data to the full list of UU criteria. Therefore, the laboratory reporting limits and achievable laboratory detection limits need to be below those criteria. Laboratory analyses will be conducted by a DoD and New York State Department of Health (NYSDOH) Environmental Laboratory Accreditation Program (ELAP)-certified laboratory, using the most current NYSDEC Analytical Services Protocol (ASP) methods, as per NYSDEC DER-10 guidance (2010a). Category B laboratory data deliverables will be obtained. Following the receipt of analytical laboratory results, Data Usability Summary Reports (DUSRs) will be prepared by an independent, third-party. The validated data will be compared to the appropriate regulatory criteria.

Additional detail on sampling methodology, analyses, and equipment is provided in subsequent QAPP worksheets.

7. Develop a Detailed Plan for Data Collection

Locations for field activities were chosen based on review of historical aerial photographs, previous investigation data, and review of underground utilities. Worksheet #17 provides the sample design and rationale; Worksheet #18 provides additional detail on sample locations, media, suite of analytes, and sample collection tools; and Worksheet #20 provides information on QC samples. Worksheets #26 through #28 in the QAPP provide specific detail on the analytical requirements.

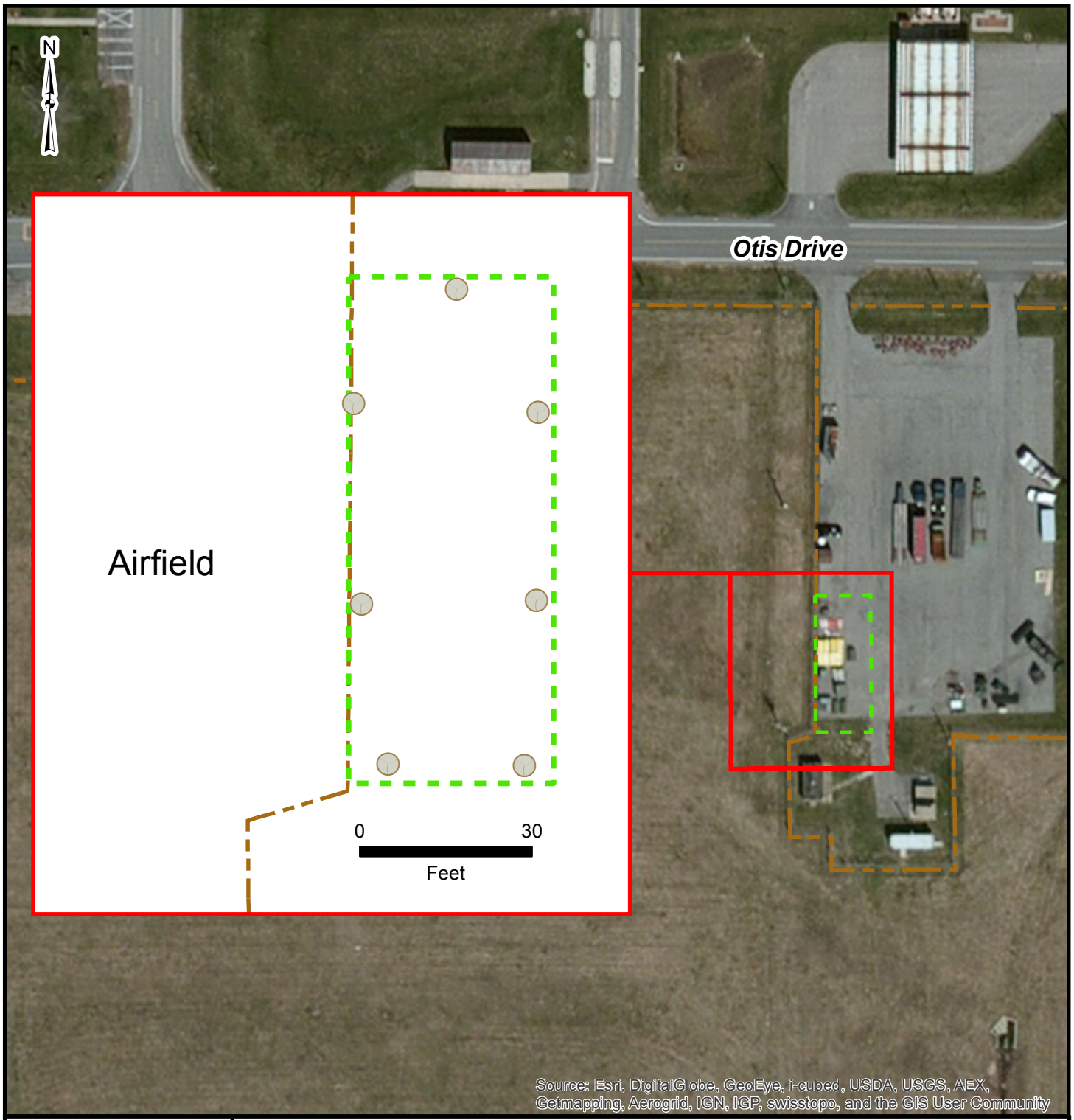
How Will Data Be Reported

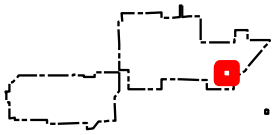
A Site Characterization Report will be prepared at the conclusion of the field operations and will consist of a comprehensive compilation of the data collected under this project. The report will include a detailed narrative of each field activity, a summary of the sampling conducted, any deviations from this QAPP, data assessment and evaluation, an interpretation of data as per the scope of this plan, an identification of any remaining data gaps, and conclusions and recommendations. Site drawings, figures, laboratory analytical reports, field forms, and photographs documenting field activities will be included as attachments to the report. The analytical data will be reported in Excel™ summary tables to facilitate

data analysis. Ultimately, the report will be comprehensive in nature and no additional sources of information will be necessary to capture the full extent of the field operations and data collected.

How Will Data Be Archived

The electronic data deliverables and laboratory data reports will be collected in project archives in existing electronic formats provided by the analytical laboratory. Data will be submitted to, and archived with, AFCEC through the Environmental Resources Program Information Management System (ERPIMS). ERPIMS is the database that the Air Force uses for data management and validation from environmental projects at all AFBs. Data will also be archived in the NFARS base-specific database, as well as the NYSDEC Environmental Information Management System in the EQuIS™ electronic data deliverable format.





Approximate Site Boundary

Fence Line

Proposed Soil Boring

0 300

Feet

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FIGURE 11-1
Proposed Sampling Locations
DS001 - Site 14
(Former AFRES Haz
Waste Drum Storage)

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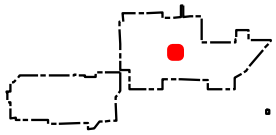

Approximate Site Boundary

Fence Line

Proposed Soil Boring

0 100

Feet

Unrestricted Use Characterization
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FIGURE 11-2
Proposed Sampling Locations
DS003 - Site 12
(Former Bldg 850
Drum Storage Area)

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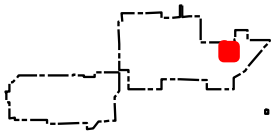

Approximate Site Boundary
Approximate Former Source Area
Approximate Groundwater Flow Direction

Proposed Soil Boring with Groundwater Sample
Proposed Soil Boring

0 120
Feet

Unrestricted Use Characterization
Quality Assurance Project Plan
Niagara Falls Air Reserve Station
Niagara Falls, New York

FIGURE 11-3
Proposed Sampling Locations
ST009 - Site 4
(Former MOGAS BX)

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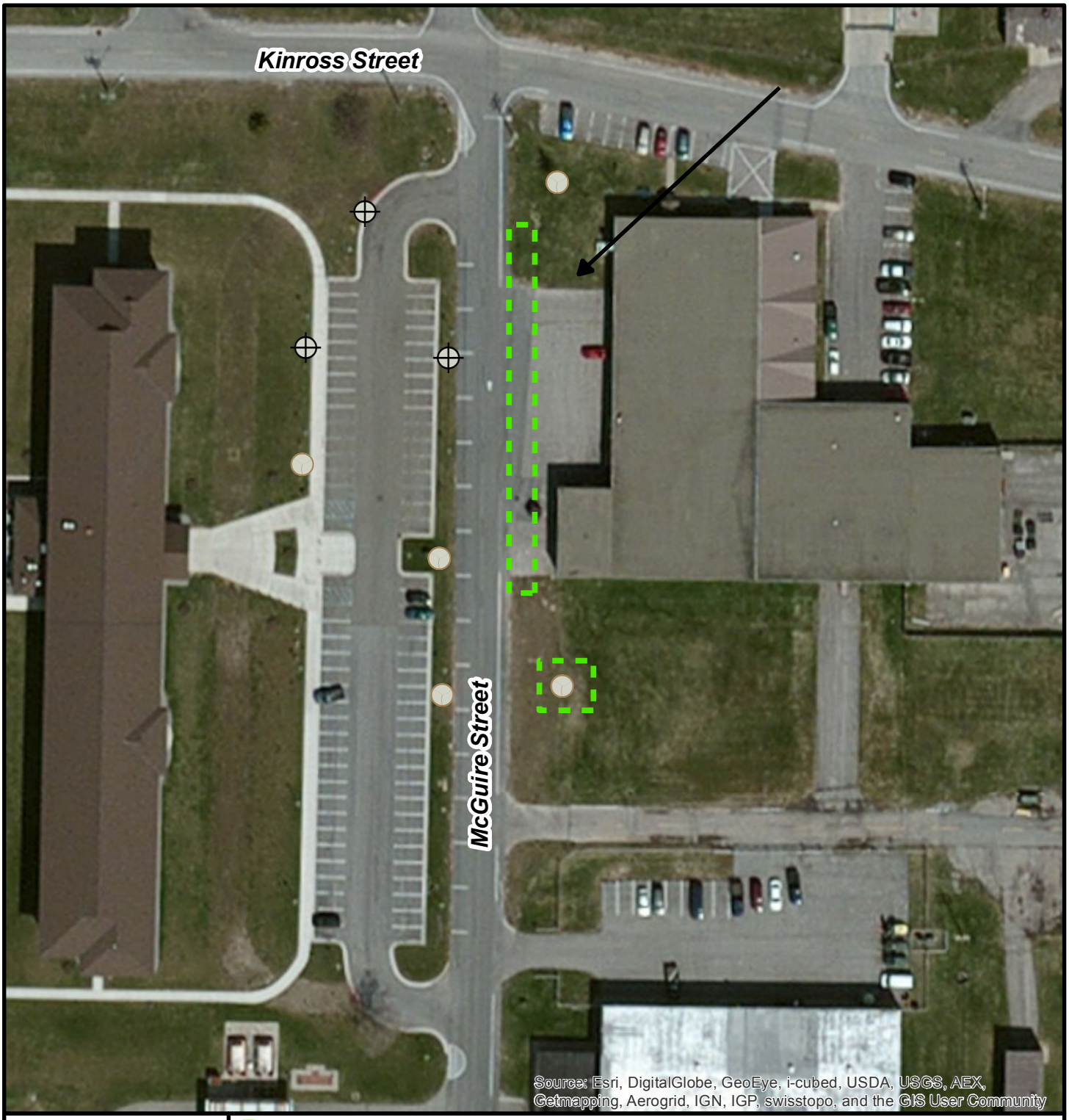

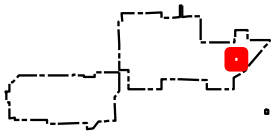

Approximate Former Source Area

Approximate Groundwater Flow Direction

Soil Boring with Groundwater Sample

Soil Boring

0 150

Feet

Unrestricted Use Characterization
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Niagara Falls Air Reserve Station
Niagara Falls, New York

FIGURE 11-4
Proposed Sampling Locations
ST011- Site 1 (Former JP-4 Leak)

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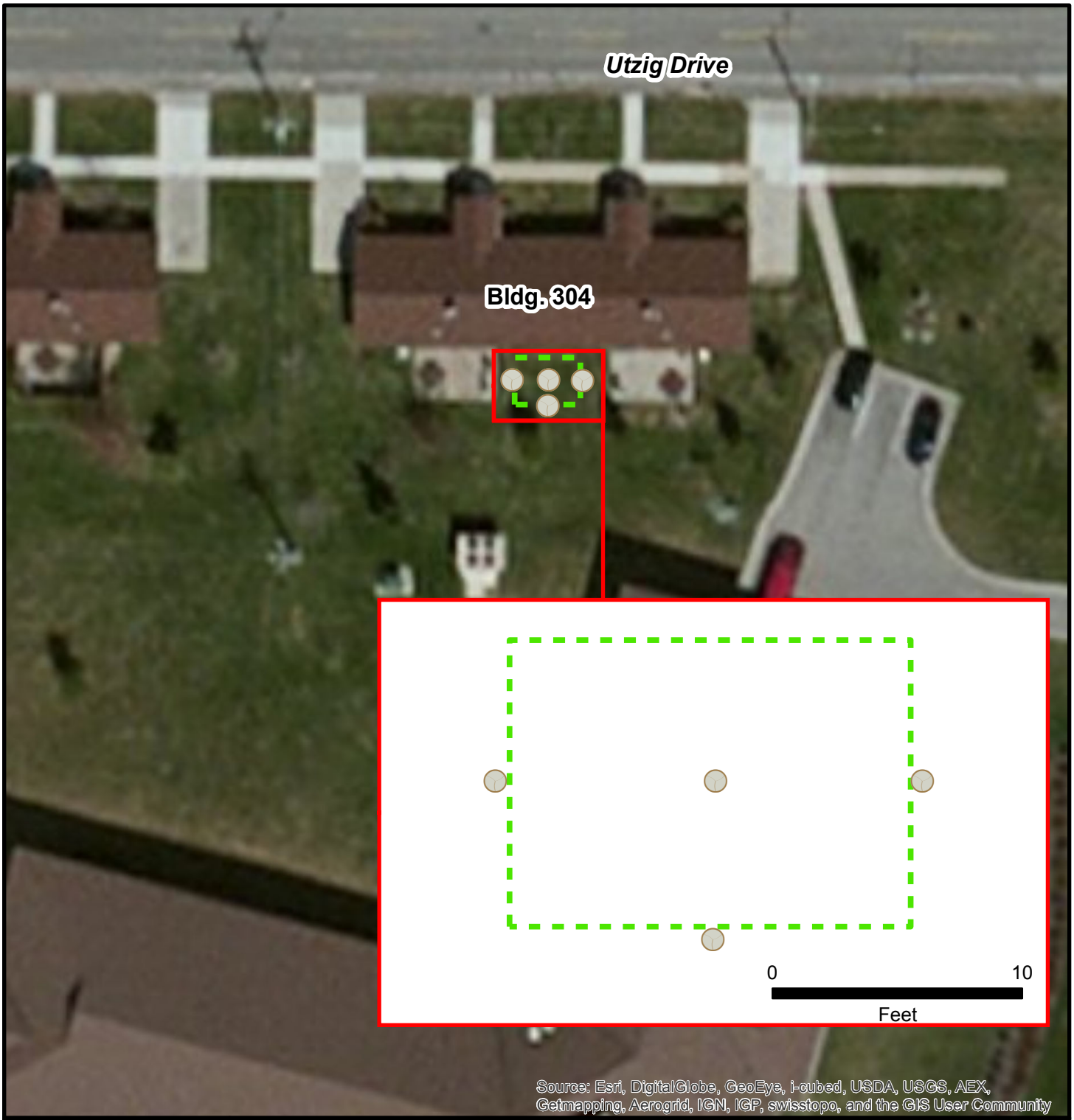

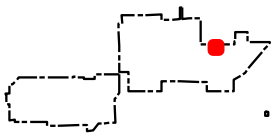

Approximate Arear of UST Excavation

Proposed Soil Boring

0 50

Feet

Unrestricted Use Characterization
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FIGURE 11-5
Proposed Sampling Locations
TU956 (UST 304)

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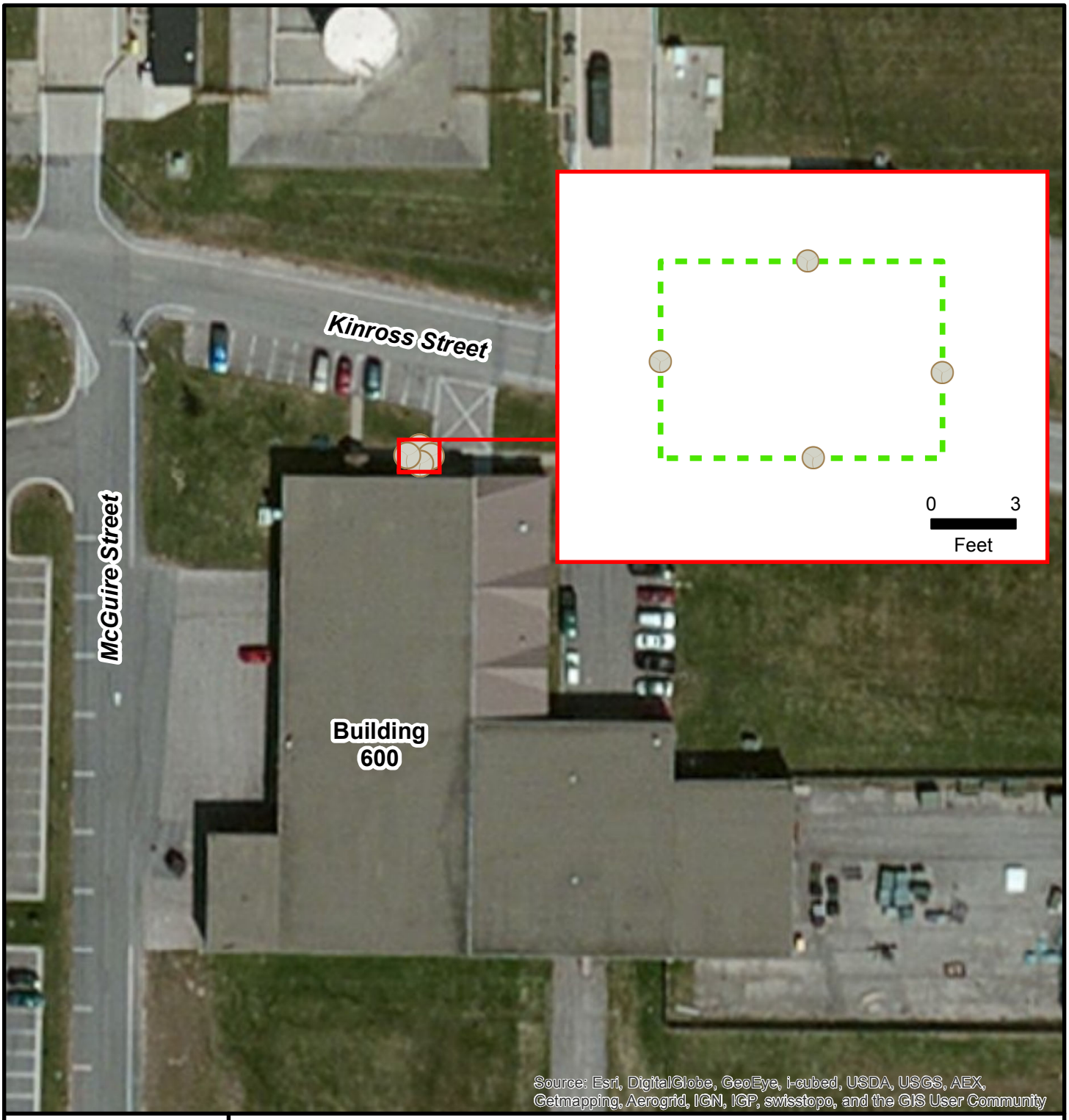

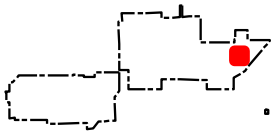

Approximate Area of UST Excavation

Proposed Soil Boring

0 80

Feet

Unrestricted Use Characterization
Quality Assurance Project Plan
Niagara Falls Air Reserve Station
Niagara Falls, New York

FIGURE 11-6
Proposed Sampling Locations
TU962 (UST 600)

PROJECT MGR: BY	DESIGNED BY: FD	CREATED BY: FD	CHECKED BY: MH	SCALE: AS SHOWN	DATE: AUGUST 2013	PROJECT NO: 6265401	FILE NO: G:\Projects\Federal\DOD\ 6265401_AFCEC_NFARS\

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QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-1

Matrix	Soil
Analytical Group	VOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 11, SOP 15, SOP 16, SOP 25, SOP 31, SOP 39, SOP 47, SOP 59 (see QAPP Worksheet #21)	SW8260C/ 90.0012	Completeness	≥90%	NA	S&A
		Precision	RPD ≤50%	Field duplicate	S&A
		Accuracy/bias/ contamination	No target analytes ≥ ½ LOQ; for common laboratory contaminants no analytes detected ≥ LOQ	Blanks (trip and other field blanks and method blank)	S&A
		Accuracy/bias	≤20 %D	ICV and CCV	A
		Qualitative Identification (ID)	Relative retention time (RRT) of each target analyte within ± 0.06 RRT units of most recent mid-point initial calibration (ICAL) or CCV	RRT	A
		Instrument performance	Tune criteria consistent with SW8260C	Mass spectrometer tuning check bromofluorobenzene (BFB)	A
		Sensitivity	Retention time (RT) ±30 seconds from RT of the internal standard (IS) of the calibration mid-point standard and extracted ion current profile area within from -50% to +100% of area from IS calibration mid-point standard	Internal Standard (IS)	A
		Accuracy	%R, DoD QSM (Version 4.2) limits.	LCS, MS, and MSD	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	Surrogates	A

NOTE: SOP = Standard Operating Procedure
RPD = Relative Percent Difference
LOQ = Limit of Quantitation
%D = Percent Difference
ICV = Initial Calibration Verification
CCV = Continuing Calibration Verification
%R = Percent Recovery
LCS = Laboratory Control Sample
MS = Matrix Spike
MSD = Matrix Spike Duplicate

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-2

Matrix	Soil
Analytical Group	SVOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 11, SOP 15, SOP 16, SOP 25, SOP 31, SOP 39, SOP 47, SOP 59 (see QAPP Worksheet #21)	SW8270D / 70.0011	Completeness	>90%	NA	S&A
		Precision	≤50% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	No target analytes ≥½ LOQ For common laboratory contaminants no analytes detected > LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	≤20%D	ICV and CCV	A
		Qualitative ID	RRT of each target analyte within ±0.06 RRT units	RRT	A
		Instrument Performance	Tune criteria consistent with SW8270D	Mass spectrometer tuning check decafluorotriphenylphosphine (DFTPP)	A
		Sensitivity	Retention time ±30 seconds from retention time of the IS of the calibration mid-point standard and extracted ion current profile area within -50% to +100% of area from IS calibration mid-point standard	IS	A
		Accuracy	%R, DoD QSM (Version 4.2) limits, if available.	LCS, MS, MSD	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	Surrogate standards	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-3

Matrix	Soil
Analytical Group	PAHs and 1,4-Dioxane
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 11, SOP 15, SOP 16, SOP 25, SOP 31, SOP 39, SOP 47, SOP 59 (see QAPP Worksheet #21)	SW8270D Selected ion monitoring (SIM)/70.0033	Completeness	>90%	NA	S&A
		Precision	≤50% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	No target analytes ≥½ LOQ For common laboratory contaminants no analytes detected > LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	≤20%D	ICV and CCV	A
		Qualitative ID	RRT of each target analyte within ±0.06 RRT units	RRT	A
		Instrument Performance	Tune criteria consistent with SW8270D	Mass spectrometer tuning check DFTPP	A
		Sensitivity	Retention time ±30 seconds from retention time of the IS of the calibration mid-point standard and extracted ion current profile area within -50% to +100% of area from IS calibration mid-point standard	IS	A
		Accuracy	%R, DoD QSM (Version 4.2) limits, if available.	LCS, MS, MSD	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	Surrogate standards	A

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Measurement Performance Criteria Table 12-4

Matrix	Soil
Analytical Group	Pesticides
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 11, SOP 15, SOP 16, SOP 25, SOP 31, SOP 39, SOP 47, SOP 59 (see QAPP Worksheet #21)	SW8081B / 60.0006	Completeness	>90%	NA	S&A
		Precision	≤50%RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	< ½ of the LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	≤20%D	ICV and CCV	A
		Accuracy	Breakdown check (Endrin/DDT)	Degradation ≤15% for both DDT and Endrin	A
		Accuracy	%R, DoD QSM (Version 4.2) limits, if available.	LCS, MS, MSD	A
		Accuracy	Results from primary and secondary columns ≤40% RPD	Confirmation of positive results	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	Surrogate	A

NOTE: DDT = Dichlorodiphenyltrichloroethane

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-5

Matrix	Soil
Analytical Group	Herbicides
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 11, SOP 15, SOP 16, SOP 25, SOP 31, SOP 39, SOP 47, SOP 59 (see QAPP Worksheet #21)	SW8151A / 60.0034	Completeness	>90%	NA	S&A
		Precision	≤50% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	<½ of theLOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	≤20%D	ICV and CCV	A
		Accuracy	%R, DoD QSM (Version 4.2) limits, if available.	LCS, MS, MSD	A
		Accuracy	Results from primary and secondary columns ≤40% RPD	Confirmation of positive results	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits, if available.	Surrogate	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-6

Matrix	Soil
Analytical Group	PCBs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 11, SOP 15, SOP 16, SOP 25, SOP 31, SOP 39, SOP 47, SOP 59 (see QAPP Worksheet #21)	SW8082A / 60.0003	Completeness	>90%	NA	S&A
		Precision	≤50% RPD	Field duplicate	S&A
		Accuracy/bias / contamination	<½ of the LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	≤20%D	ICV and CCV	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	LCS, MS, MSD	A
		Accuracy	Results from primary and secondary columns ≤40% RPD	Confirmation of positive results	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	Surrogate	A

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Measurement Performance Criteria Table 12-7

Matrix	Soil
Analytical Group	Metals
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 11, SOP 15, SOP 16, SOP 25, SOP 31, SOP 39, SOP 47, SOP 59 (see QAPP Worksheet #21)	SW6010C / 100.0111	Completeness	90%	NA	S&A
		Precision	RPD≤50%	Field duplicate	S&A
		Accuracy/bias/ contamination	≤½ of the LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias/ contamination	<Limit of detection (LOD)	Initial calibration blank (ICB) and continuing calibration blanks (CCB)	A
		Accuracy/bias	≤10%R	ICV and CCV	A
		Accuracy/bias	≤20%R	Low-level calibration check (only if one-point calibration is used)	A
		Accuracy/bias	ICS-A: the non-spiked analytes< LOQ, ICS-AB: within ±20% of expected value	Interfering element check standards (ICS)	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	LCS, MS, and MSD	A
		Precision	≤20%RPD	MS and MSD	A
		Accuracy/bias	5-fold dilution within ±10% of original value	Serial dilution	A
		Accuracy/bias	Within ±25% of expected value	Post-digestion spike	A

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Measurement Performance Criteria Table 12-8

Matrix	Soil
Analytical Group	Hexavalent Chromium
Concentration Level	Low

Sampling Procedure	**Analytical Method/SOP**	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 11, SOP 15, SOP 16, SOP 25, SOP 31, SOP 39, SOP 47, SOP 59 (see QAPP Worksheet #21)	SW7196A/ 100.0208	Completeness	90%	NA	S&A
		Precision	≤50% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	<½ of the LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	90-110%R	ICV and CCV	A
		Accuracy	80-120%R	LCS	A
		Accuracy/Precision	75-125%R, ≤30% RPD	MS and MSD	A
		Accuracy/bias	85-115%R	Post-digestion spike	A

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Measurement Performance Criteria Table 12-9

Matrix	Soil
Analytical Group	Mercury
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 11, SOP 15, SOP 16, SOP 25, SOP 31, SOP 39, SOP 47, SOP 59 (see QAPP Worksheet #21)	SW7471B / 100.0012	Completeness	90%	NA	S&A
		Precision	≤50% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	<½ of the LOQ	Field blanks, laboratory reagent blank, or method blank	A
		Accuracy/bias/ contamination	< LOD	Blanks (ICB and CCB)	A
		Accuracy/bias	90-110%R	ICV/CCV	A
		Accuracy	80-120%R	LCS , MS, and MSD	A
		Precision	≤20% RPD	MS and MSD	A

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Measurement Performance Criteria Table 12-10

Matrix	Soil
Analytical Group	Cyanide
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 11, SOP 15, SOP 16, SOP 25, SOP 31, SOP 39, SOP 47, SOP 59 (see QAPP Worksheet #21)	SW9012B / 100.0004	Completeness	90%	NA	S&A
		Precision	≤50% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	<½ of the LOQ	Field blanks, laboratory reagent blank, or method blank	A
		Accuracy/bias/ contamination	< LOD	Blanks (ICB and CCB)	A
		Accuracy/bias	85-115%R	ICV/CCV	A
		Accuracy	80-120%R	LCS , MS, and MSD	A
		Precision	≤20% RPD	MS and MSD	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-11

Matrix	Water
Analytical Group	VOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 10, SOP 13, SOP 15, SOP 16, SOP 19, SOP 28, SOP 31, SOP 39, SOP 43, SOP 47, SOP 48, SOP 59 (see QAPP Worksheet #21)	SW8260C/ 90.0012	Completeness	≥95%	NA	S&A
		Precision	≤30% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	No target analytes ≥ ½ LOQ; for common laboratory contaminants no analytes detected > LOQ	Blanks (trip and other field blanks and method blank)	S&A
		Accuracy/bias	≤20%D	ICV and CCV	A
		Qualitative ID	RRT of each target analyte within ±0.06 RRT units of most recent mid-point ICAL or CCV	RRT	A
		Instrument performance	Tune criteria consistent with SW8260B	Mass spectrometer tuning check BFB	A
		Sensitivity	RT ± 30 seconds from RT of the IS of the calibration mid-point standard and extracted ion current profile area within from -50% to +100% of area from IS calibration mid-point standard	IS	A
		Accuracy	%R, DoD QSM (Version 4.2) limits, if available.	LCS, MS, and MSD	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	Surrogates	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-12

Matrix	Water
Analytical Group	SVOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 10, SOP 13, SOP 15, SOP 16, SOP 19, SOP 28, SOP 31, SOP 39, SOP 43, SOP 47, SOP 48, SOP 59 (see QAPP Worksheet #21)	SW8270D/70.0012	Completeness	>95%	NA	S&A
		Precision	≤30% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	No target analytes ≥½ LOQ For common laboratory contaminants no analytes detected > LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	≤20%D	ICV and CCV	A
		Qualitative ID	RRT of each target analyte within ±0.06 RRT units	RRT	A
		Instrument Performance	Tune criteria consistent with SW8270D	Mass spectrometer tuning check DFTPP	A
		Sensitivity	Retention time ±30 seconds from retention time of the IS of the calibration mid-point standard and extracted ion current profile area within -50% to +100% of area from IS calibration mid-point standard	IS	A
		Accuracy	%R, DoD QSM (Version 4.2) limits, if available.	LCS, MS, MSD	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	Surrogate standards	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-13

Matrix	Water
Analytical Group	PAHs and 1,4-Dioxane
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 10, SOP 13, SOP 15, SOP 16, SOP 19, SOP 28, SOP 31, SOP 39, SOP 43, SOP 47, SOP 48, SOP 59 (see QAPP Worksheet #21)	SW8270DSIM / 70.0033	Completeness	>95%	NA	S&A
		Precision	≤30% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	No target analytes ≥½ LOQ For common laboratory contaminants no analytes detected > LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	≤20%D	ICV and CCV	A
		Qualitative ID	RRT of each target analyte within ±0.06 RRT units	RRT	A
		Instrument Performance	Tune criteria consistent with SW8270D	Mass spectrometer tuning check DFTPP	A
		Sensitivity	Retention time ±30 seconds from retention time of the IS of the calibration mid-point standard and extracted ion current profile area within -50% to +100% of area from IS calibration mid-point standard	IS	A
		Accuracy	%R, DoD QSM (Version 4.2) limits, if available.	LCS, MS, MSD	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	Surrogate standards	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-14

Matrix	Water
Analytical Group	Pesticides
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 10, SOP 13, SOP 15, SOP 16, SOP 19, SOP 28, SOP 31, SOP 39, SOP 43, SOP 47, SOP 48, SOP 59 (see QAPP Worksheet #21)	SW8081B / 60.0006	Completeness	>95%	NA	S&A
		Precision	≤30%RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	< ½ of the LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	≤20%D	ICV and CCV	A
		Accuracy	Breakdown check (Endrin/DDT)	Degradation ≤15% for both DDT and Endrin	A
		Accuracy	%R, DoD QSM (Version 4.2) limits, if available.	LCS, MS, MSD	A
		Accuracy	Results from primary and secondary columns ≤40% RPD	Confirmation of positive results	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	Surrogate	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-15

Matrix	Water
Analytical Group	Herbicides
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 10, SOP 13, SOP 15, SOP 16, SOP 19, SOP 28, SOP 31, SOP 39, SOP 43, SOP 47, SOP 48, SOP 59 (see QAPP Worksheet #21)	SW8151A / 60.0034	Completeness	>95%	NA	S&A
		Precision	≤30% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	<½ of the LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	≤20%D	ICV and CCV	A
		Accuracy	%R, DoD QSM (Version 4.2) limits, if available.	LCS, MS, MSD	A
		Accuracy	Results from primary and secondary columns ≤40% RPD	Confirmation of positive results	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits, if available.	Surrogate	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-16

Matrix	Water
Analytical Group	PCBs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 10, SOP 13, SOP 15, SOP 16, SOP 19, SOP 28, SOP 31, SOP 39, SOP 43, SOP 47, SOP 48, SOP 59 (see QAPP Worksheet #21)	SW8082A/60.0003	Completeness	>95%	NA	S&A
		Precision	≤30% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	<½ of the LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	≤20%D	ICV and CCV	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	LCS, MS, MSD	A
		Accuracy	Results from primary and secondary columns ≤40% RPD	Confirmation of positive results	A
		Precision	RPD≤30%	MS and MSD	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	Surrogate	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-17

Matrix	Water
Analytical Group	Metals
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 10, SOP 13, SOP 15, SOP 16, SOP 19, SOP 28, SOP 31, SOP 39, SOP 43, SOP 47, SOP 48, SOP 59 (see QAPP Worksheet #21)	SW6020A/ 100.0110	Completeness	90%	NA	S&A
		Precision	RPD≤50%	Field duplicate	S&A
		Accuracy/bias/ contamination	≤½ of theLOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias/ contamination	<Limit of detection (LOD)	ICB CCB	A
		Accuracy/bias	≤10%R	ICV and CCV	A
		Accuracy/bias	≤20%R	Low-level calibration check (only if one-point calibration is used)	A
		Accuracy/bias	ICS-A: the non-spiked analytes< LOQ, ICS-AB: within ±20% of expected value	Interfering element check standards (ICS)	A
		Accuracy	%R, DoD QSM (Version 4.2) limits	LCS, MS, and MSD	A
		Precision	≤20%RPD	MS and MSD	A
		Accuracy/bias	5-fold dilution within ±10% of original value	Serial dilution	A
		Accuracy/bias	Within ±25% of expected value	Post-digestion spike	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-18

Matrix	Water
Analytical Group	Hexavalent Chromium
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 10, SOP 13, SOP 15, SOP 16, SOP 19, SOP 28, SOP 31, SOP 39, SOP 43, SOP 47, SOP 48, SOP 59 (see QAPP Worksheet #21)	SM3500 B Cr+6/100.0308	Completeness	90%	NA	S&A
		Precision	≤50% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	<½ of the LOQ	Blanks (method blank and field blanks)	A
		Accuracy/bias	90-110%R	ICV and CCV	A
		Accuracy	80-120%R	LCS, MS, and MSD	A
		Precision	75-125%R, ≤20% RPD	MS and MSD	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-19

Matrix	Water
Analytical Group	Mercury
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 10, SOP 13, SOP 15, SOP 16, SOP 19, SOP 28, SOP 31, SOP 39, SOP 43, SOP 47, SOP 48, SOP 59 (see QAPP Worksheet #21)	SW7470A / 100.0012	Completeness	95%	NA	S&A
		Precision	≤50% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	<½ of the LOQ	Field blanks, laboratory reagent blank, or method blank	A
		Accuracy/bias/ contamination	< LOD	Blanks (ICB and CCB)	A
		Accuracy/bias	90-110%R	ICV/CCV	A
		Accuracy	80-120%R	LCS , MS, and MSD	A
		Precision	≤20% RPD	MS and MSD	A

QAPP WORKSHEET #12

Measurement Performance Criteria Table 12-20

Matrix	Water
Analytical Group	Cyanide
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP 1, SOP 2, SOP 3, SOP 4, SOP 5, SOP 10, SOP 13, SOP 15, SOP 16, SOP 19, SOP 28, SOP 31, SOP 39, SOP 43, SOP 47, SOP 48, SOP 59 (see QAPP Worksheet #21)	SW9012B / 100.0004	Completeness	95%	NA	S&A
		Precision	≤50% RPD	Field duplicate	S&A
		Accuracy/bias/ contamination	<½ of the LOQ	Field blanks, laboratory reagent blank, or method blank	A
		Accuracy/bias/ contamination	< LOD	Blanks (ICB and CCB)	A
		Accuracy/bias	85-115%R	ICV/CCV	A
		Accuracy	80-120%R	LCS , MS, and MSD	A
		Precision	≤20% RPD	MS and MSD	A

QAPP WORKSHEET #13

Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Organization, Data Types, Data Generation/Collection Dates	Data Quality Issues	How Data Will Be Used/Limitations on Data Use
All Sites				
Geographic Information System	Niagara Falls ARS, updated by various contractors, 2012	Uploads from multiple Air Force sources including Civil Engineering files etc.	None	Base map creation
Well Maintenance Database	Niagara Falls ARS, updated by various contractors, 2012	Multiple contractors submitted data for well construction, survey data, water quality , analytical results	None	General information regarding project areas used to develop project background
DS001 – Site 14, AFRES Haz Waste Drum Storage				
Document	USAF, Decision Document AFRES Hazardous Waste Storage Area (Site SS01), 1990	Ecology and Environment Inc., Swipe samples and composite soil, 1989	No basis for comparison between swipe samples and NYSDEC SCOs. Composite VOC soil samples collected which is contrary to DER-10 guidance. No analysis for metals and SVOC was performed.	General information regarding potential site contaminants; data will assist in developing project background.
DS003 – Site 12, Bldg 850, Drum Storage				
Document	USAF, Decision Document Building 850 Drum Storage Area (SS03), 1991	Science Applications International Corp, soil, surface water, and sediment samples, 1986	None	Information regarding potential site contaminants; data will assist in developing project background.
ST009 – Site 4 BX MOGAS Leak				
Document	Ecology and Environment Inc., No Further Response Action Planned Decision Document – IRP Site 4 BX Gas Station MOGAS Tank Leak, 1999	Multiple contractors sampling soil and groundwater, late 1980s through 1990s	None	Information regarding potential site contaminants; data will assist in developing project background.

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Organization, Data Types, Data Generation/Collection Dates	Data Quality Issues	How Data Will Be Used/Limitations on Data Use
ST011 – Site 1, JP-4 Pipeline Leak				
Document	Ecology and Environment Inc., Decision Document IRP Site 1 Building 600 JP-4 Pipeline Leak, 1999	Multiple contractors sampling soil and groundwater, late 1980s through 1990s	None	Information regarding potential site contaminants; data will assist in developing project background.
TU 956 – UST 304				
Document	Ecology and Environment, Inc., Soil Sampling and Analysis for Removed UST Sites Building 304, 306, and 308, 1996	Ecology and Environment Inc., provided soil analysis data	Soil samples submitted for TCLP analysis which does not provide basis for comparison to NYSDEC SCOs	Information regarding potential site contaminants; data will assist in developing project background.
TU962 – UST 600				
Document	Ecology and Environment Inc., Closure and Site Assessment Report Underground Storage Tank Removal North of Bldg. 600, 1998	Ecology and Environment Inc., provided soil analysis data and tank removal report	Soil samples submitted for TCLP analysis which does not provide basis for comparison to NYSDEC SCOs	Information regarding potential site contaminants; data will assist in developing project background.

QAPP WORKSHEETS #14 AND 16

Project Tasks and Schedule

This worksheet lists the project tasks and describes the procedures to be followed for activities to be performed in support of the UU characterizations at Sites DS001, DS003, ST009, ST011, TU956, and TU962. The sampling design, strategy, and sequencing are further addressed in Worksheet #17.

All field activities will be coordinated with the 914th MSG/CSV and are anticipated to occur along the same schedule for all sites. The table below outlines the activities/tasks that will be performed and details the schedule under which they will be performed.

Activity	Responsible Party	Planned Start Date	Planned Completion Date	Deliverable(s)	Deliverable Due Date
Planning	EA	4/23/13 (Draft) 6/20/13 (Draft Final) 8/6/13 (Final)	5/21/13 (Draft) 6/26/13 (Draft Final) 8/12/13 (Final)	QAPP	5/21/13 (Draft) 6/26/13 (Draft Final) 8/12/13 (Final)
Mobilization and Site Preparation	EA	8/19/13	9/3/13	None	NA
Field Activities	EA	9/4/13	9/10/13	None	NA
Data Analysis and Validation	Laboratory And Data Validator	9/4/13	10/1/13	None	NA
UU Characterization Report	EA	9/25/13 (Draft) 11/15/13 (Draft Final) 1/3/14 (Final)	10/17/13 (Draft) 11/21/13 (Draft Final) 1/9/14 (Final)	Report	10/17/13 (Draft) 11/21/13 (Draft Final) 1/9/14 (Final)

The primary on-site and off-site tasks to be completed at all six sites under this project and the responsible organizations are listed below. The project schedule detailing the specific tasks, and planned start and end dates is included after these task lists.

Mobilization and Site Preparation Tasks – EA

- Obtain necessary access, escorts, and conduct notifications for field personnel and drilling or other field subcontractors, including:
  - Inspect proposed locations to refine sampling locations as needed.
  - Obtain necessary base access for field personnel.
  - Notify Niagara Falls ARS prior to mobilizing equipment and field personnel to the base.
  - Obtain utility clearance for drilling locations.
  - Determine staging areas for equipment if necessary
  - Order sample bottles and field monitoring equipment.
  - Coordinate with subcontractors, including drillers, laboratories, and surveyors.

Field Activities – EA, Subcontractors

Soil Sample Collection

1. Collect soil samples using a Geoprobe® advancement for field screening, lithology logging, and soil sample collection.

2. Final determination of sampling locations will depend on site access and subsurface utility issues.

3. If any unexpected construction debris is encountered, cease all site work, notify the Niagara Falls Point of Contact (POC), and re-evaluate the site for additional sampling locations.

Decontamination

1. Decontaminate equipment prior to starting work on the first sampling location; decontaminate all downhole and sampling equipment between each drilling location/sample.

Groundwater Sampling

1. Collect groundwater using direct push sample techniques (e.g., the Geoprobe® SP-22 groundwater sampler or similar).

Surveying

1. Following completion of the soil borings, a GPS survey of the locations will be performed.

Management of Investigative-Derived Waste (IDW)

1. Contain soil and water IDW appropriately, if required following discussion with NFARS personnel, until it is characterized for disposal.

Demobilization

1. Following completion of field activities, demobilize equipment and clean each work area and return it to its previous condition to the extent possible.

Analysis – Laboratory

The analytical laboratory will process and analyze samples according to the sample chain-of-custody documents, and the requirements of the QAPP.

QC – EA, Laboratory

1. Implement SOPs for field and laboratory activities.
2. Implement QC Plan (requirement described in this QAPP).
3. Collect QC samples as described in this QAPP.

Data Management – EA, Laboratory, Data Validator

1. The analytical laboratory will prepare an analytical electronic data deliverable in ERPIMS-compatible format.

2. EA will review and prepare applicable databases, including field data.

3. A third party data validator will review and validate analytical data.

4. EA will submit validated electronic data to the AFCEC ERPIMS database , Niagara Falls ARS database and the NYSDEC EQUIS database

5. EA will tabulate data for report.

Documentation and Records – EA

EA personnel will document field information in dedicated field log books and on appropriate forms according to the SOPs (Worksheet #21). EA will gather documentation of all field work, and data collection and generation activities; review it for completeness, include it in reports as warranted, and place it in a central project repository.

Assessment/Audit – EA

Assessment and oversight tasks are detailed in Worksheets #31, 32, and 33 of this QAPP. No external project-specific laboratory audits are planned by EA. The laboratory is periodically audited through the DoD ELAP as part of their accreditation process.

Data Review – EA

Data review tasks are detailed in Worksheets #34, 35, 36, and 37 of this QAPP.

Data Interpretation and Reporting - EA

The UU characterization report will include the results of the investigations, data assessments and DUSRs, and supporting field documentation and maps. Using these data, the report will present a comparison of analytical results to unrestricted use soil levels and Class GA groundwater standards. In addition to summarizing analytical results the report will also include a fish and wildlife resource impact analysis if required (DER-10 Appendix 3C).

A draft document will be prepared for internal review by AFCEC and Niagara Falls ARS; a draft final document will be prepared for review by the NYSDEC; and a final document will be prepared for review and approval by the Air Force and the NYSDEC. Review comments will be addressed and incorporated into each document accordingly.

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QAPP WORKSHEET #15

Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

The potential analyte groups, potentially applicable screening levels, QAPP reporting limits, and achievable laboratory detection limits for matrices of concern at Niagara Falls ARS are presented in the following tables:

- Table 15-1, Reference Limits for Soil
- Table 15-2, Reference Limits for Groundwater

These tables detail the potential analytical groups, analytical methods, and concentration levels for each compound for which soil and groundwater samples may be analyzed at Niagara Falls ARS.

NOTE: The matrix effects or necessary dilutions may affect the laboratory limits actually reported for project samples.

Definitions for the laboratory quantitation limits are provided in Worksheet #37. Quantitative concentration results within specified limits of precision and bias can only be achieved at or above the LOQ; however, the analytical laboratories may identify analytes between the detection limit (DL) and the LOQ. In these instances, the laboratories will report concentration values between the DL and LOQ as estimated values. The laboratory will report nondetectable values as less than the LOD.

LOQs, LOD, and DLs are presented in Tables 15-1 and 15-2 for analytes to be reported by the offsite laboratory.

Specific analytical methods for each site included in this QAPP are presented in QAPP Worksheet #18.

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TABLE 15-1

Reference Limits for Soil

Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Volatile Organic Compounds									
Acetone	SW8260C	67-64-1	mg/kg	0.05	0.005	0.005	0.004	0.0016	Spectrum Analytical, Inc.
Benzene	SW8260C	71-43-2	mg/kg	0.06	0.006	0.005	0.002	0.0006	Spectrum Analytical, Inc.
Bromochloromethane	SW8260C	74-97-5	mg/kg	NS	0.005	0.005	0.002	0.0008	Spectrum Analytical, Inc.
Bromodichloromethane	SW8260C	75-27-4	mg/kg	NS	0.005	0.005	0.002	0.0010	Spectrum Analytical, Inc.
Bromoform	SW8260C	75-25-2	mg/kg	NS	0.005	0.005	0.002	0.0020	Spectrum Analytical, Inc.
Bromomethane (Methyl bromide)	SW8260C	74-83-9	mg/kg	NS	0.005	0.005	0.002	0.0011	Spectrum Analytical, Inc.
2-Butanone (Methyl ethyl ketone)	SW8260C	78-93-3	mg/kg	0.12	0.012	0.005	0.004	0.0020	Spectrum Analytical, Inc.
n-Butylbenzene	SW8260C	104-51-8	mg/kg	12	1.2	0.005	0.002	0.0007	Spectrum Analytical, Inc.
sec-Butylbenzene	SW8260C	135-98-8	mg/kg	11	1.1	0.005	0.002	0.0006	Spectrum Analytical, Inc.
t-Butylbenzene	SW8260C	98-06-6	mg/kg	5.9	0.590	0.005	0.002	0.0005	Spectrum Analytical, Inc.
Carbon disulfide	SW8260C	75-15-0	mg/kg	NS	0.005	0.005	0.002	0.0003	Spectrum Analytical, Inc.
Carbon tetrachloride	SW8260C	56-23-5	mg/kg	0.76	0.076	0.005	0.002	0.0003	Spectrum Analytical, Inc.
Chlorobenzene	SW8260C	108-90-7	mg/kg	1.1	0.110	0.005	0.002	0.0005	Spectrum Analytical, Inc.
Chloroethane (Ethyl chloride)	SW8260C	75-00-3	mg/kg	NS	0.005	0.005	0.002	0.0010	Spectrum Analytical, Inc.
Chloroform	SW8260C	67-66-3	mg/kg	0.37	0.037	0.005	0.002	0.0006	Spectrum Analytical, Inc.
Chloromethane (Methyl chloride)	SW8260C	74-87-3	mg/kg	NS	0.005	0.005	0.002	0.0008	Spectrum Analytical, Inc.
Cyclohexane	SW8260C	110-82-7	mg/kg	NS	0.005	0.005	0.002	0.0017	Spectrum Analytical, Inc.
1,2-Dibromo-3-chloropropane (DBCP)	SW8260C	96-12-8	mg/kg	NS	0.005	0.005	0.002	0.0013	Spectrum Analytical, Inc.
Dibromochloromethane (Chlorodibromomethane)	SW8260C	124-48-1	mg/kg	NS	0.005	0.005	0.002	0.0007	Spectrum Analytical, Inc.
1,2-Dibromoethane (Ethylene dibromide [EDB])	SW8260C	106-93-4	mg/kg	NS	0.005	0.005	0.002	0.0007	Spectrum Analytical, Inc.
1,2-Dichlorobenzene	SW8260C	95-50-1	mg/kg	1.1	0.110	0.005	0.002	0.0006	Spectrum Analytical, Inc.
1,3-Dichlorobenzene	SW8260C	541-73-1	mg/kg	2.4	0.240	0.005	0.002	0.0007	Spectrum Analytical, Inc.
1,4-Dichlorobenzene	SW8260C	106-46-7	mg/kg	1.8	0.180	0.005	0.002	0.0008	Spectrum Analytical, Inc.
Dichlorodifluoromethane	SW8260C	75-71-8	mg/kg	NS	0.005	0.005	0.002	0.0010	Spectrum Analytical, Inc.
1,1-Dichloroethane	SW8260C	75-34-3	mg/kg	0.27	0.027	0.005	0.002	0.0007	Spectrum Analytical, Inc.
1,2-Dichloroethane	SW8260C	107-06-2	mg/kg	0.02	0.007	0.005	0.002	0.0005	Spectrum Analytical, Inc.
1,1-Dichloroethene	SW8260C	75-35-4	mg/kg	0.33	0.033	0.005	0.002	0.0010	Spectrum Analytical, Inc.
1,2-Dichloroethene (cis)	SW8260C	156-59-2	mg/kg	0.25	0.025	0.005	0.002	0.0008	Spectrum Analytical, Inc.
1,2-Dichloroethene (trans)	SW8260C	156-60-5	mg/kg	0.19	0.019	0.005	0.002	0.0005	Spectrum Analytical, Inc.
1,2-Dichloropropane	SW8260C	78-87-5	mg/kg	NS	0.005	0.005	0.002	0.0007	Spectrum Analytical, Inc.
1,3-Dichloropropene (cis)	SW8260C	10061-01-5	mg/kg	NS	0.005	0.005	0.002	0.0007	Spectrum Analytical, Inc.
1,3-Dichloropropene (trans)	SW8260C	10061-02-6	mg/kg	NS	0.005	0.005	0.002	0.0007	Spectrum Analytical, Inc.
1,4-Dioxane	SW8270D SIM	123-91-1	mg/kg	0.1	0.033	0.0033	NA	0.0033	Spectrum Analytical, Inc.
Ethylbenzene	SW8260C	100-41-4	mg/kg	1	0.100	0.005	0.002	0.0005	Spectrum Analytical, Inc.
2-Hexanone	SW8260C	591-78-6	mg/kg	NS	0.005	0.005	0.004	0.0008	Spectrum Analytical, Inc.

1. NYSDEC, Unrestricted Use Soil Cleanup Objectives, 6 NYCRR Part 375-6.8 (effective 14 December 2006).
2. Analytes with NS for PAL will have the QAPP RL set at the achievable laboratory LOQ.
3. QAPP RLs are set to one-tenth the PAL, if achievable. If not achievable the QAPP RLs are set to at least one-third the PAL.
4. The laboratory achievable LOQs that are higher than the corresponding QAPP RLs are shown in **bold**.

NOTE: CAS = Chemical Abstract Service
PAL = Project Action Limit

TABLE 15-1

Reference Limits for Soil

Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Isopropylbenzene (Cumene)	SW8260C	98-82-8	mg/kg	NS	0.005	0.005	0.002	0.0006	Spectrum Analytical, Inc.
Methyl acetate	SW8260C	79-20-9	mg/kg	NS	0.005	0.005	0.002	0.0014	Spectrum Analytical, Inc.
4-Methyl-2-pentanone (Methyl isobutyl ketone)	SW8260C	108-10-1	mg/kg	NS	0.005	0.005	0.004	0.0007	Spectrum Analytical, Inc.
Methylcyclohexane	SW8260C	108-78-2	mg/kg	NS	0.005	0.005	0.002	0.0018	Spectrum Analytical, Inc.
Methylene chloride	SW8260C	75-09-2	mg/kg	0.05	0.005	0.005	0.002	0.0013	Spectrum Analytical, Inc.
Methyl-tertiary-butyl ether	SW8260C	1634-04-4	mg/kg	0.93	0.093	0.005	0.002	0.0006	Spectrum Analytical, Inc.
n-Propylbenzene	SW8260C	103-65-1	mg/kg	3.9	0.390	0.005	0.002	0.0004	Spectrum Analytical, Inc.
Styrene	SW8260C	100-42-5	mg/kg	NS	0.005	0.005	0.002	0.0005	Spectrum Analytical, Inc.
1,1,2,2-Tetrachloroethane	SW8260C	79-34-5	mg/kg	NS	0.005	0.005	0.002	0.0007	Spectrum Analytical, Inc.
Tetrachloroethene	SW8260C	127-18-4	mg/kg	1.3	0.130	0.005	0.002	0.0006	Spectrum Analytical, Inc.
Toluene	SW8260C	108-88-3	mg/kg	0.7	0.070	0.005	0.002	0.0005	Spectrum Analytical, Inc.
1,2,3-Trichlorobenzene	SW8260C	87-61-6	mg/kg	NS	0.005	0.005	0.002	0.0006	Spectrum Analytical, Inc.
1,2,4-Trichlorobenzene	SW8260C	120-82-1	mg/kg	NS	0.005	0.005	0.002	0.0006	Spectrum Analytical, Inc.
1,1,1-Trichloroethane	SW8260C	71-55-6	mg/kg	0.68	0.068	0.005	0.002	0.0005	Spectrum Analytical, Inc.
1,1,2-Trichloroethane	SW8260C	79-00-5	mg/kg	NS	0.005	0.005	0.002	0.0005	Spectrum Analytical, Inc.
1,1,2-Trichloro-1,2,2-trifluoroethane	SW8260C	76-13-1	mg/kg	NS	0.005	0.005	0.004	0.0030	Spectrum Analytical, Inc.
Trichloroethene	SW8260C	79-01-6	mg/kg	0.47	0.047	0.005	0.002	0.0006	Spectrum Analytical, Inc.
Trichlorofluoromethane	SW8260C	75-69-4	mg/kg	NS	0.005	0.005	0.002	0.0004	Spectrum Analytical, Inc.
1,2,4-Trimethylbenzene	SW8260C	95-63-6	mg/kg	3.6	0.360	0.005	0.002	0.0006	Spectrum Analytical, Inc.
1,3,5-Trimethylbenzene	SW8260C	108-67-8	mg/kg	8.4	0.840	0.005	0.002	0.0006	Spectrum Analytical, Inc.
Vinyl chloride	SW8260C	75-01-4	mg/kg	0.02	0.007	0.005	0.002	0.0006	Spectrum Analytical, Inc.
m- and p-Xylenes	SW8260C	179601-23-1	mg/kg	NS	0.005	0.005	0.004	0.0016	Spectrum Analytical, Inc.
o-Xylene	SW8260C	95-47-6	mg/kg	NS	0.005	0.005	0.002	0.0005	Spectrum Analytical, Inc.
Xylenes (mixed)	SW8260C	1330-20-7	mg/kg	0.26	0.026	0.005	0.002	0.0005	Spectrum Analytical, Inc.

TABLE 15-1
Reference Limits for Soil
Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Semivolatile Organic Compounds									
Acetophenone	SW8270D	98-86-2	mg/kg	NS	0.330	0.330	0.133	0.031	Spectrum Analytical, Inc.
Atrazine	SW8270D	1912-24-9	mg/kg	NS	0.330	0.330	0.133	0.047	Spectrum Analytical, Inc.
Benzaldehyde	SW8270D	100-52-7	mg/kg	NS	0.330	0.330	0.133	0.044	Spectrum Analytical, Inc.
Bis(2-chloroethoxy)methane	SW8270D	111-91-1	mg/kg	NS	0.330	0.330	0.133	0.039	Spectrum Analytical, Inc.
Bis(2-chloroethyl)ether	SW8270D	111-44-4	mg/kg	NS	0.330	0.330	0.133	0.042	Spectrum Analytical, Inc.
Bis(2-ethylhexyl) phthalate	SW8270D	117-81-7	mg/kg	NS	0.330	0.330	0.133	0.029	Spectrum Analytical, Inc.
1,1-Biphenyl	SW8270D	92-52-4	mg/kg	NS	0.330	0.330	0.133	0.042	Spectrum Analytical, Inc.
4-Bromophenyl phenyl ether	SW8270D	101-55-3	mg/kg	NS	0.330	0.330	0.133	0.032	Spectrum Analytical, Inc.
Butyl benzyl phthalate	SW8270D	85-68-7	mg/kg	NS	0.330	0.330	0.133	0.026	Spectrum Analytical, Inc.
Caprolactam	SW8270D	105-60-2	mg/kg	NS	0.330	0.330	0.133	0.021	Spectrum Analytical, Inc.
Carbazole	SW8270D	86-74-8	mg/kg	NS	0.330	0.330	0.133	0.028	Spectrum Analytical, Inc.
4-Chloro-3-methylphenol	SW8270D	59-50-7	mg/kg	NS	0.330	0.330	0.133	0.026	Spectrum Analytical, Inc.
4-Chloroaniline	SW8270D	106-47-8	mg/kg	NS	0.330	0.330	0.133	0.024	Spectrum Analytical, Inc.
2-Chloronaphthalene	SW8270D	91-58-7	mg/kg	NS	0.330	0.330	0.133	0.038	Spectrum Analytical, Inc.
2-Chlorophenol	SW8270D	95-57-8	mg/kg	NS	0.330	0.330	0.133	0.041	Spectrum Analytical, Inc.
4-Chlorophenyl phenyl ether	SW8270D	7005-72-3	mg/kg	NS	0.330	0.330	0.133	0.040	Spectrum Analytical, Inc.
Dibenzofuran	SW8270D	132-64-9	mg/kg	7	0.700	0.330	0.133	0.036	Spectrum Analytical, Inc.
3,3-Dichlorobenzidine	SW8270D	91-94-1	mg/kg	NS	0.330	0.330	0.133	0.035	Spectrum Analytical, Inc.
2,4-Dichlorophenol	SW8270D	120-83-2	mg/kg	NS	0.330	0.330	0.133	0.038	Spectrum Analytical, Inc.
Diethyl phthalate	SW8270D	84-66-2	mg/kg	NS	0.330	0.330	0.133	0.024	Spectrum Analytical, Inc.
2,4-Dimethylphenol	SW8270D	105-67-9	mg/kg	NS	0.330	0.330	0.133	0.036	Spectrum Analytical, Inc.
Dimethyl phthalate	SW8270D	131-11-3	mg/kg	NS	0.330	0.330	0.133	0.030	Spectrum Analytical, Inc.
4,6-Dinitro-2-methylphenol	SW8270D	534-52-1	mg/kg	NS	0.670	0.670	0.133	0.025	Spectrum Analytical, Inc.
2,4-Dinitrophenol	SW8270D	51-28-5	mg/kg	NS	0.670	0.670	0.330	0.180	Spectrum Analytical, Inc.
2,4-Dinitrotoluene	SW8270D	121-14-2	mg/kg	NS	0.330	0.330	0.133	0.023	Spectrum Analytical, Inc.
2,6-Dinitrotoluene	SW8270D	606-20-2	mg/kg	NS	0.330	0.330	0.133	0.028	Spectrum Analytical, Inc.
Di-n-butyl phthalate	SW8270D	84-74-2	mg/kg	NS	0.330	0.330	0.133	0.028	Spectrum Analytical, Inc.
Di-n-octyl phthalate	SW8270D	117-84-0	mg/kg	NS	0.330	0.330	0.133	0.028	Spectrum Analytical, Inc.
Hexachlorobenzene	SW8270D	118-74-1	mg/kg	0.33	0.110	**0.330**	0.133	0.032	Spectrum Analytical, Inc.

TABLE 15-1
Reference Limits for Soil
Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Hexachlorobutadiene	SW8270D	87-68-3	mg/kg	NS	0.330	0.330	0.133	0.045	Spectrum Analytical, Inc.
Hexachlorocyclopentadiene	SW8270D	77-47-4	mg/kg	NS	0.330	0.330	0.330	0.096	Spectrum Analytical, Inc.
Hexachloroethane	SW8270D	67-72-1	mg/kg	NS	0.330	0.330	0.133	0.035	Spectrum Analytical, Inc.
Isophorone	SW8270D	78-59-1	mg/kg	NS	0.330	0.330	0.133	0.034	Spectrum Analytical, Inc.
2-Methylphenol	SW8270D	95-48-7	mg/kg	0.33	0.110	**0.330**	0.133	0.038	Spectrum Analytical, Inc.
3- and 4-Methylphenol	SW8270D	NA	mg/kg	0.33[5]	0.110	**0.330**	0.133	0.035	Spectrum Analytical, Inc.
2-Nitroaniline	SW8270D	88-74-4	mg/kg	NS	0.670	0.670	0.133	0.021	Spectrum Analytical, Inc.
3-Nitroaniline	SW8270D	99-09-2	mg/kg	NS	0.670	0.670	0.133	0.024	Spectrum Analytical, Inc.
4-Nitroaniline	SW8270D	100-01-6	mg/kg	NS	0.670	0.670	0.133	0.025	Spectrum Analytical, Inc.
Nitrobenzene	SW8270D	98-95-3	mg/kg	NS	0.330	0.330	0.133	0.038	Spectrum Analytical, Inc.
2-Nitrophenol	SW8270D	88-75-5	mg/kg	NS	0.330	0.330	0.133	0.036	Spectrum Analytical, Inc.
4-Nitrophenol	SW8270D	100-02-7	mg/kg	NS	0.670	0.670	0.133	0.022	Spectrum Analytical, Inc.
N-Nitroso-di-n-propylamine	SW8270D	621-64-7	mg/kg	NS	0.330	0.330	0.133	0.032	Spectrum Analytical, Inc.
N-Nitrosodiphenylamine	SW8270D	86-30-6	mg/kg	NS	0.330	0.330	0.133	0.029	Spectrum Analytical, Inc.
2,2'-Oxybis (1-Chloropropane)	SW8270D	108-60-1	mg/kg	NS	0.330	0.330	0.133	0.051	Spectrum Analytical, Inc.
Pentachlorophenol	SW8270D	87-86-5	mg/kg	0.8	0.267	**0.670**	0.330	0.140	Spectrum Analytical, Inc.
Phenol	SW8270D	108-95-2	mg/kg	0.33	0.110	**0.330**	0.133	0.037	Spectrum Analytical, Inc.
1,2,4,5-Tetrachlorobenzene	SW8270D	95-94-3	mg/kg	NS	0.330	0.330	0.133	0.059	Spectrum Analytical, Inc.
2,3,4,6-Tetrachlorophenol	SW8270D	58-90-2	mg/kg	NS	0.330	0.330	0.133	0.031	Spectrum Analytical, Inc.
2,4,5-Trichlorophenol	SW8270D	95-95-4	mg/kg	NS	0.670	0.670	0.133	0.037	Spectrum Analytical, Inc.
2,4,6-Trichlorophenol	SW8270D	88-06-2	mg/kg	NS	0.330	0.330	0.133	0.039	Spectrum Analytical, Inc.
Polycyclic Aromatic Hydrocarbons									
Anthracene	SW8270D SIM	120-12-7	mg/kg	100	10	0.003	0.003	0.001	Spectrum Analytical, Inc.
Pyrene	SW8270D SIM	129-00-0	mg/kg	100	10	0.003	0.003	0.001	Spectrum Analytical, Inc.
Benzo(g,h,i)perylene	SW8270D SIM	191-24-2	mg/kg	100	10	0.003	0.003	0.001	Spectrum Analytical, Inc.
Indeno(1,2,3-cd)pyrene	SW8270D SIM	193-39-5	mg/kg	0.5	0.050	0.003	0.003	0.001	Spectrum Analytical, Inc.
Benzo(b)fluoranthene	SW8270D SIM	205-99-2	mg/kg	1	0.100	0.003	0.003	0.002	Spectrum Analytical, Inc.
Fluoranthene	SW8270D SIM	206-44-0	mg/kg	100	10	0.003	0.003	0.002	Spectrum Analytical, Inc.
Benzo(k)fluoranthene	SW8270D SIM	207-08-9	mg/kg	0.8	0.080	0.003	0.003	0.001	Spectrum Analytical, Inc.
Acenaphthylene	SW8270D SIM	208-96-8	mg/kg	100	10	0.003	0.003	0.001	Spectrum Analytical, Inc.
Chrysene	SW8270D SIM	218-01-9	mg/kg	1	0.100	0.003	0.003	0.002	Spectrum Analytical, Inc.
Benzo(a)pyrene	SW8270D SIM	50-32-8	mg/kg	1	0.100	0.003	0.003	0.001	Spectrum Analytical, Inc.
Dibenz(a,h)anthracene	SW8270D SIM	53-70-3	mg/kg	0.33	0.033	0.003	0.003	0.001	Spectrum Analytical, Inc.
Benzo(a)anthracene	SW8270D SIM	56-55-3	mg/kg	1	0.100	0.003	0.003	0.001	Spectrum Analytical, Inc.
Acenaphthene	SW8270D SIM	83-32-9	mg/kg	20	2.0	0.003	0.003	0.001	Spectrum Analytical, Inc.
Phenanthrene	SW8270D SIM	85-01-8	mg/kg	100	10	0.003	0.003	0.001	Spectrum Analytical, Inc.
Fluorene	SW8270D SIM	86-73-7	mg/kg	30	3.0	0.003	0.003	0.001	Spectrum Analytical, Inc.
1-Methylnaphthalene	SW8270D SIM	90-12-0	mg/kg	NS	0.003	0.003	0.003	0.001	Spectrum Analytical, Inc.
Naphthalene	SW8270D SIM	91-20-3	mg/kg	12	1.2	0.003	0.003	0.001	Spectrum Analytical, Inc.
2-Methylnaphthalene	SW8270D SIM	91-57-6	mg/kg	NS	0.003	0.003	0.003	0.001	Spectrum Analytical, Inc.

5. Applies to the sum of 3- and 4-Methylphenol.

TABLE 15-1

Reference Limits for Soil

Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Pesticides									
Aldrin	SW8081B	309-00-2	mg/kg	0.005	0.002	**0.0017**	0.00043	0.000110	Spectrum Analytical, Inc.
alpha-BHC	SW8081B	319-84-6	mg/kg	0.02	0.002	0.0017	0.00043	0.000056	Spectrum Analytical, Inc.
beta-BHC	SW8081B	319-85-7	mg/kg	0.036	0.0036	0.0017	0.00043	0.000063	Spectrum Analytical, Inc.
delta-BHC	SW8081B	319-86-8	mg/kg	0.04	0.004	0.0017	0.00043	0.000120	Spectrum Analytical, Inc.
gamma-BHC (Lindane)	SW8081B	58-89-9	mg/kg	0.1	0.010	0.0017	0.00043	0.000055	Spectrum Analytical, Inc.
alpha-Chlordane	SW8081B	5103-71-9	mg/kg	0.094	0.0094	0.0017	0.00043	0.000087	Spectrum Analytical, Inc.
gamma-Chlordane	SW8081B	5103-74-2	mg/kg	NS	0.0017	0.0017	0.00043	0.000210	Spectrum Analytical, Inc.
4,4-DDD	SW8081B	72-54-8	mg/kg	0.0033	0.001	**0.0033**	0.00085	0.000220	Spectrum Analytical, Inc.
4,4-DDE	SW8081B	72-55-9	mg/kg	0.0033	0.001	**0.0033**	0.00085	0.000250	Spectrum Analytical, Inc.
4,4-DDT	SW8081B	50-29-3	mg/kg	0.0033	0.001	**0.0033**	0.00085	0.000330	Spectrum Analytical, Inc.
Dieldrin	SW8081B	60-57-1	mg/kg	0.005	0.002	**0.0033**	0.00085	0.000160	Spectrum Analytical, Inc.
Endosulfan I	SW8081B	959-98-8	mg/kg	2.4	0.240	0.0017	0.00043	0.000060	Spectrum Analytical, Inc.
Endosulfan II	SW8081B	33213-65-9	mg/kg	2.4	0.240	0.0033	0.00085	0.000150	Spectrum Analytical, Inc.
Endosulfan sulfate	SW8081B	1031-07-8	mg/kg	2.4	0.240	0.0033	0.00085	0.000130	Spectrum Analytical, Inc.
Endrin	SW8081B	72-20-8	mg/kg	0.014	0.005	0.0033	0.00085	0.000140	Spectrum Analytical, Inc.
Endrin aldehyde	SW8081B	7421-93-4	mg/kg	NS	0.0033	0.0033	0.00085	0.000230	Spectrum Analytical, Inc.
Endrin ketone	SW8081B	53494-70-5	mg/kg	NS	0.0033	0.0033	0.00085	0.000120	Spectrum Analytical, Inc.
Heptachlor	SW8081B	76-44-8	mg/kg	0.042	0.0042	0.0017	0.00043	0.000072	Spectrum Analytical, Inc.
Heptachlor epoxide	SW8081B	1024-57-3	mg/kg	NS	0.0017	0.0017	0.00043	0.000160	Spectrum Analytical, Inc.
Methoxychlor	SW8081B	72-43-5	mg/kg	NS	0.017	0.017	0.00425	0.000880	Spectrum Analytical, Inc.
Toxaphene	SW8081B	8001-35-2	mg/kg	NS	0.170	0.170	0.017	0.008800	Spectrum Analytical, Inc.
Herbicides									
2,4,5-TP (Silvex)	SW8151A	93-72-1	mg/kg	3.8	0.380	0.0067	0.00368	0.0067	Spectrum Analytical, Inc.
Polychlorinated Biphenyls									
Aroclor 1016	SW8082A	12674-11-2	mg/kg	NS	0.033	0.033	0.0083	0.0025	Spectrum Analytical, Inc.
Aroclor 1221	SW8082A	11104-28-2	mg/kg	NS	0.033	0.033	0.0166	0.0044	Spectrum Analytical, Inc.
Aroclor 1232	SW8082A	11141-16-5	mg/kg	NS	0.033	0.033	0.0083	0.0024	Spectrum Analytical, Inc.
Aroclor 1242	SW8082A	53469-21-9	mg/kg	NS	0.033	0.033	0.0083	0.0025	Spectrum Analytical, Inc.
Aroclor 1248	SW8082A	12672-79-6	mg/kg	NS	0.033	0.033	0.0083	0.0038	Spectrum Analytical, Inc.
Aroclor 1254	SW8082A	11097-69-1	mg/kg	NS	0.033	0.033	0.0083	0.0044	Spectrum Analytical, Inc.
Aroclor 1260	SW8082A	11096-82-5	mg/kg	NS	0.033	0.033	0.0083	0.0018	Spectrum Analytical, Inc.
Aroclor 1262	SW8082A	37324-23-5	mg/kg	NS	0.033	0.033	0.0083	0.0020	Spectrum Analytical, Inc.
Aroclor 1268	SW8082A	11100-14-4	mg/kg	NS	0.033	0.033	0.0083	0.0016	Spectrum Analytical, Inc.
Total PCBs	SW8082A	1336-36-3	mg/kg	0.1	0.010	NA	NA	NA	Spectrum Analytical, Inc.

TABLE 15-1

Reference Limits for Soil

Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Total Metals									
Aluminum	SW6010C	7429-90-5	mg/kg	NS	10	10	5.0	1.2	Spectrum Analytical, Inc.
Antimony	SW6010C	7440-36-0	mg/kg	NS	1.0	1.0	0.750	0.380	Spectrum Analytical, Inc.
Arsenic	SW6010C	7440-38-2	mg/kg	13	1.3	1.0	0.500	0.410	Spectrum Analytical, Inc.
Barium	SW6010C	7440-39-3	mg/kg	350	35	10.0	0.075	0.031	Spectrum Analytical, Inc.
Beryllium	SW6010C	7440-41-7	mg/kg	7.2	0.720	0.250	0.025	0.002	Spectrum Analytical, Inc.
Cadmium	SW6010C	7440-43-9	mg/kg	2.5	0.250	0.250	0.075	0.015	Spectrum Analytical, Inc.
Calcium	SW6010C	7440-70-2	mg/kg	NS	40	40	15	6.1	Spectrum Analytical, Inc.
Chromium	SW6010C	7440-47-3	mg/kg	NS	0.050	0.050	1.0	0.019	Spectrum Analytical, Inc.
Chromium, hexavalent	SW7196A	18540-29-9	mg/kg	1[6]	0.333	**4.0**	4.0	4.0	Spectrum Analytical, Inc.
Chromium, trivalent	NS	16065-83-1	mg/kg	30	10	4.0	NA	4.0	Spectrum Analytical, Inc.
Cobalt	SW6010C	7440-48-4	mg/kg	NS	2.5	2.5	0.050	0.044	Spectrum Analytical, Inc.
Copper	SW6010C	7440-50-8	mg/kg	50	5.0	1.5	0.250	0.110	Spectrum Analytical, Inc.
Cyanide	SW9012B	57-12-5	mg/kg	27	2.7	1.0	0.500	0.450	Spectrum Analytical, Inc.
Iron	SW6010C	7439-89-6	mg/kg	NS	10	10	2.5	1.5	Spectrum Analytical, Inc.
Lead	SW6010C	7439-92-1	mg/kg	63	6.3	0.500	0.250	0.170	Spectrum Analytical, Inc.
Magnesium	SW6010C	7439-95-4	mg/kg	NS	25	25	1.0	0.630	Spectrum Analytical, Inc.
Manganese	SW6010C	7439-96-5	mg/kg	1600	160	2.5	0.150	0.130	Spectrum Analytical, Inc.
Mercury	SW7471B	7439-97-6	mg/kg	0.18	0.060	**0.100**	NA	0.100	Spectrum Analytical, Inc.
Nickel	SW6010C	7440-02-0	mg/kg	30	3.0	2.5	0.050	0.043	Spectrum Analytical, Inc.
Potassium	SW6010C	7440-09-7	mg/kg	NS	50	50	5.0	3.4	Spectrum Analytical, Inc.
Selenium	SW6010C	7782-49-2	mg/kg	3.9	1.3	**1.5**	1.0	0.640	Spectrum Analytical, Inc.
Silver	SW6010C	7440-22-4	mg/kg	2	0.667	**1.5**	0.100	0.064	Spectrum Analytical, Inc.
Sodium	SW6010C	7440-23-5	mg/kg	NS	50	50	5.0	1.1	Spectrum Analytical, Inc.
Thallium	SW6010C	7440-28-0	mg/kg	NS	1.0	1.0	0.500	0.220	Spectrum Analytical, Inc.
Vanadium	SW6010C	7440-62-2	mg/kg	NS	2.5	2.5	0.100	0.060	Spectrum Analytical, Inc.
Zinc	SW6010C	7440-66-6	mg/kg	109	10.9	2.5	0.375	0.180	Spectrum Analytical, Inc.

6. The LOQ, LOD, and DL for Hexavalent Chromium are dependent on the percent moisture in the sample and may result in higher reported values.

TABLE 15-2

Reference Limits for Groundwater

Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Volatile Organic Compounds									
Acetone	SW8260C	67-64-1	µg/L	50	5.0	5.0	2.5	2.2	Spectrum Analytical, Inc.
Benzene	SW8260C	71-43-2	µg/L	1	0.333	**5.0**	0.500	0.330	Spectrum Analytical, Inc.
Bromochloromethane	SW8260C	74-97-5	µg/L	5	1.67	**5.0**	0.500	0.430	Spectrum Analytical, Inc.
Bromodichloromethane	SW8260C	75-27-4	µg/L	50	5.0	5.0	0.500	0.260	Spectrum Analytical, Inc.
Bromoform	SW8260C	75-25-2	µg/L	50	5.0	5.0	1.0	0.770	Spectrum Analytical, Inc.
Bromomethane (Methyl bromide)	SW8260C	74-83-9	µg/L	5	1.67	**5.0**	1.0	0.800	Spectrum Analytical, Inc.
2-Butanone (Methyl ethyl ketone)	SW8260C	78-93-3	µg/L	50	5.0	5.0	2.5	2.1	Spectrum Analytical, Inc.
n-Butylbenzene	SW8260C	104-51-8	µg/L	5	1.67	**5.0**	0.500	0.330	Spectrum Analytical, Inc.
sec-Butylbenzene	SW8260C	135-98-8	µg/L	5	1.67	**5.0**	0.500	0.280	Spectrum Analytical, Inc.
t-Butylbenzene	SW8260C	98-06-6	µg/L	5	1.67	**5.0**	0.500	0.370	Spectrum Analytical, Inc.
Carbon disulfide	SW8260C	75-15-0	µg/L	60	6.0	5.0	0.500	0.340	Spectrum Analytical, Inc.
Carbon tetrachloride	SW8260C	56-23-5	µg/L	5	1.67	**5.0**	1.0	0.540	Spectrum Analytical, Inc.
Chlorobenzene	SW8260C	108-90-7	µg/L	5	1.67	**5.0**	0.500	0.260	Spectrum Analytical, Inc.
Chloroethane (Ethyl chloride)	SW8260C	75-00-3	µg/L	5	1.67	**5.0**	0.500	0.480	Spectrum Analytical, Inc.
Chloroform	SW8260C	67-66-3	µg/L	7	2.33	**5.0**	0.500	0.330	Spectrum Analytical, Inc.
Chloromethane (Methyl chloride)	SW8260C	74-87-3	µg/L	5	1.67	**5.0**	0.500	0.260	Spectrum Analytical, Inc.
Cyclohexane	SW8260C	110-82-7	µg/L	NS	5.0	5.0	1.0	0.710	Spectrum Analytical, Inc.
1,2-Dibromo-3-chloropropane (DBCP)	SW8260C	96-12-8	µg/L	0.04	0.013	**5.0**	1.0	0.750	Spectrum Analytical, Inc.
Dibromochloromethane (Chlorodibromomethane)	SW8260C	124-48-1	µg/L	50	5.0	5.0	1.0	0.570	Spectrum Analytical, Inc.
1,2-Dibromoethane (Ethylene dibromide [EDB])	SW8260C	106-93-4	µg/L	0.0006	0.0002	**5.0**	0.500	0.500	Spectrum Analytical, Inc.
1,2-Dichlorobenzene	SW8260C	95-50-1	µg/L	3	1.0	**5.0**	0.500	0.330	Spectrum Analytical, Inc.
1,3-Dichlorobenzene	SW8260C	541-73-1	µg/L	3	1.0	**5.0**	0.500	0.290	Spectrum Analytical, Inc.
1,4-Dichlorobenzene	SW8260C	106-46-7	µg/L	3	1.0	**5.0**	0.500	0.400	Spectrum Analytical, Inc.
Dichlorodifluoromethane	SW8260C	75-71-8	µg/L	5	1.67	**5.0**	1.0	0.660	Spectrum Analytical, Inc.
1,1-Dichloroethane	SW8260C	75-34-3	µg/L	5	1.67	**5.0**	0.500	0.250	Spectrum Analytical, Inc.
1,2-Dichloroethane	SW8260C	107-06-2	µg/L	0.6	0.200	**5.0**	0.500	0.410	Spectrum Analytical, Inc.
1,1-Dichloroethene	SW8260C	75-35-4	µg/L	5	1.67	**5.0**	0.500	0.390	Spectrum Analytical, Inc.
1,2-Dichloroethene (cis)	SW8260C	156-59-2	µg/L	5	1.67	**5.0**	0.500	0.480	Spectrum Analytical, Inc.
1,2-Dichloroethene (trans)	SW8260C	156-60-5	µg/L	5	1.67	**5.0**	1.0	0.650	Spectrum Analytical, Inc.
1,2-Dichloropropane	SW8260C	78-87-5	µg/L	1	0.333	**5.0**	1.0	0.610	Spectrum Analytical, Inc.
1,3-Dichloropropene (cis and trans)	SW8260C	542-75-6	µg/L	0.4[5]	0.133	**5.0**	0.500	0.450	Spectrum Analytical, Inc.
1,4-Dioxane	SW8270D SIM	123-91-1	µg/L	NS	0.070	0.070	0.100	0.070	Spectrum Analytical, Inc.

1. Criteria based on NYSDEC Ambient Water Quality Standards and Guidance Values, Class GA Water, June 1998.
2. Analytes with NS for PAL will have the QAPP RL set at the achievable laboratory LOQ.
3. QAPP RLs are set to one-tenth the PAL, if achievable. If not achievable the QAPP RLs are set to at least one-third the PAL.
4. The laboratory achievable LOQs that are higher than the corresponding QAPP RLs are shown in **bold**.
5. Applies to the sum of cis- and trans-1,3-dichloropropene.

TABLE 15-2

Reference Limits for Groundwater

Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Ethylbenzene	SW8260C	100-41-4	µg/L	5	1.67	**5.0**	0.500	0.350	Spectrum Analytical, Inc.
2-Hexanone	SW8260C	591-78-6	µg/L	50	5.0	5.0	2.5	1.7	Spectrum Analytical, Inc.
Isopropylbenzene (Cumene)	SW8260C	98-82-8	µg/L	5	1.67	**5.0**	0.500	0.380	Spectrum Analytical, Inc.
Methyl acetate	SW8260C	79-20-9	µg/L	NS	5.0	5.0	1.0	0.290	Spectrum Analytical, Inc.
4-Methyl-2-pentanone (Methyl isobutyl ketone)	SW8260C	108-10-1	µg/L	NS	5.0	5.0	1.0	0.820	Spectrum Analytical, Inc.
Methylcyclohexane	SW8260C	108-78-2	µg/L	NS	5.0	5.0	1.0	0.760	Spectrum Analytical, Inc.
Methylene chloride	SW8260C	75-09-2	µg/L	5	1.67	**5.0**	0.500	0.410	Spectrum Analytical, Inc.
Methyl-tertiary-butyl ether	SW8260C	1634-04-4	µg/L	10	3.33	**5.0**	0.500	0.240	Spectrum Analytical, Inc.
n-Propylbenzene	SW8260C	103-65-1	µg/L	5	1.67	**5.0**	1.0	0.640	Spectrum Analytical, Inc.
Styrene	SW8260C	100-42-5	µg/L	5	1.67	**5.0**	0.500	0.500	Spectrum Analytical, Inc.
1,1,2,2-Tetrachloroethane	SW8260C	79-34-5	µg/L	5	1.67	**5.0**	0.500	0.420	Spectrum Analytical, Inc.
Tetrachloroethene (PCE)	SW8260C	127-18-4	µg/L	5	1.67	**5.0**	1.0	0.650	Spectrum Analytical, Inc.
Toluene	SW8260C	108-88-3	µg/L	5	1.67	**5.0**	0.500	0.320	Spectrum Analytical, Inc.
1,2,3-Trichlorobenzene	SW8260C	87-61-6	µg/L	5	1.67	**5.0**	0.500	0.330	Spectrum Analytical, Inc.
1,2,4-Trichlorobenzene	SW8260C	120-82-1	µg/L	5	1.67	**5.0**	0.500	0.260	Spectrum Analytical, Inc.
1,1,1-Trichloroethane	SW8260C	71-55-6	µg/L	5	1.67	**5.0**	0.500	0.500	Spectrum Analytical, Inc.
1,1,2-Trichloroethane	SW8260C	79-00-5	µg/L	1	0.333	**5.0**	1.0	0.380	Spectrum Analytical, Inc.
1,1,2-Trichlorotrifluoroethane	SW8260C	76-13-1	µg/L	5	1.67	**5.0**	1.0	0.820	Spectrum Analytical, Inc.
Trichloroethene (TCE)	SW8260C	79-01-6	µg/L	5	1.67	**5.0**	0.500	0.360	Spectrum Analytical, Inc.
Trichlorofluoromethane	SW8260C	75-69-4	µg/L	5	1.67	**5.0**	1.0	0.540	Spectrum Analytical, Inc.
1,2,4-Trimethylbenzene	SW8260C	95-63-6	µg/L	5	1.67	**5.0**	0.500	0.400	Spectrum Analytical, Inc.
1,3,5-Trimethylbenzene	SW8260C	108-67-8	µg/L	5	1.67	**5.0**	0.500	0.450	Spectrum Analytical, Inc.
Vinyl chloride	SW8260C	75-01-4	µg/L	2	0.667	**5.0**	0.500	0.500	Spectrum Analytical, Inc.
m- and p-Xylene	SW8260C	179601-23-1	µg/L	5	1.67	**5.0**	1.0	0.770	Spectrum Analytical, Inc.
o-Xylene	SW8260C	95-47-6	µg/L	5	1.67	**5.0**	0.500	0.360	Spectrum Analytical, Inc.
Xylenes (mixed)	SW8260C	1330-20-7	µg/L	NS	5.0	5.0	1.0	0.360	Spectrum Analytical, Inc.

TABLE 15-2

Reference Limits for Groundwater

Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Semivolatile Organic Compounds									
Acenaphthene	SW8270D	83-32-9	µg/L	20	6.67	**10**	2.0	0.650	Spectrum Analytical, Inc.
Acenaphthylene	SW8270D	208-96-8	µg/L	NS	10	10	2.0	0.420	Spectrum Analytical, Inc.
Acetophenone	SW8270D	98-86-2	µg/L	NS	10	10	2.0	0.510	Spectrum Analytical, Inc.
Atrazine	SW8270D	1912-24-9	µg/L	7.5	2.5	**10**	2.0	1.3	Spectrum Analytical, Inc.
Benzaldehyde	SW8270D	100-52-7	µg/L	NS	10	10	2.0	0.510	Spectrum Analytical, Inc.
Bis(2-chloroethoxy)methane	SW8270D	111-91-1	µg/L	5	1.67	**10**	2.0	1.1	Spectrum Analytical, Inc.
Bis(2-chloroethyl)ether	SW8270D	111-44-4	µg/L	1	0.333	**10**	2.0	0.750	Spectrum Analytical, Inc.
Bis(2-ethylhexyl) phthalate	SW8270D	117-81-7	µg/L	5	1.67	**10**	2.0	1.3	Spectrum Analytical, Inc.
1,1-Biphenyl	SW8270D	92-52-4	µg/L	5	1.67	**10**	2.0	0.650	Spectrum Analytical, Inc.
4-Bromophenyl phenyl ether	SW8270D	101-55-3	µg/L	NS	10	10	2.0	0.540	Spectrum Analytical, Inc.
Butyl benzyl phthalate	SW8270D	85-68-7	µg/L	50	16.7	10	2.0	0.320	Spectrum Analytical, Inc.
Caprolactam	SW8270D	105-60-2	µg/L	NS	10	10	5.0	1.1	Spectrum Analytical, Inc.
Carbazole	SW8270D	86-74-8	µg/L	NS	10	10	2.0	0.640	Spectrum Analytical, Inc.
4-Chloro-3-methylphenol	SW8270D	59-50-7	µg/L	NS	10	10	2.0	0.600	Spectrum Analytical, Inc.
4-Chloroaniline	SW8270D	106-47-8	µg/L	5	1.67	**10**	2.0	2.0	Spectrum Analytical, Inc.
2-Chloronaphthalene	SW8270D	91-58-7	µg/L	10	3.33	**10**	2.0	0.810	Spectrum Analytical, Inc.
2-Chlorophenol	SW8270D	95-57-8	µg/L	NS	10	10	2.0	0.610	Spectrum Analytical, Inc.
4-Chlorophenyl phenyl ether	SW8270D	7005-72-3	µg/L	NS	10	10	2.0	0.410	Spectrum Analytical, Inc.
Dibenzofuran	SW8270D	132-64-9	µg/L	NS	10	10	2.0	0.520	Spectrum Analytical, Inc.
3,3-Dichlorobenzidine	SW8270D	91-94-1	µg/L	5	1.67	**10**	10	1.7	Spectrum Analytical, Inc.
2,4-Dichlorophenol	SW8270D	120-83-2	µg/L	5	1.67	**10**	2.0	0.570	Spectrum Analytical, Inc.
Diethyl phthalate	SW8270D	84-66-2	µg/L	50	16.7	10	2.0	0.450	Spectrum Analytical, Inc.
2,4-Dimethylphenol	SW8270D	105-67-9	µg/L	50	16.7	10	2.0	1.80	Spectrum Analytical, Inc.
Dimethyl phthalate	SW8270D	131-11-3	µg/L	50	16.7	10	2.0	0.370	Spectrum Analytical, Inc.
4,6-Dinitro-2-methylphenol	SW8270D	534-52-1	µg/L	NS	20	20	2	0.790	Spectrum Analytical, Inc.
2,4-Dinitrophenol	SW8270D	51-28-5	µg/L	10	3.33	**20**	10	3.5	Spectrum Analytical, Inc.
2,4-Dinitrotoluene	SW8270D	121-14-2	µg/L	5	1.67	**10**	2.0	0.410	Spectrum Analytical, Inc.
2,6-Dinitrotoluene	SW8270D	606-20-2	µg/L	5	1.67	**10**	2.0	0.520	Spectrum Analytical, Inc.
Di-n-butyl phthalate	SW8270D	84-74-2	µg/L	50	16.7	10	2.0	0.480	Spectrum Analytical, Inc.
Di-n-octyl phthalate	SW8270D	117-84-0	µg/L	50	16.7	10	2.0	0.470	Spectrum Analytical, Inc.

TABLE 15-2

Reference Limits for Groundwater

Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Hexachlorobenzene	SW8270D	118-74-1	µg/L	0.04	0.013	**10**	2.0	0.440	Spectrum Analytical, Inc.
Hexachlorobutadiene	SW8270D	87-68-3	µg/L	0.5	0.167	**10**	2	0.750	Spectrum Analytical, Inc.
Hexachlorocyclopentadiene	SW8270D	77-47-4	µg/L	5	1.67	**10**	10	1.0	Spectrum Analytical, Inc.
Hexachloroethane	SW8270D	67-72-1	µg/L	5	1.67	**10**	2.0	0.550	Spectrum Analytical, Inc.
Isophorone	SW8270D	78-59-1	µg/L	50	16.7	10	2.0	0.470	Spectrum Analytical, Inc.
2-Methylphenol	SW8270D	95-48-7	µg/L	NS	10	10	2.0	0.960	Spectrum Analytical, Inc.
3- and 4-Methylphenol	SW8270D	NA	µg/L	NS	10	10	2.0	1.4	Spectrum Analytical, Inc.
2-Nitroaniline	SW8270D	88-74-4	µg/L	5	1.67	**20**	2.0	0.710	Spectrum Analytical, Inc.
3-Nitroaniline	SW8270D	99-09-2	µg/L	5	1.67	**20**	2.0	1.0	Spectrum Analytical, Inc.
4-Nitroaniline	SW8270D	100-01-6	µg/L	5	1.67	**20**	2.0	1.0	Spectrum Analytical, Inc.
Nitrobenzene	SW8270D	98-95-3	µg/L	0.4	0.133	**10**	10	1.6	Spectrum Analytical, Inc.
2-Nitrophenol	SW8270D	88-75-5	µg/L	NS	10	10	2.0	0.600	Spectrum Analytical, Inc.
4-Nitrophenol	SW8270D	100-02-7	µg/L	NS	20	20	2	0.530	Spectrum Analytical, Inc.
N-Nitroso-di-n-propylamine	SW8270D	621-64-7	µg/L	NS	10	10	2.0	0.630	Spectrum Analytical, Inc.
N-Nitrosodiphenylamine	SW8270D	86-30-6	µg/L	50	16.7	10	2.0	1.1	Spectrum Analytical, Inc.
2,2'-Oxybis (1-Chloropropane)	SW8270D	108-60-1	µg/L	5	1.67	**10**	2.0	0.780	Spectrum Analytical, Inc.
Pentachlorophenol	SW8270D	87-86-5	µg/L	NS	20	20	10	1.7	Spectrum Analytical, Inc.
Phenols (Total)	SW8270D	108-95-2	µg/L	1[6]	0.333	**10**	2.0	0.750	Spectrum Analytical, Inc.
1,2,4,5-Tetrachlorobenzene	SW8270D	95-94-3	µg/L	5	1.67	**10**	2.0	0.920	Spectrum Analytical, Inc.
2,3,4,6-Tetrachlorophenol	SW8270D	58-90-2	µg/L	NS	25	25	2.0	0.650	Spectrum Analytical, Inc.
2,4,5-Trichlorophenol	SW8270D	95-95-4	µg/L	NS	20	20	2.0	0.260	Spectrum Analytical, Inc.
2,4,6-Trichlorophenol	SW8270D	88-06-2	µg/L	NS	10	10	2.0	0.530	Spectrum Analytical, Inc.

6. Applies to the sum of phenolic compounds (including pentachlorophenol).

TABLE 15-2

Reference Limits for Groundwater

Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Polycyclic Aromatic Hydrocarbons									
Anthracene	SW8270D SIM	120-12-7	µg/L	50	5.0	0.100	0.100	0.017	Spectrum Analytical, Inc.
Pyrene	SW8270D SIM	129-00-0	µg/L	50	5.0	0.100	0.100	0.016	Spectrum Analytical, Inc.
Benzo(g,h,i)perylene	SW8270D SIM	191-24-2	µg/L	NS	0.100	0.100	0.100	0.021	Spectrum Analytical, Inc.
Indeno(1,2,3-cd)pyrene	SW8270D SIM	193-39-5	µg/L	0.002	0.0007	**0.100**	0.100	0.019	Spectrum Analytical, Inc.
Benzo(b)fluoranthene	SW8270D SIM	205-99-2	µg/L	0.002	0.0007	**0.100**	0.100	0.056	Spectrum Analytical, Inc.
Fluoranthene	SW8270D SIM	206-44-0	µg/L	50	5.0	0.100	0.100	0.019	Spectrum Analytical, Inc.
Benzo(k)fluoranthene	SW8270D SIM	207-08-9	µg/L	0.002	0.0007	**0.100**	0.100	0.020	Spectrum Analytical, Inc.
Acenaphthylene	SW8270D SIM	208-96-8	µg/L	NS	0.100	0.100	0.100	0.017	Spectrum Analytical, Inc.
Chrysene	SW8270D SIM	218-01-9	µg/L	0.002	0.0007	**0.100**	0.100	0.073	Spectrum Analytical, Inc.
Benzo(a)pyrene	SW8270D SIM	50-32-8	µg/L	ND	0.100	0.100	0.100	0.017	Spectrum Analytical, Inc.
Dibenz(a,h)anthracene	SW8270D SIM	53-70-3	µg/L	NS	0.100	0.100	0.100	0.018	Spectrum Analytical, Inc.
Benzo(a)anthracene	SW8270D SIM	56-55-3	µg/L	0.002	0.0007	**0.100**	0.100	0.042	Spectrum Analytical, Inc.
Acenaphthene	SW8270D SIM	83-32-9	µg/L	20	2.0	0.100	0.100	0.019	Spectrum Analytical, Inc.
Phenanthrene	SW8270D SIM	85-01-8	µg/L	50	5.0	0.100	0.100	0.019	Spectrum Analytical, Inc.
Fluorene	SW8270D SIM	86-73-7	µg/L	50	5.0	0.100	0.100	0.017	Spectrum Analytical, Inc.
1-Methylnaphthalene	SW8270D SIM	90-12-0	µg/L	NS	0.100	0.100	0.100	0.016	Spectrum Analytical, Inc.
Naphthalene	SW8270D SIM	91-20-3	µg/L	10	1.0	0.100	0.100	0.050	Spectrum Analytical, Inc.
2-Methylnaphthalene	SW8270D SIM	91-57-6	µg/L	NS	0.100	0.100	0.100	0.018	Spectrum Analytical, Inc.
Pesticides									
Aldrin	SW8081B	309-00-2	µg/L	ND	0.050	0.050	0.013	0.0043	Spectrum Analytical, Inc.
alpha-BHC	SW8081B	319-84-6	µg/L	0.01	0.003	**0.050**	0.013	0.0018	Spectrum Analytical, Inc.
beta-BHC	SW8081B	319-85-7	µg/L	0.04	0.013	**0.050**	0.013	0.002	Spectrum Analytical, Inc.
delta-BHC	SW8081B	319-86-8	µg/L	0.04	0.013	**0.050**	0.013	0.0027	Spectrum Analytical, Inc.
gamma-BHC (Lindane)	SW8081B	58-89-9	µg/L	0.05	0.017	**0.050**	0.013	0.0019	Spectrum Analytical, Inc.
alpha-Chlordane	SW8081B	5103-71-9	µg/L	NS	0.050	0.050	0.013	0.0024	Spectrum Analytical, Inc.
gamma-Chlordane	SW8081B	5103-74-2	µg/L	NS	0.050	0.050	0.013	0.0026	Spectrum Analytical, Inc.
4,4-DDD	SW8081B	72-54-8	µg/L	0.3	0.100	0.100	0.025	0.0064	Spectrum Analytical, Inc.
4,4-DDE	SW8081B	72-55-9	µg/L	0.2	0.067	**0.100**	0.025	0.0056	Spectrum Analytical, Inc.
4,4-DDT	SW8081B	50-29-3	µg/L	0.2	0.067	**0.100**	0.025	0.007	Spectrum Analytical, Inc.
Dieldrin	SW8081B	60-57-1	µg/L	0.004	0.001	**0.100**	0.025	0.0056	Spectrum Analytical, Inc.
Endosulfan I	SW8081B	959-98-8	µg/L	NS	0.050	0.050	0.013	0.0029	Spectrum Analytical, Inc.
Endosulfan II	SW8081B	33213-65-9	µg/L	NS	0.100	0.100	0.025	0.0031	Spectrum Analytical, Inc.
Endosulfan sulfate	SW8081B	1031-07-8	µg/L	NS	0.100	0.100	0.025	0.0045	Spectrum Analytical, Inc.
Endrin	SW8081B	72-20-8	µg/L	ND	0.100	0.100	0.025	0.0035	Spectrum Analytical, Inc.
Endrin aldehyde	SW8081B	7421-93-4	µg/L	5	0.500	0.100	0.025	0.015	Spectrum Analytical, Inc.
Endrin ketone	SW8081B	53494-70-5	µg/L	5	0.500	0.100	0.025	0.0046	Spectrum Analytical, Inc.
Heptachlor	SW8081B	76-44-8	µg/L	0.04	0.013	**0.050**	0.013	0.0039	Spectrum Analytical, Inc.
Heptachlor epoxide	SW8081B	1024-57-3	µg/L	0.03	0.010	**0.050**	0.013	0.0028	Spectrum Analytical, Inc.
Methoxychlor	SW8081B	72-43-5	µg/L	35	3.5	0.500	0.125	0.031	Spectrum Analytical, Inc.
Toxaphene	SW8081B	8001-35-2	µg/L	0.06	0.020	**5.0**	0.500	0.140	Spectrum Analytical, Inc.

TABLE 15-2

Reference Limits for Groundwater

Unrestricted Use Characterization Quality Assurance Project Plan For Sites DS001, DS003, ST009, ST011, TU956, and TU962

Analyte	Analytical Method	CAS	Units	PAL[1, 2]	QAPP RLs[3]	Achievable Laboratory Limits			Laboratory
						LOQ[4]	LOD	DL	
Polychlorinated biphenyls									
Aroclor 1016	SW8082A	12674-11-2	µg/L	NS	1.0	1.0	0.250	0.119	Spectrum Analytical, Inc.
Aroclor 1221	SW8082A	11104-28-2	µg/L	NS	1.0	1.0	0.500	0.095	Spectrum Analytical, Inc.
Aroclor 1232	SW8082A	11141-16-5	µg/L	NS	1.0	1.0	0.250	0.185	Spectrum Analytical, Inc.
Aroclor 1242	SW8082A	53469-21-9	µg/L	NS	1.0	1.0	0.250	0.030	Spectrum Analytical, Inc.
Aroclor 1248	SW8082A	12672-79-6	µg/L	NS	1.0	1.0	0.250	0.063	Spectrum Analytical, Inc.
Aroclor 1254	SW8082A	11097-69-1	µg/L	NS	1.0	1.0	0.250	0.204	Spectrum Analytical, Inc.
Aroclor 1260	SW8082A	11096-82-5	µg/L	NS	1.0	1.0	0.250	0.105	Spectrum Analytical, Inc.
Aroclor 1262	SW8082A	37324-23-5	µg/L	NS	1.0	1.0	0.250	0.042	Spectrum Analytical, Inc.
Aroclor 1268	SW8082A	11100-14-4	µg/L	NS	1.0	1.0	0.250	0.102	Spectrum Analytical, Inc.
Polychlorinated biphenyls	SW8082A	NA	µg/L	0.09[7]	0.009	NA	NA	NA	Spectrum Analytical, Inc.
Herbicides									
2,4,5-TP (Silvex)	SW8151A	93-72-1	µg/L	0.26	0.087	**0.100**	0.050	0.046	Spectrum Analytical, Inc.
Total Metals									
Aluminum	SW6020A	7429-90-5	µg/L	NS	20	20	6.75	2.9	Spectrum Analytical, Inc.
Antimony	SW6020A	7440-36-0	µg/L	3	1.0	**2.0**	0.200	0.200	Spectrum Analytical, Inc.
Arsenic	SW6020A	7440-38-2	µg/L	25	2.5	2.0	0.375	0.190	Spectrum Analytical, Inc.
Barium	SW6020A	7440-39-3	µg/L	1000	100	10	2.0	1.3	Spectrum Analytical, Inc.
Beryllium	SW6020A	7440-41-7	µg/L	3	1.0	1.0	0.150	0.072	Spectrum Analytical, Inc.
Cadmium	SW6020A	7440-43-9	µg/L	5	1.67	1.0	0.150	0.084	Spectrum Analytical, Inc.
Calcium	SW6020A	7440-70-2	µg/L	NS	500	500	37.5	24	Spectrum Analytical, Inc.
Chromium	SW6020A	7440-47-3	µg/L	50	5.0	2.0	0.250	0.160	Spectrum Analytical, Inc.
Chromium, hexavalent	SW3500 Cr+6 B	18540-29-9	µg/L	50	5.0	0.030	0.030	0.030	Spectrum Analytical, Inc.
Chromium, trivalent	NS	16065-83-1	µg/L	NS	0.030	0.030	NA	0.030	Spectrum Analytical, Inc.
Cobalt	SW6020A	7440-48-4	µg/L	NS	1	1.0	0.050	0.024	Spectrum Analytical, Inc.
Copper	SW6020A	7440-50-8	µg/L	200	20	2.0	0.375	0.230	Spectrum Analytical, Inc.
Cyanide	SW9012B	57-12-5	µg/L	200	20	20	10	7.5	Spectrum Analytical, Inc.
Iron	SW6020A	7439-89-6	µg/L	300	100	**200**	20	14	Spectrum Analytical, Inc.
Lead	SW6020A	7439-92-1	µg/L	25	2.5	1.0	0.150	0.068	Spectrum Analytical, Inc.
Magnesium	SW6020A	7439-95-4	µg/L	35000	3500	500	12	7.8	Spectrum Analytical, Inc.
Manganese	SW6020A	7439-96-5	µg/L	300	30	5.0	1.0	0.830	Spectrum Analytical, Inc.
Mercury	SW7470A	7439-97-6	µg/L	0.7	0.233	0.200	0.050	0.028	Spectrum Analytical, Inc.
Nickel	SW6020A	7440-02-0	µg/L	100	10	1.0	0.250	0.170	Spectrum Analytical, Inc.
Potassium	SW6020A	9/7/7440	µg/L	NS	500	500	20	14	Spectrum Analytical, Inc.
Selenium	SW6020A	7782-49-2	µg/L	10	3.33	**5.0**	0.250	0.150	Spectrum Analytical, Inc.
Silver	SW6020A	7440-22-4	µg/L	50	5.0	1.0	0.100	0.022	Spectrum Analytical, Inc.
Sodium	SW6020A	7440-23-5	µg/L	20000	2000	500	50	33	Spectrum Analytical, Inc.
Thallium	SW6020A	7440-28-0	µg/L	0.5	0.167	**1.0**	0.075	0.048	Spectrum Analytical, Inc.
Vanadium	SW6020A	7440-62-2	µg/L	NS	5	5.0	1.0	0.610	Spectrum Analytical, Inc.
Zinc	SW6020A	7440-66-6	µg/L	2000	200	5.0	1.0	0.730	Spectrum Analytical, Inc.

7. Applies to the sum of detected polychlorinated biphenyls.

QAPP WORKSHEET #17

Sample Design and Rationale

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach)

The rationale for choosing the sampling approach focused on obtaining the additional data necessary to evaluate whether current site concentrations in potentially affected media meet UU criteria. As described in Worksheet #11, while the COPCs associated with the subject sites are primarily petroleum-related VOCs and SVOCs (specifically BTEX constituents and PAHs), a subset of environmental samples need to be analyzed for a full suite of analytes. Demonstrating that potentially affected site media meet the UU criteria for a full suite of analytes will allow for SC without reliance on institutional or administrative controls (NYSDEC 2010a). The potentially affected medium at each of the sites is soil. Based on previous investigations, it is anticipated that potential groundwater impacts are limited to two of the six sites: ST011 (JP-4 Pipeline Leak) and ST009 (BX MOGAS Leak).

In general, sampling locations were selected to:

- Assess current conditions at locations where previous sampling results indicated exceedances of UU criteria
- Verify that current conditions meet UU criteria for a full suite of analytes for soil at all sites and groundwater at ST009 and ST011.

General Procedures

Field methodologies will be consistent with the SOPs listed in Worksheet #21 and included in Appendix B, as appropriate. Field activities will be conducted in accordance with Appendix C—Health and Safety Plan Addendum. Field activities will be documented in a field logbook as per SOPs 16 and 59. Photographs will also be taken (with prior approval of NFARS personnel) to document field activities, as appropriate.

Soil Sampling

Proposed soil boring locations are shown in Figures 11-1 through 11-5. During mobilization activities, soil boring locations will be staked in the field for utility clearance. Prior to the initiation of any subsurface soil sampling activities, utility clearance will be performed as per SOP 3. Based upon the presence and absence of visual contamination or subsurface debris, utility clearance, or field observations, soil boring locations may be shifted or stepped out toward target subsurface corridors in order to delineate the extent of site-related impacts. Locations will be agreed upon with the base personnel and the NYSDEC Project Manager. The field team leader will be in communication with the Site Project Manager as field decisions are made based upon subsurface conditions encountered.

A total of 39 soil borings will be completed using direct-push drilling techniques, as per SOP 47. Sampling locations were selected after reviewing previous investigation sampling locations, evaluating current knowledge of COC delineation, and determining data gaps. Target depths and rationale for soil borings at each site are summarized in the following table.

Site Name	Estimated Depth to Water (ft bgs)	Estimated Depth to Bedrock (ft bgs)	Target Boring Depth (ft bgs)	Rationale
DS001 (Site 14)	4-10	10	0-2	Evaluate surface soil around the perimeter of the former drum storage area based on: lack of documented spills or monitoring requirement. NYSDEC approved NFA. Additional sampling to verify that a surface release did not occur.
DS003 (Site 12)	4-10	10	0-2 and 6-8	Assess shallow overburden around the perimeter of the former drum storage area on a modified grid in associated unpaved areas. The target depth is the surface soil based on the lack of documented spills or monitoring requirements. This additional sampling is to verify that a surface release did not occur. One sample will also be collected from the soil/groundwater interface based on a historic detection of TCE at 6-8 ft bgs. NYSDEC approved NFA.
ST009 (Site 4)	7	12	12	Assess overburden to the top of bedrock based on documented gasoline release and past groundwater monitoring requirements. Locations are at the site boundaries and areas where historically higher COC concentrations were observed. This additional sampling is to verify that subsurface soil conditions meet UU criteria in the groundwater fluctuation zone.
ST011 (Site 1)	5	10	10	Assess overburden to top of bedrock, based on documented JP-4 release and past groundwater monitoring requirements. Locations are at the site boundaries and areas where historically higher COC concentrations were observed. This additional sampling is to verify that subsurface soil conditions meet UU criteria in the groundwater fluctuation zone.
TU956 (UST304)	4-10	10	10	Assess overburden to top of bedrock, based on tank installed with a pad installed on bedrock. Classified as “inactive” due to low concentrations detected after tank removal. One boring will be completed on each side of former tank excavation area. This additional sampling at the approximate depth of the former tank bottom (i.e., 6-8 ft.) is to verify that post-remedial action subsurface soil conditions meet UU criteria.
TU962 (UST600)	5	10	10	Assess overburden to top of bedrock, based on tank installed with a saddle on top of rock. Classified as “inactive” due to low concentrations detected during tank removal. One boring on each of three accessible sides and one boring through backfill of former tank excavation area. Additional sampling at the approximate depth of the former tank bottom (i.e., 6–8ft.) to verify adequacy of previous excavation.

Soil will be recovered continuously, described by the onsite geologist according to the Unified Soil Classification System (USCS) and screened with a PID. Indicators of potential site-related impacts (e.g., staining, obvious odors) will be noted and used to select intervals for laboratory analysis. The number of soil samples to be submitted for laboratory analysis from each site was determined based on the estimated volume of potentially impacted soil at each site, per DER-10 guidance (NYSDEC 2010a), as summarized in the following table.

Site Name	Site Acreage	Volume (yd^3)	No. Soil Borings	Minimum No. Soil Samples(1)	Minimum No. Groundwater Samples
DS001 (Site 14)	0.07	1,442	7	7	0
DS003 (Site 12)	0.28	5,684	7	7	0
ST009 (Site 4)	0.35	4,482	8	8	3
ST011 (Site 1)	0.09	1,171	8	8	3
TU956 (UST304)	0.004	68	4	4	0
TU962 (UST600)	0.002	26	4	4	0

1. Calculated based on the guidance provided in Table 5-4 of DER-10 (NYSDEC 2010a).

Soil samples will be submitted for laboratory analysis as follows:

- DS001—seven soil samples from the 0 to 2 ft bgs interval to verify that a surface release did not occur.
- DS003—six samples from the 0 to 2 ft bgs interval to verify that a surface release did not occur. An additional soil sample will be collected from the 6 to 8 ft bgs interval at one location to verify that soil at the groundwater interface has not been impacted. This additional boring location will be located near a historic sample location where TCE was detected at 0.55 mg/kg.
- ST009—eight subsurface soil samples at the soil/groundwater interface (or refusal) to verify subsurface soil conditions in the groundwater fluctuation zone.
- ST011—eight subsurface soil samples at the soil/groundwater interface (or refusal) to verify subsurface soil conditions in the groundwater fluctuation zone.
- TU956—four subsurface soil samples at the soil/groundwater interface (or refusal) to verify subsurface soil conditions in the groundwater fluctuation zone based on the depth of the former USTs.
- TU962—four subsurface soil samples at the soil/groundwater interface (or refusal) to verify subsurface soil conditions in the groundwater fluctuation zone based on the depth of the former USTs.

At least one soil sample will be submitted for analysis from each soil boring location. Soil samples from additional depth intervals within a boring may be selected for laboratory analysis based on field observations indicative of potential site-related impacts (e.g., soil staining, PID measurements). Also, in the event that an interval exhibits evidence of site-related impacts, the soil boring will be advanced to the next depth interval and sampled.

Sampling Plan

a) Soil samples will be collected from locations near identified site boundaries in order to assess current site conditions and compare to UU criteria. Sample locations were chosen based on available historical site use information and sampling data in order to fill any potential data gaps.

b) Samples will be collected based on field observation of each interval. In general; the soil interval containing the highest PID reading, or greatest visual evidence of sheens or staining, will be collected. If there is more than one zone with contamination, a shallow and deep location may be warranted to capture contaminant fluctuation from the water bearing zone. If no interval shows visual evidence of contamination or increased PID readings, a soil boring sample will be taken from the sample interval specified in Worksheet #18. Once the sample interval has been

determined, soil samples will be placed in the sample container provided by the analytical laboratory with nitrile gloves. The sample containers will be labeled, tracked via chain of custody forms, and packed and shipped to an offsite laboratory for analysis, as per SOPs 1, 2, and 4, respectively.

c) Subsurface soil samples will be collected using acetate sleeves deployed using direct-push drilling techniques as per SOP 47. Samples will be generally collected using 2-ft subsample intervals; the depth of the sample collection interval will be documented in the field notes, which will include the sample location/identification number (Worksheet #18).

d) Coordinates of sample locations will be recorded with a global positioning system unit in North American Datum (NAD) 1983.

e) PID operation, calibration, and maintenance procedures will be as per SOP 11.

f) Non-dedicated downhole and sampling equipment will be decontaminated prior to use, between sampling locations, and following sampling completion. Decontamination procedures will include a non-phosphate detergent wash, distilled water rinse, etc.; or steam-cleaning; as per SOP 5.

g) IDW will be containerized appropriately, if required following discussions with NFARS personnel, until it is characterized for disposal.

h) Soil borings that penetrate asphalt will be backfilled with hydrated bentonite pellets to approximately 6 in. bgs. An asphalt cold patch will be applied from the top of the hydrated bentonite to the top of the ground surface and will be emplaced in order to match the surrounding material. Soil borings in grassy areas will be backfilled with soil and/or bentonite, as needed.

i) Borings will be abandoned as discussed in SOP 28 and Worksheet #21.

Groundwater Sampling

In addition to soil borings, discrete *in situ* groundwater samples will be collected at ST009 and ST011 using direct-push drilling techniques. Based on previous site investigations, low-level groundwater concentrations of COCs may remain at these sites. Groundwater contamination at ST009, as of the last sampling event in 1998, was limited to low concentrations of benzene (8.3 µg/L). At ST011, sampling results from a 2009 field event indicated that only one of six groundwater samples had detections of petroleum-related COCs above groundwater standards. As such, groundwater samples are required for both of these sites to confirm that potential COCs have degraded to concentrations below UU criteria. Three groundwater grab samples will be collected at each of these two sites from depths at or just below the water table to assess any existing site-related impacts.

In the event that concentrations of site-specific COPCs in subsurface soil at Sites DS001, DS003, UST300, and UST600 indicate that the potential for impacts to groundwater, EA will remobilize and collect groundwater samples from these sites to evaluate for potential impacts to groundwater..

Sampling Plan

a) Groundwater samples will be collected from locations where UU criteria exceedances were previously observed to assess current site conditions and compare to UU criteria.

b) Groundwater samples will be collected near downgradient site boundaries to assess current site conditions and compare to UU criteria.

c) Groundwater samples will be collected using direct-push sample techniques (e.g., the Geoprobe® SP-22 groundwater sampler or similar), as per SOP 47. After advancing the drill casing to the desired depth, the discrete interval sampler (e.g., SP-22 sampler or similar) will be advanced a minimum of 4 ft below the depth of the drill casing. The drill rod will be pulled back, exposing a minimum of 2 ft of screen. A groundwater sample will be collected using a peristaltic or mini-inertial pump (i.e., check valve and tubing) to directly fill the sample containers provided by the analytical laboratory. The sample containers will be labeled, tracked via chain of custody forms, and packed and shipped to an offsite laboratory for analysis, as per SOPs 1, 2, and 4, respectively.

d) The groundwater sample collected will be a grab sample; therefore, field parameters such as pH, temperature, dissolved oxygen, and specific conductivity will not be recorded during sampling.

e) If discrete interval sampling techniques are not feasible, then a temporary monitoring well may be installed. In this case, a small diameter (e.g., 1-in. inner diameter) PVC slotted screen with sufficient riser pipe to reach the ground surface would be installed. The subsurface materials would be allowed to collapse around the temporary well. A groundwater grab sample would be collected using a peristaltic pump, mini-inertial pump, or mini-bailer following purging of at least one well volume of water. Following sample collected, the casing will be pulled and the soil boring will be backfilled consistent with the approach for soil borings and SOP 8.

f) Non-dedicated downhole and sampling equipment will be decontaminated prior to use, between sampling locations, and following sampling completion. Decontamination procedures will include a non-phosphate detergent wash, distilled water rinse, etc., as per SOP 5.

g) IDW will be containerized appropriately, if required following discussions with NFARS personnel, until it is characterized for disposal.

Additional information regarding concentration levels, sampling locations, number of samples to be taken, and sampling frequency is presented in Worksheets #18 and #20.

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QAPP WORKSHEET #18

Sampling Locations and Methods

Sampling Location/ Identification Number (XX-XX designates the sample interval, in feet)	Matrix/ Collection Method	Depth (ft bgs)	Analytical Group(s)	Number of Samples (identify field duplicates)[1]	Sampling SOP Reference[2]	Rationale for Sampling Location[3]
DS001 (Site 14)						
DS001-SB-01-XX-XX through DS001-SB-07-XX-XX	Surface Soil/direct-push drilling rig; VOCs collected with En Core® or similar.	0-2	• TCL organics and TAL inorganics (only one sample from 0-2 or 4-6 ft interval at site) • TCL VOCs and SVOCs at all other locations	• Approximately 7 • 1 duplicate • 1 matrix spike/ matrix spike duplicate	1-5, 11, 15-16, 25, 31, 39, 47, and 59	Evaluate surface soil at the perimeter of the former drum storage area to verify that a surface release did not occur.
DS003 (Site 12)						
DS003-SB-01- XX-XX through DS003-SB-08-XX-XX	Surface and Subsurface Soil/direct-push drilling rig; VOCs collected with En Core® or similar.	0-2 6-8	• TCL organics and TAL inorganics (only one sample from 0-2 or 4-6 ft. interval at site) • TCL VOCs and SVOCs at all other locations	• Approximately 7 • 1 duplicate • 1 matrix spike/ matrix spike duplicate	1-5, 11, 15-16, 25, 31, 39, 47, and 59	Evaluate surface soil at the former drum storage area in unpaved areas to verify that a surface release did not occur. The target depth is the surface soil based on the lack of documented spills or monitoring requirements. One sample will also be collected from the soil/groundwater interface based on a historic detection of TCE at 6-8 ft bgs.

1. QA/QC may vary depending on sampling groups. If multiple sites are sampled in a field event, duplicates and MS/MSDs may be collected a rate of one per 20 samples per matrix, per analyte group, and per sample delivery group, as a minimum.
2. From the Project Sampling SOP References table (Worksheet #21).
3. Based upon field observations, soil boring locations may be adjusted based on utility clearance or accessibility. Additional soil borings may be required to delineate the extent of site-related impacts. Additional samples for laboratory analysis may be selected based on field observations to target potentially impacted intervals. Soil samples for full TCL/TAL analysis will be selected from intervals above the water table.
4. Dependent on groundwater recharge rates, sufficient volume for full TCL/TAL analysis may require installation of a temporary monitoring well. If samples appear cloudy, then a filtered sample will also be submitted for analysis of TAL metals.
5. Additional groundwater samples will be collected at locations where soil sample results indicate the potential for impacts to groundwater.

Sampling Location/ Identification Number (XX-XX designates the sample interval, in feet)	Matrix/ Collection Method	Depth (ft bgs)	Analytical Group(s)	Number of Samples (identify field duplicates)[1]	Sampling SOP Reference[2]	Rationale for Sampling Location[3]
ST009 (Site 4)						
ST009-GW-01 through ST009-GW-03	Groundwater/direct-push drilling rig; samples collected with *in situ* sampling device (e.g., Geoprobe® SP-22 sampler or similar) or temporary well	0-10	• TCL organics and TAL inorganics (at least one sample at site)[4] • TCL VOCs and SVOCs (minimum required analysis for each groundwater sample)	• Approximately 3 • 1 duplicate • 1 matrix spike/ matrix spike duplicate	1-5, 10, 15-16, 31, 39, 43, 47, and 59	Groundwater samples are required to confirm that COCs meet UU criteria. Samples will be collected from: one location upgradient of former source area, one downgradient of source area, and one in an area near the former well where benzene was detected at low concentrations (8.3 µg/L).
ST009-SB-01-XX-XX through ST009-SB-05-XX-XX	Subsurface Soil/direct-push drilling rig; samples collected at the soil/groundwater interface. VOCs collected with En Core® or similar.	6-8	• TCL organics and TAL inorganics (only one sample from 0-2 ft. interval or directly above the water table) • TCL VOCs and SVOCs at all other locations	• Approximately 8 • 1 duplicate • 1 matrix spike/ matrix spike duplicate	1-5, 11, 15-16, 25, 31, 39, 47, and 59	Proximity to original MOGAS pipe leak to evaluate any residual soil contamination. Soil samples will be collected from the soil/groundwater interface to confirm UU criteria have been met.
ST011 (Site 1)						
ST011-GW-01 through ST011-GW-03	Groundwater/direct-push drilling rig; samples collected with in-situ sampling device (e.g., Geoprobe® SP-22 sampler or similar) or temporary well	0-10	• TCL organics and TAL inorganics (at least one sample at site)[4] • TCL VOCs and SVOCs (minimum required analysis for each groundwater sample)	• Approximately 3 • 1 duplicate • 1 matrix spike/ matrix spike duplicate	1-5, 10, 13, 15-16, 19, 28, 31, 39, 43, 47-48, and 59	Groundwater samples are required to confirm that COCs meet UU criteria. Samples will be collected from: locations north/north-west of the former pipeline leak. Previous groundwater results from locations northwest of the pipeline area indicated potential for groundwater exceedances.

Sampling Location/ Identification Number (XX-XX designates the sample interval, in feet)	Matrix/ Collection Method	Depth (ft bgs)	Analytical Group(s)	Number of Samples (identify field duplicates)[1]	Sampling SOP Reference[2]	Rationale for Sampling Location[3]
ST011-SB-01-XX-XX through ST011-SB-05-XX-XX	Subsurface Soil/direct-push drilling rig; samples collected at the soil/groundwater interface. VOCs collected with En Core® or similar.	6-8	• TCL organics and TAL inorganics (only one sample from 0-2 ft. interval or directly above the water table) • TCL VOCs and SVOCs at all other locations	• Approximately 8 • 1 duplicate • 1 matrix spike/ matrix spike duplicate	1-5, 11, 15-16, 25, 31, 39, 47, and 59	Proximity to original JP-4 pipeline leak to evaluate any residual soil contamination. Soil samples will be collected from the soil/groundwater interface to confirm UU criteria have been met.
TU956 (UST304)						
TU956-SB-01-XX-XX through TU956-SB-04-XX-XX	Subsurface Soil/direct-push drilling rig; samples collected at the soil/groundwater interface. VOCs collected with En Core® or similar.	4-6	• TCL organics and TAL inorganics (only one sample from 4-6 ft.) • TCL VOCs and SVOCs at all other locations	• Approximately 4 • 1 duplicate • 1 matrix spike/ matrix spike duplicate	1-5, 11, 15-16, 25, 31, 39, 47, and 59	Samples will be collected from the 4-6ft. interval (the bottom of the UST excavation) to confirm UU criteria have been met.
TU962 (UST600)						
TU962-SB-01-XX-XX through TU962-SB-04-XX-XX	Subsurface Soil/direct-push drilling rig; samples collected at the soil/groundwater interface. VOCs collected with En Core® or similar.	4-6	• TCL organics and TAL inorganics (only one sample from 4-6ft.) • TCL VOCs and SVOCs at all other locations	• Approximately 4 • 1 duplicate • 1 matrix spike/ matrix spike duplicate	1-5, 11, 15-16, 25, 31, 39, 47, and 59	Samples will be collected from the 4-6ft. interval (the bottom of the UST excavation) to confirm UU criteria have been met.
QC Samples						
GW-TB-YYMMDD	Groundwater Trip Blank	NA	• VOCs	1 per cooler containing samples for VOC analysis	1-5, 10, 15-16, 31, 39, 43, 47, and 59	QC sample, VOC trip blank for water samples

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QAPP WORKSHEETS #19 AND 30

Sample Containers, Preservation, and Hold Times

Laboratory: Spectrum Analytical, Inc. RI Division

List any required accreditations/certifications: DoD ELAP and State of NYSDOH Environmental Laboratory Certification Program (Appendix A)

Back-up Laboratory: None

Sample Delivery Method: Expedited courier service

Analyte/ Analytical Group	Matrix	Method/SOP	Accreditation Expiration Date	Container Size/Type[1]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
VOCs	Water	SW8260C/ 90.0012	Appendix A	(2) 40-mL VOA vials with Teflon™-lined septum cap	HCl to pH≤2; cool to ≤4°C; not frozen	14 days from sample collection until analysis	14 days from sample collection until analysis	5 working days unless otherwise specified in project planning documents
SVOCs	Water	SW8270D/ 70.0012	Appendix A	(2) 1-L amber glass with Teflon®-lined cap	Cool to ≤4°C; not frozen	7 days from sample collection until extraction	40 days from extraction until analysis	5 working days unless otherwise specified in project planning documents
PAHs and 1,4-Dioxane	Water	SW8270D SIM/ 70.0033	Appendix A	(2) 1-L amber glass with Teflon®-lined cap	Cool to ≤4°C; not frozen	7 days from sample collection until extraction	40 days from extraction until analysis	5 working days unless otherwise specified in project planning documents
Pesticides	Water	SW8081B/ 60.0006	Appendix A	(2) 1-L amber glass with Teflon®-lined cap	Cool to ≤4°C; not frozen	7 days from sample collection until extraction	40 days from extraction until analysis	5 working days unless otherwise specified in project planning documents
PCBs	Water	SW8082A/ 60.0003	Appendix A	(2) 1-L amber glass with Teflon®-lined cap	Cool to ≤4°C; not frozen	7 days from sample collection until extraction	40 days from extraction until analysis	5 working days unless otherwise specified in project planning documents

1. For soil samples, in some cases analyses may be combined into few jars. Please consult the analytical laboratory or the Project Chemist.

NOTE: VOA = Volatile organic analysis
HCl = Hydrochloric acid

Analyte/ Analytical Group	Matrix	Method/SOP	Accreditation Expiration Date	Container Size/Type[(1)]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
Herbicides	Water	SW8151A/ 60.0034	Appendix A	(2) 1-L amber glass with Teflon®-lined cap	Cool to ≤4°C; not frozen	7 days from sample collection until extraction	40 days from extraction until analysis	5 working days unless otherwise specified in project planning documents
Metals	Water	SW6020A/ 100.0110	Appendix A	(1) 500mL HDPE	HNO_3 to pH≤2; cool to ≤4°C; not frozen	180 days from sample collection until analysis	180 days from sample collection until analysis	5 working days unless otherwise specified in project planning documents
Hexavalent Chromium	Water	SM3500 B Cr+6/100.0308	Appendix A	(1) 250mL HDPE	Cool to ≤4°C	24 hours from sample collection until analysis	24 hours from sample collection until analysis	5 working days unless otherwise specified in project planning documents
Mercury	Water	SW7470A/ 100.0012	Appendix A	(1) 500-mL HDPE	HNO_3 to pH≤2; cool to ≤4°C; not frozen	28 days from sample collection until analysis	28 days from sample collection until analysis	5 working days unless otherwise specified in project planning documents
Cyanide	Water	SW9012B/ 100.0004	Appendix A	(1) 250mL HDPE	NaOH to pH > 12 Cool to ≤4°C	14 days from sample collection until analysis	14 days from sample collection until analysis	5 working days unless otherwise specified in project planning documents
VOCs	Soil	SW8260C/ 90.0012	Appendix A	(2) 40-mL VOA vial with Teflon®-lined lid (pre-tared) containing stir bar, sodium bisulfate, and reagent water and (1) 40-mL VOA vial with methanol	Cool to ≤4°C; sodium bisulfate; frozen; methanol	14 days from sample collection until analysis (if not reagent water aliquot is not frozen - 48 hours)	14 days from sample collection until analysis (if not reagent water aliquot is not frozen - 48 hours)	5 working days unless otherwise specified in project planning documents
SVOCs	Soil	SW8270D/ 70.0011	Appendix A	(1) 8-ounce glass jar with Teflon®-lined lid	Cool to ≤4°C; not frozen	14 days from sample collection until extraction	40 days from extraction until analysis	5 working days unless otherwise specified in project planning documents
PAHs and 1,4-Dioxane	Soil	SW8270D SIM/ 70.0033	Appendix A	(1) 8-ounce glass jar with Teflon®-lined lid	Cool to ≤4°C; not frozen	14 days from sample collection until extraction	40 days from extraction until analysis	5 working days unless otherwise specified in project planning documents

NOTE: HDPE = High density polyethylene
HNO3 = Nitric acid.
NaOH = Sodium hydroxide.

Analyte/ Analytical Group	Matrix	Method/SOP	Accreditation Expiration Date	Container Size/Type[(1)]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
Pesticides	Soil	SW8081B/ 60.0006	Appendix A	(1) 8-ounce glass jar with Teflon®-lined lid	Cool to ≤4°C; not frozen	14 days from sample collection until extraction	40 days from extraction until analysis	5 working days unless otherwise specified in project planning documents
PCBs	Soil	SW8082A/ 60.0003	Appendix A	(1) 4- or 8-ounce glass jar with Teflon®-lined lid	Cool to ≤4°C; not frozen	14 days from sample collection until extraction	40 days from extraction until analysis	5 working days unless otherwise specified in project planning documents
Herbicides	Soil	SW8151A/ 60.0034	Appendix A	(1) 4- or 8-ounce glass jar with Teflon®-lined lid	Cool to ≤4°C; not frozen	14 days from sample collection until extraction	40 days from extraction until analysis	5 working days unless otherwise specified in project planning documents
Metals	Soil	SW6010C/ 100.0111	Appendix A	(1) 2-ounce glass jar with Teflon®-lined lid	Cool to ≤4°C; not frozen	180 days from sample collection until analysis	180 days from sample collection until analysis	5 working days unless otherwise specified in project planning documents
Hexavalent Chromium	Soil	SW7196A/ 100.0208	Appendix A	(1) 2-ounce glass jar with Teflon®-lined lid	Cool to ≤4°C; not frozen	14 days from sample collection until preparation	14 days from sample collection until analysis	5 working days unless otherwise specified in project planning documents
Mercury	Soil	SW7471B/ 100.0012	Appendix A	(1) 2-ounce glass jar with Teflon®-lined lid	Cool to ≤4°C; not frozen	28 days from sample collection until analysis	28 days from sample collection until analysis	5 working days unless otherwise specified in project planning documents
Cyanide	Soil	SW9012B/ 100.0004	Appendix A	(1) 2-ounce glass jar with Teflon®-lined lid	Cool to ≤4°C; not frozen	14 days from sample collection until extraction	14 days from sample collection until extraction	5 working days unless otherwise specified in project planning documents

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QAPP WORKSHEET #20

Field Quality Control Summary

Matrix	Analytical Group	Concentration Level	Analytical Preparation Method/SOP Reference[1]	No. of Samples[2]	No. of Trip Blanks	No. of Equipment Blanks[3]	No. of Field Duplicate Pairs	No. of Matrix Spike/Matrix Spike Duplicate Pairs
DS001 (Site 14)								
Subsurface Soil	• TCL organics and TAL inorganics (1 location) • TCL VOCs and SVOCs at all other locations	Low	Contract Laboratory Program – Method 5035 Field Preservation	7	NA	0	1	1
DS003 (Site 12)								
Subsurface Soil	• TCL organics and TAL inorganics (1 location) • TCL VOCs and SVOCs at all other locations	Low	Contract Laboratory Program – Method 5035 Field Preservation	8	NA	0	1	1
ST009 (Site 4)								
Subsurface Soil	• TCL organics and TAL inorganics (1 location) • TCL VOCs and SVOCs at all other locations	Low	Contract Laboratory Program – Method 5035 Field Preservation	8	NA	0	1	1
Groundwater	• TCL organics and TAL inorganics (at least 1 location at site) • TCL VOCs and SVOCs (minimum required analysis for each groundwater sample)	Low	Contract Laboratory Program – Current Method	3	One for each cooler containing sampling containers for analysis of VOCs	1	1	1

1. SOPs included in Appendices A (lab) and B (field). Note that SOPs for standard methods are not included.
2. Standard, non-QC samples. Sample numbers listed are estimates and may change based upon field conditions. See Worksheet #11 for more details on sample numbers, broken down by task.
3. Equipment Blanks are only required for groundwater sampling – may be required for soil depending on method used.

Matrix	Analytical Group	Concentration Level	Analytical Preparation Method/SOP Reference[1]	No. of Samples[2]	No. of Trip Blanks	No. of Equipment Blanks[3]	No. of Field Duplicate Pairs	No. of Matrix Spike/Matrix Spike Duplicate Pairs
ST011 (Site 1)								
Subsurface Soil	• TCL organics and TAL inorganics (1 location) • TCL VOCs and SVOCs at all other locations	NA	Contract Laboratory Program – Method 5035 Field Preservation	8	NA	0	1	1
Groundwater	• TCL organics and TAL inorganics (at least 1 location at site) • TCL VOCs and SVOCs (minimum required analysis for each groundwater sample)	NA	Contract Laboratory Program – Current Method	3	One for each cooler containing sampling containers for analysis of VOCs	1	1	1
TU956 (UST304)								
Subsurface Soil	• TCL organics and TAL inorganics (1 location) • TCL VOCs and SVOCs at all other locations	NA	Contract Laboratory Program – Method 5035 Field Preservation	4	NA	0	1	1
TU962 (UST600)								
Subsurface Soil	• TCL organics and TAL inorganics (1 location) • TCL VOCs and SVOCs at all other locations	NA	Contract Laboratory Program – Method 5035 Field Preservation	4	NA	0	1	1

QAPP WORKSHEET #21

Field Standard Operating Procedures

The table below presents the EA SOPs that will be used during PBR activities.

Reference Number[(1)]	Title, Revision Date, and/or Number	Organizing Organization	Equipment Type	Modified for Project Work (Check if yes)	Comments
SOP 1	Sample Labels	EA	Documents		
SOP 2	Chain of Custody Form	EA	Documents		
SOP 3	Subsurface Utility Clearance	EA	Documents		
SOP 4	Sample Packing and Shipping	EA	Supplies		
SOP 5	Field Decontamination – REV 2	EA	Various, including but not limited to non-phosphate detergent and distilled water, aluminum foil		
SOP 10	Water Level and Well Depth Measurements – REV 2	EA	Solinst Model 101 Water Level Meter		
SOP 11	PID MiniRae	EA	Various including but not limited to MiniRae, calibration gas (100 ppm isobutylene), battery pack, tedlar bag, tygon tubing, regulator		
SOP 13	Monitoring Well Sample Collection	EA	Various including but not limited to Bladder pump (dedicated to one well only), Peristaltic pump with tubing for filtering samples, Submersible pump and hose (for purging only), Variable speed low-flow submersible pump (e.g. Grundfos MP1 sampling pump), Polytetrafluoroethelyne (PTFE) bailer, Polyvinyl chloride bailer, transparent bailer 0.45µM filters, Horiba U-52 water quality probe, Generator, Field book and field parameter forms, MiniRae probe, Plastic sheeting, Polypropylene rope, sample bottles and lables, Tygon tubing, Solinst Model 101 water level meter.		

1. Field SOPs can be found in Appendix B

Reference Number[(1)]	Title, Revision Date, and/or Number	Organizing Organization	Equipment Type	Modified for Project Work (Check if yes)	Comments
SOP 15	Document Control System	EA	Documents		
SOP 16	Surface Water, Groundwater, Soil-Sediment Field Logbooks	EA	Field logbook and indelible ink		
SOP 19	Monitoring Well Installation	EA	Various including but not limited to drill rig, augers, bits, drill stem, steam cleaner and water, Photoionization detector, water level indicator, weighted steel tape measure, lower explosive limit oxygen monitor, steel drums, heavy plastic sheeting, well screens, riser pipes, well plugs/caps, filter pack, bentonite, cement	X	Monitoring well installation (if required) will follow procedures detailed in the NFARS Installation-Wide Groundwater Monitoring Work Plan (EA, 2010).
SOP 25	Soil Sampling	EA	Various including but not limited to Geoprobe, spoons, shovels, hand-augers, split-spoon samplers, backhoes, acetate sleeves, coring devices, and methanol (for VOC) sample containers		
SOP 28	Well and Boring Abandonment	EA	Various, including but not limited to Drill rig, Filter pack material, Bentonite, Cement, Water		
SOP 31	Sample Container Cleaning	EA	Performed by laboratories		
SOP 39	Sample Preservation and Container Requirements	EA	Documents		
SOP 43	Multi-Probe Water Quality Monitoring Instruments	EA	Multi-probe instrument (Horiba U-52), Accessories (batteries, charger, case), Field logbook, Operations Manual		
SOP 47	Direct-Push Technology Sampling	EA	Various including direct-push technology rig and equipment, Logbook, personal protective equipment, Decontamination equipment, Steel drums, heavy plastic sheeting		

Reference Number[(1)]	Title, Revision Date, and/or Number	Organizing Organization	Equipment Type	Modified for Project Work (Check if yes)	Comments
SOP 48	Low-Flow Sampling	EA	Various including but not limited to Bladder pump (dedicated to one well only), Peristaltic pump with tubing for filtering samples, Submersible pump and hose (for purging only), Variable speed low-flow submersible pump (e.g. Grundfos MP1 sampling pump), PTFE bailer, Polyvinyl chloride bailer, Stainless steel bailer, transparent bailer with a double check valve, 0.45µM filters, Horiba U-52 water quality probe, Generator, Field book and field parameter forms, MiniRae probe, Plastic sheeting, Polypropylene rope, sample bottles and lables, Tygon tubing, Solinst Model 101 water level meter.		
SOP 59	Field Logbook	EA	Logbook and indelible ink		
SOP 61	GIS Data Management and Deliverables	EA	Documents		

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QAPP WORKSHEET #22

Field Equipment Calibration, Maintenance, Testing, and Inspection

Field Equipment	Activity	SOP Reference	Title or Position of Responsible Person	Frequency	Acceptance Criteria	Corrective Action
Water Quality Meter (QED, Troll, YSI, or Horiba)	Calibration – follow manufacturer's instructions. Two point calibration for pH, specific conductance, turbidity	SOP 43 and equipment manual	Field personnel	Calibrate daily before use and when unstable readings occur. Maintain and inspect daily when used.	Within calibration standard(s) range.	Cleaning and recalibration
	Maintenance – decontaminate and store in water short term, long-term storage according to the manufacturer for each sensor	SOP 43 and equipment manual	Field Team Leader	Daily, before use and when unstable readings occur	None	None
	Field test in accordance with the manual	SOP 43 and equipment manual	Field personnel	As needed	See equipment manual	None
	Visually inspect probes for cleanliness and wear	SOP 43 and equipment manual	Field personnel	As needed	None	None

Field Equipment	Activity	SOP Reference	Title or Position of Responsible Person	Frequency	Acceptance Criteria	Corrective Action
Electronic Water Level Meter or Oil/water Interface Probe	Maintenance -– decontaminate between wells	SOP 10 and equipment manual	Field Team Leader	Daily	Response	Replace battery if no response during test button check.
	Inspect tape for kinks and cuts, inspect probe for dirt, check batteries	SOP 10 and equipment manual	Field personnel	As needed	None	None
	Field test in accordance with the manual	SOP 10 and equipment manual	Field personnel	As needed	See equipment manual	None
Trimble® GeoXT™ Global Positioning System Unit	Validate accuracy using nearby benchmark	See equipment manual	Field personnel	Daily	Refer to manufacturer's instructions	Refer to manufacturer's instructions
	Charge battery and place in case at the end of each day	See equipment manual	Field personnel	As needed	None	None
	Testing	See equipment manual	Field personnel	Daily	Refer to manufacturer's instructions	Refer to manufacturer's instructions
	Inspect for external damage (i.e., liquid crystal display [LCD] screen, dents, etc.).	See equipment manual	Field Team Leader	As needed	None	None
MiniRAE Lite Photoionization Detector (10.2 eV lamp) MultiRae Plus (4-gas Meter)	Two-point calibration using fresh air and span gas (100 parts per million isobutylene)	SOP 11 and See equipment manual	Field personnel	Daily and when unstable readings occur	Stable reading with no drift	Recalibrate. If necessary, change moisture traps and clean lamp.
	Keep clean and replace moisture traps as needed. Place in case at the end of each day	SOP 11 and See equipment manual	Field personnel	As needed	None	None
	Field test in accordance with the manual	SOP 11 and See equipment manual	Field personnel	Daily	Refer to manufacturer's instructions	Refer to manufacturer's instructions
	Inspect for external damage (i.e., LCD screen, dents, etc.). Recalibrate as needed.	SOP 11 and See equipment manual	Field Team Leader	As needed	None	None

Field Equipment	Activity	SOP Reference	Title or Position of Responsible Person	Frequency	Acceptance Criteria	Corrective Action
Gram Scale	Calibrate according to manufacturer's Instructions	See equipment manual	Field personnel	Daily, before use and when unstable readings occur	Within calibration standard(s) range	Recalibration
	Decontaminate and place in hard case between sampling activities	See equipment manual	Field personnel	As needed	None	None
	Testing	See equipment manual	Field personnel	Daily	Refer to manufacturer's instructions	Refer to manufacturer's instructions
	Inspect for external damage (i.e., LCD screen, etc.)	See equipment manual	Field Team Leader	As needed	None	None
Grundfos Pump and Controller	Maintenance – Decontaminate between wells	See equipment manual	Field personnel	Daily and between wells	None	None
	Inspect electrical leads for kinks and cuts	See equipment manual	Field personnel	As needed	None	None
	Field test in accordance with the manual	See equipment manual	Field personnel	As needed	See equipment manual	Refer to manufacturer's instructions
	Inspect pump and controller for external damage	See equipment manual	Field personnel	As needed	None	None
Peristaltic Pump	Field test in accordance with the manual	See equipment manual	Field personnel	As needed	None	None
	Inspect pump for external damage	See equipment manual	Field personnel	As needed	None	None

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QAPP WORKSHEET #23

Analytical Standard Operating Procedures

SOP#	Title	Organization Performing Analysis	Revision Date	Revision	Definitive or Screening Data	Matrix/Analytical Group	Instrument	Modified for Project Work
NA	DoD QSM for Environmental Laboratories	DoD Environmental Quality Workgroup	10/1/10	4.2	Definitive	General	NA	No
NA	QA Manual	Spectrum RI	10/19/12	2012	Definitive	General	NA	No
80.0005	Method Detection Limit (MDL) Determination	Spectrum RI	03/16/11	12	Definitive	QA	NA	No
110.0008	Manual Integration of GC, Ion chromatography and GC/Mass Spectrometry Chromatograms	Spectrum RI	7/15/11	10	Definitive	QA	NA	No
110.0039	Sub-Sampling for Soil and Solid Samples	Spectrum RI	04/06/06	3	Definitive	Sample Preparation	NA	No
50.0027	Organic Preparation of Aqueous Samples for Chlorinated Herbicides (Method 8151A)	Spectrum RI	10/19/12	9	Definitive	Organic Preparation	NA	No
50.0054	Organic Extract Filtration and Concentration Techniques	Spectrum RI	8/17/11	3	Definitive	Organic Preparation	NA	No
50.0050	Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid (Method 3520)	Spectrum RI	4/26/11	6	Definitive	Organic Preparation	NA	No
50.0053	Organic Preparation of Soil Samples by Soxhlet (Method 3540)	Spectrum RI	2/2/10	3	Definitive	Organic Preparation	NA	No
50.0051	Organic Preparation of Aqueous Samples by Separatory Funnel (Method 3510)	Spectrum RI	2/2/10	2	Definitive	Organic Preparation	NA	No
30.0003	Sample Receipt, Storage, Tracking and Disposal	Spectrum RI	9/19/12	17	Definitive	NA	NA	No

SOP#	Title	Organization Performing Analysis	Revision Date	Revision	Definitive or Screening Data	Matrix/Analytical Group	Instrument	Modified for Project Work
50.0052	Organic Preparation of Soil Samples by Sonication (Method 3550)	Spectrum RI	2/2/10	3	Definitive	Organic Preparation	NA	No
60.0006	Determination of Pesticides by GC/Electron Capture Detector (ECD) Analysis by SW846 Method 8081B	Spectrum RI	4/11/11	10	Definitive	Pesticides	GC/ECD	No
60.0003	Determination of PCBs by GC/ECD Analysis by SW846 Method 8082A	Spectrum RI	4/11/11	10	Definitive	PCBs	GC/ECD	No
60.0034	Determination of Chlorinated Herbicides by GC/ECD Analysis by SW846 Method 8151A	Spectrum RI	4/8/11	8	Definitive	Herbicides	GC/ECD	No
70.0033	SIM Analysis by GC/ Mass Spectrometry (Modified EPA Method 8270D)	Spectrum RI	8/22/11	7	Definitive	SVOCs	GC/ Mass Spectrometry	No
90.0012	Determination of VOCs by GC/Mass Spectrometry Analysis by SW846 Method 8260C	Spectrum RI	9/17/12	13	Definitive	VOCs	GC/ Mass Spectrometry	No
70.0011	Determination of SVOCs by GC/Mass Spectrometry Analysis by SW846 Method 8270D	Spectrum RI	7/18/11	11	Definitive	SVOCs	GC/ Mass Spectrometry	No
100.0003	Sample Preparation of Aqueous Samples by Acid Digestion ICP and ICP/ Mass Spectrometry (3005/3010)	Spectrum RI	2/16/10	8	Definitive	Inorganic Preparation	NA	No
100.0104	Sample Preparation of Soils by Acid Digestion for ICP/ Atomic Emission Spectrometry (AES) (3050B/6010C)	Spectrum RI	3/26/10	8	Definitive	Inorganic Preparation	NA	No
20.0003	Logging Work orders into Omega	Spectrum RI	9/18/12	5	Definitive	NA	NA	No

SOP#	Title	Organization Performing Analysis	Revision Date	Revision	Definitive or Screening Data	Matrix/Analytical Group	Instrument	Modified for Project Work
100.0111	Determination of Metals in Water and Wastes by Inductively Coupled Argon Plasma AES by SW846 Method 6010C	Spectrum RI	12/22/10	13	Definitive	Metals	ICP	No
100.0100	Sample Preparation of Soils by Acid Digestion for ICP/ MS (3050B/6020A)	Spectrum RI	3/09/10	0	Definitive	Inorganic Preparation	NA	No
100.0110	Determination of Metals in Water and Wastes by Inductively Coupled Argon Plasma MS by SW846 Method 6020A	Spectrum RI	04/16/10	2	Definitive	Metals	ICP	No
100.0308	Hexavalent Chromium in Aqueous Samples by Standard Methods SM3500 Cr +6 B	Spectrum RI	10/12/12	8	Definitive	Inorganic	SPEC	No
100.0208	Hexavalent Chromium in Soil Samples by SW846 Methods 3060A & 7196A	Spectrum RI	1/1/10	8	Definitive	Inorganic	SPEC	No
100.0012	Mercury Analysis in Aqueous Samples by Flow Injection Analysis System for Atomic Analysis by Method 7470A/7471B	Spectrum RI	6/15/10	10	Definitive	Metals	Cold vapor atomic absorption (CVAAS)	No
100.0004	Total Cyanide by Automated Colorimetric with Midi-distillation by SW846 9012B	Spectrum RI	3/12/12	8	Definitive	Inorganic	Flame Ionization Detector	No

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QAPP WORKSHEET #24

Analytical Instrument Calibration

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/ECD	Minimum five-point ICAL for target analytes; lowest concentration standard at or near the LOQ.	Low	ICAL is performed prior to sample analysis initially upon instrument set up, after major changes to system, or when ICAL or CCV cannot be met. A second source ICV standard is run after an acceptable calibration, and a CCV standard is analyzed after every 10 samples and at the end of the analytical sequence: %D≤20%	One of the options below: *Option 1*: linear – relative standard deviation (RSD) for each analyte ≤ 20% *Option 2*: linear – least squares regression r ≥ 0.995 for each analyte or *Option 3*: non-linear – COD ≥ 0.99 (six points shall be used for second order, seven points shall be used for third order)	Correct problem, document in maintenance log, then repeat ICAL.	Analyst/ Supervisor	60.0003, 60.0006, 60.0034
	Second-source ICV		Once per ICAL	Analytes within ±20%D of expected value	Correct problem and verify second-source standard. Rerun second-source verification. If that fails, correct problem and repeat ICAL.	Analyst/ Supervisor	
	CCV		Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence	Analytes within ±20% D of expected value	Correct problem, then rerun CCV. If that fails, repeat ICAL. Reanalyze all samples since the last successful CCV.	Analyst/ Supervisor	
	Breakdown check (Endrin/DDT) Method 8081B only		At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤15% for both DDT and endrin	Correct problem then repeat breakdown check.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/Mass Spectrometry	Minimum five-point ICAL for target analytes; lowest concentration standard at or near the LOQ.	Low	ICAL is performed prior to sample analysis initially upon instrument set up, after major changes to system, or when ICAL or calibration check compound (CCC) cannot be met. A second source ICV standard is run after acceptable calibration, and a CCV standard is analyzed daily and every 12 hours: %D≤20%	One of the options below: *Option 1*: linear – RSD for each analyte ≤20% *Option 2*: linear – least squares regression r ≥0.995 for each analyte or *Option 3*: non-linear – COD ≥0.99 (six points shall be used for second order, seven points shall be used for third order) Ten percent of target analytes can exceed the 20%RSD with a max. of 50%RSD.	Correct problem, document in maintenance log, then repeat ICAL.	Analyst/ Supervisor	70.0011, 70.0033, 90.0012
	Mass spectrometer tuning check		Before ICAL and calibration verification and at the beginning of each 12-hour shift	Refer to criteria listed in method description	Retune instrument and verify. Rerun affected samples.	Analyst/ Supervisor	
	Second-source ICV		Once per ICAL	Analytes within ± 20% of expected value.	Correct problem and verify second-source standard. Rerun second-source verification. If that fails, correct problem and repeat ICAL.	Analyst/ Supervisor	
	CCV		Daily, before sample analysis (unless ICAL performed on same day), and after every 12 hours of analysis time	Analytes within ± 20%D of expected value from ICAL.20% of target analytes can exceed the 20%RSD with a max. of 50%RSD.	Correct problem, then rerun CCV. If that fails, repeat ICAL.	Analyst/ Supervisor	
	Evaluation of RRT		Each sample, standard, and QC sample	RRT of each target analyte within ± 0.06 RRT units of ICAL or CCV	Correct problem, then repeat ICAL.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
ICP AES/Mass Spectrometry	If more than one standard is used, correlation coefficient must be $\geq$0.995	Low	ICAL is performed daily prior to sample analysis and initially upon instrument set up, after major changes to system, or when ICAL or CCC cannot be met. A second source ICV standard is run after an acceptable calibration, and a continuing calibration standard is analyzed after every 10 samples and at the end of the analytical sequence.	Calibration curve correlation coefficient r $\geq$0.995 if more than one standard and a blank; calibration verification acceptance ranges must be met: ICV/CCV shall be ±10% recovery of true value.	Correct problem then repeat initial calibration. Reported samples must be bracketed by compliant QC.	Analyst/ Supervisor	100.0110, 100.0111
	Mass Spectrometer performance check (ICP MS only).		Tuning is performed daily prior to any standard or sample analysis. Demonstrate instrument stability and precision by analyzing the tuning solution as a single analysis with at least five integrations.	The %RSD of the absolute signals for all of the multiple integrations in the tuning solution (as calculated by the instrument) must be ≤ 5.0% for each analyte	Retune the instrument before any further analyses.	Analyst/ Supervisor	
	Second-source ICV		Once per ICAL (after), prior to beginning a sample run.	Analytes within ± 10% of true value.	Correct problem and verify second-source standard. Rerun second-source verification. If that fails, correct problem and repeat ICAL.	Analyst/ Supervisor	
	CCV		Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	Analytes within ± 10% of true value.	Correct problem, then rerun CCV. If that fails, repeat ICAL. Reanalyze all samples since the last successful CCV.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
ICP AES/ Mass Spectrometry	Linear dynamic range or high-level check standard	Low	Every 6 months	Within ± 10% of true value.	NA	Analyst/ Supervisor	100.0110, 100.0111
	ICS		At the beginning of an analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes<LOQ (unless they are a verified trace impurity from one of the spiked analytes) and; ICS-AB: within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	Analyst/ Supervisor	
	Low-level calibration check standard		Daily, after ICAL.	Within ± 30% of true value.	Correct problem, then reanalyze.	Analyst/ Supervisor	
	Calibration blank (ICB/CCB)		Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
CVAAS	Minimum five-point ICAL and a calibration blank, lowest concentration standard at or near the LOQ.	Low	ICAL is performed daily prior to sample analysis and initially upon instrument set up, after major changes to system, or when calibration criteria or ICV/CCV acceptance criteria cannot be met.	Calibration curve correlation coefficient r $\geq$0.995; calibration verification acceptance ranges must be met: ICV ±10% recovery of true value; CCV ±20% recovery of true value.	Correct problem then repeat ICAL. Reported samples must be bracketed by compliant QC.	Analyst/ Supervisor	100.0012
	Second-source ICV		Once per ICAL, prior to beginning a sample run.	Analytes within ± 10% of true value.	Correct problem and verify second-source standard. Rerun second-source verification. If that fails, correct problem and repeat ICAL.	Analyst/ Supervisor	
	CCV		Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	Analytes within ± 20% of true value.	Correct problem, then rerun CCV. If that fails, repeat ICAL. Reanalyze all samples since the last successful CCV.	Analyst/ Supervisor	
	Calibration blank (ICB/CCB)		Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Automated Colorimetric	Minimum three-point ICAL with blank for target analytes, with one point at or below the LOQ.	Low	ICAL is performed prior to sample analysis and initially upon instrument set up, after major changes to system, or when calibration criteria or ICV/CCV acceptance criteria cannot be met.	Correlation coefficient, r, ≥ 0.995; calibration verification acceptance ranges must be met:	Correct problem then repeat ICAL. Reported samples must be bracketed by compliant QC.	Laboratory Manager / Analyst	100.0004
	Second-source ICV		Once per ICAL, prior to beginning a sample run.	Analytes within ± 15% of true value.	Correct problem and verify second-source standard. Rerun second-source verification. If that fails, correct problem and repeat ICAL.	Analyst/ Supervisor	
	CCV		Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	Analytes within ± 15% of true value.	Correct problem, then rerun CCV. If that fails, repeat ICAL. Reanalyze all samples since the last successful CCV.	Analyst/ Supervisor	
	Calibration blank (ICB/CCB)		Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Manual Colorimetric	Minimum three-point ICAL with blank for target analytes, with one point at or below the LOQ.	Low	ICAL is performed daily prior to sample analysis and initially upon instrument set up, after major changes to system, or when calibration criteria or ICV/CCV acceptance criteria cannot be met.	Correlation coefficient, r, ≥ 0.995; calibration verification acceptance ranges must be met:	Correct problem then repeat ICAL. Reported samples must be bracketed by compliant QC.	Laboratory Manager / Analyst	100.0208, 100.0308
	Second-source ICV		Once per ICAL, prior to beginning a sample run.	Analytes within ± 10% of true value.	Correct problem and verify second-source standard. Rerun second-source verification. If that fails, correct problem and repeat ICAL.	Analyst/ Supervisor	
	CCV		Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	Analytes within ± 10% of true value.	Correct problem, then rerun CCV. If that fails, repeat ICAL. Reanalyze all samples since the last successful CCV.	Analyst/ Supervisor	

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QAPP WORKSHEET #25

Analytical Instrument and Equipment Maintenance, Testing, and Inspection

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/ECD	**Daily:** check carrier gas supply; check temperatures of inlet and detectors; verify temperature program. **As needed:** check septa, clean injection port or replace injection port liner, and cut column if needed; reactivate carrier gas drying agents; replace or repair flow controllers if constant flow cannot be maintained; replace disposables; bake out instrument; recondition column; and perform detector cleaning.	Detector signals and chromato-gram review	Instrument performance and sensitivity	Maintenance as needed	Calibration verification standards (ICV and CCVs) pass QC criteria	Corrective action may include inspection of system; correct problem; and rerun calibration and affected samples, as well as calling the service engineer.	Laboratory Manager/ Analyst	60.0003, 60.0006, 60.0034
GC/Mass Spectrometry	**Daily:** check inlet pressure and sufficient supply of carrier gas; check temperatures of inlet and detectors; verify temperature program; check septa, clean injection port or replace injection port liner, and cut column if needed; check carrier gas supply; check tune parameters. **As needed:** check oil levels in mechanical pumps and the diffusion pump if vacuum is insufficient; replace electron multiplier; clean source; replace filaments; change rough pump oil and exhaust filters.	Tuning and sensitivity check	Instrument performance and sensitivity	Maintenance as needed	Tune and calibration verification standard (ICV and CCVs) pass QC criteria	Corrective action may include inspection of system; correct problem; and rerun calibration and affected samples, as well as calling the service engineer.	Laboratory Manager/ Analyst	70.0011, 70.0033, 90.0012

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
ICP AES or Mass Spectrometry	**Daily or as needed:** Perform leak test, clean torch and cones; replace peristaltic pump tubing as needed.	Normal analysis	Daily Performance check. Clean torch and cones; replace peristaltic pump tubing as needed.	Daily and as needed	CCV passes QC criteria	Corrective action may include inspection of system; correct problem; and rerun calibration and affected samples, as well as calling the service engineer.	Laboratory Manager/ Analyst	100.0110, 100.0111
CVAAS	**Daily or as needed:** Perform leak test, check tubing, clean window, and clean filters.	Normal analysis	Perform leak test, check tubing, clean window, and clean filters.	Daily and as needed	CCV passes QC criteria	Corrective action may include inspection of system; correct problem; and rerun calibration and affected samples, as well as calling the service engineer.	Laboratory Manager/ Analyst	100.0004
Manual Colorimetric	**Daily or as needed:** Keep external surfaces clean and free from dust. Any accidental spillage should be wiped away immediately. Routine maintenance may include replacement of light source if it fails. Clean Cells.	Normal analysis	Instrument performance and sensitivity	Daily, and as needed	CCV passes QC criteria	Clean and replace parts; recalibrate	Laboratory Manager/ Analyst	100.0208, 100.0308
Automated Colorimetric	**Daily or as needed:** Keep external surfaces clean and free from dust. Any accidental spillage should be wiped away immediately. Routine maintenance may include replacement of tubing if flattened or discolored.	Normal analysis	Instrument performance and sensitivity	Daily, and as needed	CCV passes QC criteria	Clean and replace parts; recalibrate	Laboratory Manager/ Analyst	100.0004

QAPP WORKSHEETS #26 AND #27

Sample Handling, Custody, and Disposal

To ensure sample authenticity and data defensibility, a proper sample handing system will be followed from the time of sample collection to final sample disposal.

Sampling Organization: Contractor (EA)

Laboratory: Designated offsite laboratory (Spectrum Analytical, Inc.)

Method of sample delivery (shipper/carrier): Hand delivery or expedited courier service (i.e., Federal Express)

Number of days from reporting until sample disposal: 60 days

Activity	Organization and Title or Position of Person Responsible for the Activity	SOP Reference
Sample labeling	Field Technician/Contractor	SOP 01
Chain-of-custody (COC) form completion	Field Technician/Contractor	SOP 02
Sample packing and shipping	Field Technician/Contractor	SOP 04
Shipping coordination	Field Technician/Contractor	SOP 04
Sample receipt, inspection, and log-in	Sample log-in staff and laboratory Project Manager/ designated analytical Laboratory	Laboratory specific SOP
Sample custody and storage	Laboratory Project Manager/Designated offsite laboratory	Laboratory specific SOP
Sample disposal	Laboratory Waste Manager/Designated offsite laboratory	Laboratory specific SOP

Sample Custody and Documentation

Sampling information will be recorded on a COC record and in a permanently bound field logbook. The entries will be legible and recorded in indelible ink.

Sample Identification

Sample identification numbers will be affixed to each sample container and entered on the COC record. The sample identification number will uniquely identify the sample in relation to a specified location. A sample identification system has been developed to provide uniform classification and to assist project personnel to interpret data reports and field notes.

Sample Packaging and Shipping

The laboratory will supply sample containers and appropriate preservation additives, if needed. Onsite personnel will be responsible for ensuring that adequate sample containers are available for the work scheduled at the sample collection points. After the appropriate labeling and COC records are completed, the sample containers will be placed in coolers, temperature preserved to $\leq 4^{o}C$ if necessary, and secured for transport to the laboratory.

Environmental samples from this project will be packaged and shipped in a manner that will ensure the safety and accountability of each sample, and the procedures implemented will be in accordance with

applicable federal and local requirements. The persons packing and shipping environmental samples should review and be aware of state, federal, Department of Transportation, and International Air Transport Association regulations governing environmental and hazardous sample packaging. The person(s) shipping the samples is responsible for being in compliance with applicable packaging, labeling, and shipping requirements.

Chain of Custody

Chain of custody documentation is required for each sample to track collection, shipments, laboratory receipt, custody, and disposal. Each individual who has the samples in their possession will sign the chain of custody record. A sample is considered to be in custody under the following conditions:

- It is in actual possession or in view of the person who collected the sample.
- It is locked in a secure area.
- It is placed in an area restricted to authorized personnel.

Each sample will be assigned a unique sample identification number, which will be entered on the chain of custody record. If the samples are transported to an offsite laboratory by a courier service, the courier name and/or air bill number will be noted on the chain of custody record. As a final step, custody seals are attached to the front and back or sides of the lid of the shipping container. Upon arrival at the laboratory, the samples in the cooler are checked against the chain of custody record by laboratory personnel. If discrepancies are noted, the samples in question will be segregated and field personnel will be immediately notified. The person accepting the delivery will sign and date the chain of custody.

QAPP WORKSHEET #28

Analytical Quality Control and Corrective Action

Matrix: Soil/Water
Concentration Level: Low
Sampling SOP: See Worksheet #21
Number of Sampling Locations: See Worksheet #18

TABLE 28-1
Summary of QC Procedures for GC Methods: Pesticides (SW8081B/60.0006), PCBs (SW8082A/60.0003), and Herbicides (SW8151A/60.0034)

QC Sample	Number/ Frequency	Method/SOP QC Acceptance Limits	Corrective Action	Title/Position of Person(s) Responsible for Corrective Action	Project-Specific Measurement Performance Criteria
Method blank	One per analytical batch	No analytes detected >½ LOQ	Identify and correct problem. If necessary, reprepare, and analyze method blank and the samples processed with the contaminated blank.	Analyst/ Supervisor	No analytes detected >½ LOQ
LCS	One LCS per analytical batch	Acceptance criteria: DoD limits or in-house if unavailable.	Identify and correct problem, then reanalyze. If %R is still out, reprepare and reanalyze the LCS and the samples in the affected batch.	Analyst/ Supervisor	%R
MS/MSD	One per 20 samples per matrix as a minimum	Acceptance criteria: DoD limits or in-house if unavailable.	Assess data to determine whether there is a matrix effect or analytical error. Potential matrix effects should be discussed in case narrative of laboratory report.	Analyst/ Supervisor	%R and RPD
Confirmation of positive results	Positive results must be confirmed	Results between primary and second column or detector RPD ≤40%	Sample results with RPD≥40% are flagged with a P on Form 1.	Analyst/ Supervisor	RPD ≤40%
Surrogate spike	Every sample, spiked sample, standard, and method blank	Acceptance criteria: DoD limits or in-house if unavailable.	Identify and correct problem, then reprepare and reanalyze the affected samples. If matrix effect is verified, discuss in case narrative.	Analyst/ Supervisor	%R

TABLE 28-2
Summary of QC Procedures for GC/Mass Spectrometry Methods: VOCS (SW8260C/90.0012), SVOCs (SW8270D/70.0012),and PAHs/1,4-Dioxane (SW8270DSIM/70.0033)

QC Sample	Number/ Frequency	Method/SOP QC Acceptance Limits	Corrective Action	Title/Position of Person(s) Responsible for Corrective Action	Project-Specific Measurement Performance Criteria
IS	Each sample, standard, and QC sample	Retention time ± 30 seconds from retention time of the IS in the ICAL midpoint standard. Extracted ion current profile area within -50% to +100% of area from IS in ICAL mid-point standard.	Inspect mass spectrometer and GC for malfunctions and make corrections as appropriate. Reanalysis of samples that were analyzed while the system was malfunctioning is mandatory.	Analyst/ Supervisor	Retention time ± 30 seconds from retention time of the IS in the ICAL midpoint standard. Extracted ion current profile area within -50% to +100% of area from IS in ICAL mid-point standard.
Method blank	One per analytical batch	No analytes detected > ½ LOQ For common laboratory contaminants, no analytes detected > LOQ.	Identify and correct problem. If necessary, reprepare and analyze method blank and the samples processed with the contaminated blank.	Analyst/ Supervisor	No analytes detected > ½ LOQ For common laboratory contaminants, no analytes detected > LOQ.
LCS	One LCS per analytical batch	Acceptance criteria: DoD limits or in-house if unavailable.	Identify and correct problem, then reanalyze. If %R is still out, reprepare and reanalyze the LCS and the samples in the affected batch.	Analyst/ Supervisor	%R
MS/MSD	Minimum of one set per 20 samples per matrix as a minimum.	Acceptance criteria: DoD limits or in-house if unavailable.	Assess data to determine whether there is a matrix effect or analytical error. Analyze LCS for failed target analytes. Potential matrix effects should be discussed in the case narrative.	Analyst/ Supervisor	%R and RPD
Surrogate spike	Every sample, spiked sample, standard, and method blank	Acceptance criteria: DoD limits or in-house if unavailable. One surrogate allowed out per fraction.	Identify and correct problem, then reprepare and reanalyze the affected samples. If matrix effect is verified, discuss in case narrative.	Analyst/ Supervisor	%R

TABLE 28-3
Summary of QC Procedures for ICP AES/MS Methods: Metals (SW6010C/100.0111) Metals (SW6020A/100.0110)

QC Sample	Number/ Frequency	Method/SOP QC Acceptance Limits	Corrective Action	Title/Position of Person(s) Responsible for Corrective Action	Project-Specific Measurement Performance Criteria
Internal Standard	Each sample, standard, and QC sample	Internal standard intensity limits are 30-120% of the IS in the initial calibration	Evaluate sequence. If not instrument issue, dilute sample five fold and reanalyze.	Analyst/ Supervisor	Intensity of response/matrix issues
Method blank	One per analytical batch	No analytes detected > ½ LOQ For common laboratory contaminants, no analytes detected >LOQ.	Identify and correct problem. If necessary, reprepare and analyze method blank and the samples processed with the contaminated blank.	Analyst/ Supervisor	No analytes detected > ½ LOQ For common laboratory contaminants, no analytes detected >LOQ.
LCS for all analytes	One LCS per analytical batch	Acceptance criteria: DoD QSM	Identify and correct problem, then reanalyze. If %R is still out, reprepare and reanalyze the LCS and the samples in the affected batch.	Analyst/ Supervisor	%R
MS/MSD or sample duplicate	One per 20 samples per matrix as a minimum.	Acceptance criteria: %R DoD QSM. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Assess data to determine whether there is a matrix effect or analytical error. Analyze LCS for failed target analytes. Potential matrix effects should be presented in the laboratory report's case narrative. Outliers will be flagged on forms.	Analyst/ Supervisor	%R RPD
Dilution test	One per preparatory batch.	Five-fold dilution must agree within ± 10% of the original measurement if sample results >50xLOQ.	Perform post-digestion spike addition. Outliers will be flagged on forms.	Analyst/ Supervisor	Five-fold dilution must agree within ± 10% of the original measurement if sample results >50xLOQ.
Post-digestion spike addition	When dilution test fails or analyte concentration in all samples <50 x LOD.	%R within 75-125%.	Note in narrative	Analyst/ Supervisor	%R

TABLE 28-4
Summary of QC Procedures for SPEC Methods: Hexavalent Chromium (SW7196A/100.0208) (SM3500CR+6 B/100.0308)

QC Sample	Number/ Frequency	Method/SOP QC Acceptance Limits	Corrective Action	Title/Position of Person(s) Responsible for Corrective Action	Project-Specific Measurement Performance Criteria
Method blank	One per analytical batch	No Hexavalent chromium>½ LOQ	Identify and correct problem. If necessary, reprepare and analyze method blank and the samples processed with the contaminated blank.	Analyst/ Supervisor	No Hexavalent chromium>½ LOQ
LCS	One LCS per analytical batch	Acceptance criteria: ±20%	Identify and correct problem, then reanalyze. If %R is still out, reprepare and reanalyze the LCS and the samples in the affected batch.	Analyst/ Supervisor	%R
MS/MSi(pre-digestion) one soluble and one insoluble (Soil only)	One per 20 samples per matrix as a minimum and as defined on the COC record	Acceptance criteria: ±25%	Assess data to determine whether there is a matrix effect or analytical error. Potential matrix effects should be presented in the laboratory report's case narrative. Evaluate against LCS recovery	Analyst/ Supervisor	%R
MSDand/or Sample Duplicate	One per 20 samples per matrix as a minimum and as defined on the COC record	MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate-soil only) RPD ≤ 20% Aqueous	Assess data to determine whether there is a matrix effect or analytical error. Potential matrix effects should be presented in the laboratory report's case narrative.	Analyst/ Supervisor	RPD
Post Digestion Spike	One per 20 solid samples at a minimum	Acceptance criteria: ±15%	Assess data to determine whether there is a matrix effect or analytical error. Potential matrix effects should be presented in the laboratory report's case narrative.	Analyst/ Supervisor	%R

TABLE 28-5
Summary of QC Procedures for CVAAS Methods: Mercury (SW7470A/SW7471B/100.0012)

QC Sample	Number/ Frequency	Method/SOP QC Acceptance Limits	Corrective Action	Title/Position of Person(s) Responsible for Corrective Action	Project-Specific Measurement Performance Criteria
Method blank	One per analytical batch	No mercury >½ LOQ	Identify and correct problem. If necessary, reprepare and analyze method blank and the samples processed with the contaminated blank.	Analyst/ Supervisor	No mercury >½ LOQ
LCS	One LCS per analytical batch	Acceptance criteria: DoD QSM.	Identify and correct problem, then reanalyze. If %R is still out, reprepare and reanalyze the LCS and the samples in the affected batch.	Analyst/ Supervisor	%R
MS/MSD or sample duplicate	One per 20 samples per matrix as a minimum.	Acceptance criteria: %R DoD QSM. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Assess data to determine whether there is a matrix effect or analytical error. Potential matrix effects should be presented in the laboratory report's case narrative.	Analyst/ Supervisor	%R and RPD

TABLE 28-6
Summary of QC Procedures for FIA Methods: Cyanide(SW9012B/100.0004)

QC Sample	Number/ Frequency	Method/SOP QC Acceptance Limits	Corrective Action	Title/Position of Person(s) Responsible for Corrective Action	Project-Specific Measurement Performance Criteria
Method blank	One per analytical batch	>½ LOQ	Identify and correct problem. If necessary, reprepare and analyze method blank and the samples processed with the contaminated blank.	Analyst/ Supervisor	> ½ LOQ
LCS	One LCS per analytical batch	Acceptance criteria: ±20%	Identify and correct problem, then reanalyze. If %R is still out, reprepare and reanalyze the LCS and the samples in the affected batch.	Analyst/ Supervisor	%R
MS/MSD or sample duplicate	One per 20 samples per matrix as a minimum.	Acceptance criteria: ±25% MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Assess data to determine whether there is a matrix effect or analytical error. Potential matrix effects should be presented in the laboratory report's case narrative.	Analyst/ Supervisor	%R and RPD

QAPP WORKSHEET #29

Project Documents and Records

The project documentation and records will be maintained by the Contractor Project Manager or designee.

Sample Collection and Field Records			
Record	**Generation**	**Verification**	**Storage location/archival**
Field Logbook or Data Collection Sheets	Field Technician	Field Team Leader	Project File
COC Forms	Field Technician	Field Team Leader	Project File
Air Bills	Field Technician	Field Team Leader	Project File
Contractor Daily QC Reports	QC Officer	Project Manager	Project File
Deviations	QC Officer	Project Manager	Project File
Corrective Action Reports	QC Officer	Project Manager	Project File
Correspondence	Project Team	Project Manager	Project File

Project Assessments			
Record	**Generation**	**Verification**	**Storage location/archival**
Field Audit Checklists	QC Officer	Project Manager	Project File
Data Verification Checklists	Data Validator	Project Manager	Project File
Data Validation Report	Data Validator	Project Manager	Project File
Data Usability Assessment Report	Project Chemist or designee	Project Manager	Project File

Laboratory Records			
Record	**Generation**	**Verification**	**Storage location/archival**
Offsite Laboratory Report and Electronic Data Deliverable (see details below)	Laboratory Project Manager	QA Manager	Contractor and Laboratory Project Files
Documentation of Laboratory Method Deviations, Analytical Audit Checklist, and Lab Assessment	QA Manager	Laboratory Manager	Offsite Laboratory
Laboratory QA Manual	QA Manager	Laboratory Manager	Offsite Laboratory
Laboratory Name and ELAP Accreditation	QA Manager	Laboratory Manager	Offsite Laboratory
LOD Study Information	Analyst and Supervisor	QA Manager	Offsite Laboratory
Instrument Calibration, Initial Precision, and Accuracy Tests	Analyst and Supervisor	QA Manager	Offsite Laboratory
Documentation of Manual Integrations including Chromatograms Showing the Before and After with the Analyst Name and Reason for the Manual Integration.	Analyst and Supervisor	QA Manager	Offsite Laboratory
Sample Chronology (Time of Receipt, Tracking, Extraction, and Analysis) and Associated Forms	Analyst and Supervisor	QA Manager	Offsite Laboratory

The offsite laboratory report will include the information specified in the DoD QSM (DoD 2010), including the information for third-party review, as appropriate to the analytical methodology, as follows:

- Case narrative on laboratory letterhead
- Corrective action reports, if applicable
- Data report for each sample and blank, which will include field and laboratory sample identification, date and time of sample collection, date received, extracted, and analyzed, LOD, DL, LOQ, and dilution factors
- Blank results
- Blank spike forms including acceptance criteria, amount spiked, %R, and RPD, if blank spike duplicates are reported
- Surrogate recovery forms including acceptance criteria
- MS, MSD, and laboratory duplicate results forms including acceptance criteria, original sample results, amounts spiked, %R, and RPD
- Confirmatory results for GC methods and RT windows and shift results and acceptance criteria

- Instrument performance check (tuning) report
- Initial calibration results including acceptance criteria
- Second source and continuing calibration standard results including acceptance criteria
- IS areas and RT reports, including limits and out-of-control flags
- Post-digestion spike recovery reports including acceptance criteria, original sample results, amounts spiked, and %R
- Interference check sample report
- Serial dilution results
- Low-level standard check report
- Interelement correction factor report
- Associated raw data for each sample, blank, spike, duplicates, and standards (i.e., quantitation reports, chromatograms, mass spectra, instrument printouts, and bench sheets)
- Sample preparation/extraction logs
- Extract cleanup logs
- Sample bench sheets
- Analysis run logs.

Data generated during this investigation will be archived in the ERPIMS database. The electronic data will be verified by comparison to the hard copy data packages. Although manual data entry will be avoided if possible, manual entry may be necessary for some field parameters or survey data. If manual data entry is performed, verification will be performed by a second person. The final verified data will be electronically uploaded and stored in the ERPIMS database using guidance and tools located at http://www.afcee.af.mil/resources/restoration/erpims/index.asp.

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QAPP Worksheets #31, #32, and #33

Assessments and Corrective Action

Assessments:

Assessment Type	Responsible Party and Organization	Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Field Sampling Audit	Contractor QA/QC Manager or designee	A field audit will occur once during each type of field activity (i.e., soil sampling, groundwater sampling, etc.) and subsequently if there is a gap of 6 months or more.	Specified in site-specific work plans	Audit Memo	7 days after assessment
Field Documentation Audit	Contractor QA/QC Manager or designee	At the conclusion of the field event.	Specified in site-specific work plans	Audit Memo	7 days after assessment
Laboratory Technical Systems Audit (may be documentation review or onsite)	Contractor Project Chemist	Before the start of sampling and as required by AFCEC.	Specified in site-specific work plans	Audit Memo	Immediate correction – written documentation within 7 days
Performance Evaluation Sample	AFCEC Performance Evaluation Project Manager	As required by AFCEC.	Specified in site-specific work plans	Audit Memo	7 days after assessment
Data Review Technical Systems Audit	Contractor QA/QC Manager or designee	Reviewed data.	Specified in site-specific work plans	Data Evaluation Summary Report	30 days after review
Health and Safety Compliance Audit	Contractor QA/QC Manager or designee	Once.	Specified in site-specific work plans	Quarterly Project Review Summary	Immediate correction – written documentation due within 1 week
Management Systems Review	Contractor QA/QC Manager or designee	Once.	Specified in site-specific work plans	Quarterly Project Review Summary	Immediate correction - written documentation due within 1 week

Assessment Response and Corrective Action:

Assessment Type	Responsibility for Responding to Assessment Findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsible for monitoring Corrective Action implementation
Field Sampling Audit	Field Task Leader	Corrective Action Response (email or memorandum)	7 days from receipt of memorandum	Field Task Leader	Contractor Project Manager or designee
Field Documentation Audit	Field Task Leader	Corrective Action Response (email or memorandum)	7 days from receipt of memorandum	Field Task Leader	Contractor Project Manager or designee
Health and Safety Compliance Audit	Field Task Leader	Corrective Action Response (email or memorandum)	7 days from receipt of memorandum	Field Task Leader	Contractor Project Manager or designee
Laboratory Technical Systems Audit (may be documentation review or onsite)	Offsite Laboratory QA Manager	Corrective Action Response (email or memorandum)	Immediate correction- written documentation within 7 days of receipt of memorandum	Laboratory Manager	Contractor Project Chemist
Performance Evaluation Sample	Offsite Laboratory QA Manager	Corrective Action Response	7 days from receipt of memorandum	Laboratory Technical Director	Contractor Project Chemist
Data Review Technical Systems Audit	Appropriate persons depending on the area of the findings, Contractor	Corrective Action Response (email or memorandum)	21 days for reissuance	Appropriate persons depending on the area of the findings, Contractor	Contractor QA/QC Manager or designee
Management Systems Review	Contractor Project Manager or designee	Corrective Action Response	7 days from receipt of memorandum	As assigned in response	Contractor QA/QC Manager or designee

QAPP WORKSHEET #34

Data Verification and Validation Input

This worksheet is used to list the inputs that will be used during data verification and validation. Inputs include planning documents, field records, and laboratory records. To ensure that scientifically-sound data of known and documented quality are used in making environmental decisions, the following three-step data review will be performed:

- Step I (verification) will confirm that the sampling and analytical requirements have been met.
- Step II (validation) will assess whether the sampling and analytical processes comply with the contract-specific and project-specific requirements.
- Step III (usability assessment) will determine whether the resulting data are suitable as a basis for the decision being made.

Records subject to verification and validation are listed below:

Item	Description	Verification (completeness)	Validation (conformance to specifications)
	Planning Documents/Records		
1	Approved planning documents (i.e., work plan)	**X**	
2	Contract	**X**	
4	Field SOPs	**X**	
5	Laboratory SOPs	**X**	
	Field Records		
6	Field logbooks	**X**	**X**
7	Equipment calibration records	**X**	**X**
8	COC records	**X**	**X**
9	Sampling diagrams/surveys	**X**	**X**
10	Drilling logs	**X**	**X**
11	Geophysics reports	**X**	**X**
12	Relevant correspondence	**X**	**X**
13	Change orders/deviations	**X**	**X**
14	Field audit reports	**X**	**X**
15	Field corrective action reports	**X**	**X**
	Analytical Data Package		
16	Cover sheet (laboratory identifying information)	**X**	**X**
17	Case narrative	**X**	**X**
18	Internal laboratory COC	**X**	**X**
19	Sample receipt records	**X**	**X**
20	Sample chronology (i.e., dates and times of receipt, preparation, and analysis)	**X**	**X**
21	Communication records	**X**	**X**
22	Project-specific PT sample results	**X**	**X**
23	LOD/LOQ establishment and verification	**X**	**X**
24	Standards traceability	**X**	**X**
25	Instrument calibration records	**X**	**X**
26	Definition of laboratory qualifiers	**X**	**X**
27	Results reporting forms	**X**	**X**
28	QC sample results	**X**	**X**
29	Corrective action reports	**X**	**X**
30	Raw data	**X**	**X**
31	Electronic data deliverable	**X**	**X**

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QAPP WORKSHEET #35

Data Verification Procedures

This worksheet establishes the procedures that will be followed to verify project data. Data verification is a completeness check to confirm that the required activities were conducted, the specified records are present, and the contents of the records are complete.

Note that verification often is performed at more than one step by more than one person.

Records Reviewed	Required Documents	Process Description	Responsible Person, Organization
Chain of Custody Records and Shipping Forms	Basewide PBR work plan, and field SOPs 2 and 4	Chain of custody records and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent to verify their completeness and accuracy and consistency with the field logbook. The review will include verification of appropriate analytical methods, preservation, sufficient volume for analyses and necessary QC samples (i.e., MS/MSD), and all required signatures and dates. A check for transcription errors will occur. A copy of the chain of custody retained in the project file, and the original and remaining copies taped inside the cooler for shipment.	Daily – Field Task Leader or designee; at the conclusion of field activities – Project Chemist
Field Notes/ Logbook	Basewide PBR work plan, and field SOPs 16 and 59	Field notes will be reviewed internally for completeness. The review will include verification that records are present and complete for each day of field activities, the planned samples including field QC samples were collected, sample collection locations are documented, and meteorological data were provided for each day of field activities. Review will also include verification that changes/exceptions are documented and were reported in accordance with requirements. If field data are collected, review will verify that results are documented.	Daily – Field Task Leader or designee; at the conclusion of field activities – Contractor QA/QC Officer
Analytical Data Package Deliverables	Basewide PBR work plan	The laboratory deliverable will be reviewed to verify that content contains the records specified in the planning documents. Verification will include check of sample receipt records to ensure sample condition upon receipt was noted, and that missing/broken sample containers were noted and reported according to planning documents. The data package will be compared to chain of custody records to verify that results were provided for the collected samples. The narrative will be reviewed to ensure that QC exceptions are described. The review will verify that necessary signatures and dates are present.	Before release of data package – Laboratory QA Manager Upon receipt – Contractor Project Chemist
Audit Reports	Basewide PBR work plan	The project QA/QC Manager will verify that all planned audits were conducted and examine audit reports. For any deficiencies noted, the QA/QC Manager will verify that corrective action was implemented according to plan.	Project QA/QC Manager

Analytical data verification, a sample-specific assessment, will be performed concurrent with analytical data validation (see Worksheet #36) to determine whether the requested sample information has been gathered; assess sample data for conformance with the applicable QC requirements; and assess sample data for compliance with the quality objectives of this QAPP and contract requirements. All relevant data inputs are reviewed to ensure that the laboratory data packages (analytical report and raw data) are complete and adequate for data verification and validation. Data packages must contain the necessary items to completely document the analytical testing, and it must be sufficient for a method-specific definitive

data validation. The data verifier will evaluate whether: (1) samples were collected and analyzed for the list of analytes and the specific test methods specified in the project plan; (2) sample detection limits met project-specific objectives; (3) analytical holding times were met; and (4) measurement acceptance criteria for QC samples were met. Sample-specific data verification will be performed on all field samples collected and analyzed for definitive data. Worksheet #34 contains a list of potential data verification input items for the data verification process.

QAPP WORKSHEET #36

Data Validation Procedures

Data Validator: SGD Environmental

Analytical Group/Method:	See WS #12
Data deliverable requirements:	See WS #29
Analytical specifications:	See WS #23
Measurement performance criteria:	See WS #10, 12, 15, 20, and 28
Percent of data packages to be validated	100% of the laboratory data packages consisting of definitive data
Validation Procedure:	See Analytical Data Validation Process below

Data validation is an analyte and sample-specific process for evaluating compliance with contract requirements, methods/SOPs, and method performance criteria.

The objectives of the data validation review step are to: (1) quantitatively reproduce a subset of the analytical results while confirming that the specified analytical procedures were followed as written, and (2) ensure that data qualifiers have been applied appropriately and consistently based on the DQIs used to assess the measurement quality objectives for the project. The level of data validation will include 100% of all data packages. Definitive data will be validated according to the general requirements for chemical measurements and using the specific analytical method criteria listed in Appendix F of the DoD QSM Version 4.2 as applicable (DoD 2010). Worksheet #34 contains a list of potential data validation input items for the data validation process.

The data validator will document the actual level of data verification and validation in each data review report using the recommended terminology and labels for communicating the stages and processes used for laboratory analytical data verification and validation in *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (EPA 540-R-08-005, January 2009). Final data qualifier flags will be consistent with those listed in the DoD QSM version 4.2, and include:

- U – The analyte was not detected and is reported as less than the LOD.
- J – The reported result is an estimated value.
- B – The recorded result is associated with a contaminated blank.
- Q – One or more QC criteria failed (used by the laboratory only; final usability qualification will be determined by the data reviewer)

Note that third party data validation will not include the rejection of data (noted by the designation of the "R" data qualifier), but will rather state when the performance criteria are not met. The final rejection of data and their use is a decision reserved specifically for the project team.

The data validators that will evaluate project data include the Contractor Project Chemist and/or a third-party validator.

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QAPP WORKSHEET #37

Data Usability Assessment

This worksheet documents procedures that will be used to perform the data usability assessment. The data usability assessment is performed at the conclusion of data collection activities, using the outputs from data verification and data validation. It is the data interpretation phase that involves a qualitative and quantitative evaluation of environmental data to determine if the project data are of the right type, quality, and quantity to support the decisions that need to be made. It involves a retrospective evaluation of the systematic planning process and, like the systematic planning process, involves participation by key members of the project team. The data usability assessment evaluates whether underlying assumptions used during systematic planning are supported, sources of uncertainty have been accounted for and are acceptable, data are representative of the population of interest, and the results can be used as intended, with the acceptable level of confidence.

The personnel that are responsible for participating in the data usability assessment include the following, as applicable to project scope:

- Contractor Project Manager
- Project QA Manager
- Risk Assessor
- Geologist
- Hydrogeologist
- Project Chemist
- Field Task Leader
- Statistician.

The following steps will be followed during the data usability assessment process:

Step 1	**Review the project's objectives and sampling design** *Review the key outputs defined during systematic planning (i.e., project quality objectives or data quality objectives and measurement performance criteria) to make sure they are still applicable. Review the sampling design for consistency with stated objectives. This provides the context for interpreting the data in subsequent steps.*
Step 2	**Review the data verification and data validation outputs** *Review available QA reports, including the data verification and data validation reports. Perform basic calculations and summarize the data (using graphs, maps, tables, etc.). Look for patterns, trends, and anomalies (i.e., unexpected results). Review deviations from planned activities (e.g., number and locations of samples, holding time exceedances, damaged samples, non-compliant PT sample results, and SOP deviations) and determine their impacts on the data usability. Evaluate implications of unacceptable QC sample results.*
Step 3	**Verify the assumptions of the selected statistical method** *Verify whether underlying assumptions for selected statistical methods (if documented in the QAPP) are valid. Common assumptions include the distributional form of the data, independence of the data, dispersion characteristics, homogeneity, etc. Depending on the robustness of the statistical method, minor deviations from assumptions usually are not critical to statistical analysis and data interpretation. If serious deviations from assumptions are discovered, then another statistical method may need to be selected.*
Step 4	**Implement the statistical method** *Implement the specified statistical procedures for analyzing the data and review underlying assumptions. For decision projects that involve hypothesis testing (e.g., "concentrations of lead in groundwater are below the action level"), consider the consequences for selecting the incorrect alternative; for estimation projects (e.g., establishing a boundary for surface soil contamination), consider the tolerance for uncertainty in measurements.*

Step 5	**Document data usability and draw conclusions** *Determine if the data can be used as intended, considering implications of deviations and corrective actions. Discuss data quality indicators. Assess the performance of the sampling design and Identify limitations on data use. Update the conceptual site model and document conclusions. Prepare the data usability summary report, which can be in the form of text and/or a table.*

The following sections describe how the usability assessment will be documented.

Precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity are the DQIs used to assess the data produced during the project. Each DQI is described below, including a definition of the terminology, the referenced process for calculating the indicator, and the referenced measurement performance criteria. A description of how the DQIs should be incorporated into the usability section is found under each parameter heading.

Precision—Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance, %D, or range, in either absolute or relative terms. The QC measures for precision include field duplicates, laboratory duplicates, MS, MSDs, and analytical replicates. To meet the needs of the data users, project data must meet the measurement performance criteria for precision specified in QAPP Worksheet #12, Measurement Performance Criteria, and supporting worksheets.

Precision may be the result of one or more of the following: field instrument variation, analytical measurement variation, poor sampling technique, sample transport problems, or spatial variation (heterogeneous sample matrices). To identify the cause of imprecision, the field sampling design rationale and sampling techniques will be evaluated by the reviewer, and both field and analytical duplicate/replicate sample results will be reviewed. The process for calculating precision is detailed in and will be in accordance with the UFP-QAPP Manual, Section 2.6.2.1 (EPA 2005). If poor precision is indicated in both the field and analytical duplicates/replicates, then the laboratory may be the source of error. If poor precision is limited to the field duplicate/replicate results, then the sampling technique, field instrument variation, sample transport, and/or spatial variability may be the source of error.

The usability report will:

- Discuss and compare overall field duplicate/replicate precision data from data collected for the project for each matrix, analytical group, and concentration level.

- Discuss and describe the limitations on the use of project data when overall precision is poor or when poor precision is limited to a specific sampling or laboratory (analytical) group, data set or sample delivery group, matrix, analytical group, or concentration level.

Accuracy/Bias—Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) that are due to sampling and analytical operations. Examples of QC measures for accuracy include MS, surrogates, and LCSs. In order to meet the needs of the data users, project data must meet the measurement performance criteria for accuracy/bias specified in QAPP Worksheet #12, Measurement Performance Criteria and associated worksheets. The process for calculating accuracy/bias is detailed in, and will be in accordance with, the UFP-QAPP Manual, Section 2.6.2.2 (EPA 2005).

The usability report will:

- Discuss and compare overall contamination and accuracy/bias for data collected for the project for each matrix, analytical group, and concentration level.

- Describe the limitations on the use of project data if extensive contamination and/or inaccuracy or bias exists, or when inaccuracy is limited to a specific sampling or laboratory group, data set or sample delivery group, matrix, analytical group, or concentration level.

- Discuss the impact of any qualitative and quantitative trends in bias on the sample data.

***Representativeness*—**Representativeness is the measure of the degree to which data accurately and precisely represent a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition. To meet the needs of the data users, project data must meet the measurement performance criteria for sample representativeness specified in the QAPP Worksheet #12, Measurement Performance Criteria and associated worksheets. The process for calculating representativeness is detailed in, and will be in accordance with, the UFP-QAPP Manual, Section 2.6.2.4 (EPA 2005).

If field duplicate precision checks indicate potential spatial variability, additional scoping meetings and subsequent re-sampling may be needed in order to collect data that are more representative of a non-homogeneous site.

The usability report will:

- Discuss and compare overall sample representativeness for each matrix, analytical group, and concentration level.

- Describe the limitations on the use of project data when overall non-representative sampling has occurred, or when non-representative sampling is limited to a specific sampling, group, data set or sample delivery group, matrix, analytical group, or concentration level.

***Comparability*—**Comparability is the degree to which different methods, data sets, and decisions agree or can be represented as similar. Comparability describes the confidence (expressed qualitatively or quantitatively) that two data sets can contribute to a common analysis and interpolation. In order to meet the needs of the data users, project data must meet the measurement performance criteria for comparability specified in QAPP Worksheet #12, Measurement Performance Criteria and associated worksheets.

Additional information regarding the process of assessing comparability is detailed in, and will be in accordance with, UFP-QAPP Manual, Section 2.6.2.5 (EPA 2005). Different situations require different assessments of comparability, as in the following:

- If two or more sampling procedures or sampling teams will be used to collect samples, describe how comparability will be assessed for each matrix, analytical group, and concentration level.

- If two or more analytical methods or SOPs will be used to analyze samples of the same matrix and concentration level for the same analytical group, the comparability will be assessed between the two data sets and discussed.

- If replicate samples are analyzed, the specific method and %D formula that will be used to assess replicate sample comparability for individual data points will be discussed.

The usability report will:

- Discuss and compare overall comparability for the project for each matrix, analytical group, and concentration level.

- Discuss if screening data will be confirmed by definitive methods, and document the specific method and %D formula that will be used to assess comparability for individual data points.

- Document overall comparability, describe the procedures used to perform overall assessment of comparability, and include mathematical and statistical formulas for evaluating screening and confirmatory data comparability.

- Discuss if the project is long-term monitoring; project data should be compared with previously generated data to ascertain the possibility of false-positives and false negatives, and positive and negative trends in bias. Data comparability is extremely important in these situations.

- Discuss anomalies detected in the data that may reflect a changing environment or indicate sampling and/or analytical error. Comparability criteria should be established to evaluate these data sets to identify outliers and the need for re-sampling as warranted.

- Describe the limitations on the use of project data when project-required data comparability is not achieved for the overall project or when comparability is limited to a specific sampling or laboratory group, data set or sample delivery group, matrix, analytical group, or concentration level.

- Document the failure to meet screening/confirmatory (onsite and offsite data) comparability criteria and discuss the impact on usability.

- Document the failure to meet replicate sampling comparability criteria and discuss the impact on usability.

- If data are not usable to adequately address environmental questions or support project decision-making, address how this problem will be resolved and discuss the potential need for re-sampling.

- If long-term monitoring data are not comparable, address whether the data indicate a changing environment, or are a result of sampling or analytical error.

Sensitivity and Quantitation Limits—Sensitivity is the capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. Examples of QC measures for determining sensitivity include laboratory fortified blanks, DL and LOD studies, and low level calibration standards. In order to meet the needs of the data users, the project data must meet the measurement performance criteria for sensitivity and project DLs specified in Worksheet #12, Measurement Performance Criteria and associated worksheets. The process for assessing sensitivity is detailed in the UFP-QAPP Manual, Section 2.6.2.3 (EPA 2005).

The laboratory will establish a DL, typically the MDL, using a scientifically valid and documented procedure. The MDL is the minimum concentration of a substance that can be measured and reported

with 99 percent confidence that the analyte concentration is greater than zero. The DL is the laboratory's "best case" sensitivity for a given analytical method. The laboratory may establish MDLs for each method, matrix, and analyte for each instrument the laboratory plans to use for the project using the statistical method presented in the 40 Code of Federal Regulations Part 136, Appendix B.

The LOD will be established quarterly by spiking a blank matrix at from two to three times the DL for single analyte standards or from one to four times the DL for multi-analyte standards. This spike concentration is the LOD for each analyte and is specific for each matrix, method, and instrument.

The LOQ will be determined at least quarterly for each analyte of concern following a documented procedure at the laboratory. The validity of the LOQ will be determined by the analysis of a QC sample containing the analyte at from one to two times the estimated LOQ and within the calibration range of the instrument. The LOQ is valid if the recovery of the analyte is within the test methods acceptance recovery limits for accuracy.

A general summary of the relationship between the DL terms used above is presented below:

$$DL < LOD \leq LOQ \leq RL$$

The following requirements apply to the determination of DL, LOD, and LOQ:

- The apparent signal to noise ratio at the LOD must be at least three and in the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). If no measurement of noise is available for a given method, then the LOD must yield a result that is at least three standard deviations greater than the mean blank concentration.

- If multiple instruments are used, the lab must verify the DL, LOD, and LOQ on each.

- If the LOD verification fails, then the laboratory must repeat the DL and LOD determinations at higher concentrations.

- The laboratory will maintain documentation of the DL, LOD, and LOQ studies and these measures of instrument sensitivity will be performed at least quarterly.

- A non-detectable result will be reported as less than the LOQ. The "F" flag will be applied to the detectable results that fall between the DL and the LOQ, indicating the variability associated with the result. No detectable results will be reported below the DL.

The usability report will:

- Discuss and compare overall sensitivity from multiple data sets collected for the project for each matrix, analytical group, and concentration level.

- Discuss the impact of that lack of sensitivity or higher detection limits on data usability; if information is available, indicate that sensitivity or project-specific detection limits were not achieved.

- Describe the limitations on the use of project data if project-required sensitivity and detection limits are not achieved for the project data, or when sensitivity is limited to a specific sampling or

laboratory group, data set or sample delivery group, matrix, analytical group, or concentration level.

Completeness—Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under correct, normal circumstances. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. The completeness target is 90 percent.

Completeness measures the effectiveness in sample collection, analysis, and result reporting of the entire investigation, and is calculated on a per-analyte basis by the following equation:

$$\%Completeness = \frac{Number\ of\ valid\ results}{Number\ of\ possible\ results} \times 100$$

For any instances of samples that could not be analyzed for any reason (holding time violations in which resampling and analysis were not possible, samples spilled or broken, etc.), the numerator of this calculation becomes the number of possible results minus the number of possible results not reported.

A completeness check will be done on the data generated by the laboratory. For each analyte, completeness will be calculated as the number of data points for each analyte that meets the measurement performance criteria for precision, accuracy/bias, and sensitivity, divided by the total number of data points for each analyte. A discussion will follow summarizing the calculation of data completeness. Conclusions about the completeness of the data for each analyte will be drawn and any limitations on the use of the data will be described.

For this project, 90 percent of usable sample data is considered the minimal acceptance criteria for completeness; the goal is to achieve 100 percent completeness.

The usability report will:

- Discuss and compare overall completeness for each matrix, analytical group, and concentration level

- Describe the limitations on the use of project data if project-required completeness is not achieved for the overall project, or when completeness is limited to a specific sampling or laboratory group, data set or sample delivery group, matrix, analytical group, or concentration level.

Activities

The entire project team will participate in the performance of the usability assessment to ensure that the project quality objectives are understood and the full scope is considered. If, for whatever reason, (precision, accuracy/bias, comparability, sensitivity, completeness) measurement performance criteria are not achieved and it has been determined that certain project data are not usable, then the project team will determine if it is necessary to take further action, i.e., resampling, to ensure that the data quality objectives have been met.

Assessment Documentation

A usability report will be written that discusses precision, accuracy/bias, representativeness, comparability, and completeness as detailed within this worksheet. This narrative report will include worksheets and supporting documentation to assess the project quality objectives and any conclusions and limitations of the associated data. The specific details of each section of the usability assessment documentation can be found above under the individual data quality indicators.

Considerations for Usability Assessment

The following items shall be considered during the performance of the data usability assessment:

- ***Data Deliverables and QAPP, Deviation—***Ensure that the necessary information was provided.
- ***Deviations—***Determine the impact of deviations on the usability of data.
- ***Sampling Locations—***Determine if alterations to sample locations continue to satisfy the project objectives.
- ***Chain of Custody Record—***Establish that problems with documentation or custody procedures do not prevent the data from being used for the intended purpose.
- ***Holding Times—***Determine the acceptability of data if holding times were exceeded.
- ***Damaged Samples—***Determine whether the data from damaged samples are usable. If the data cannot be used, determine whether resampling is necessary.
- ***SOPs and Methods—***Evaluate the impact of deviations from SOPs and specified methods on data.
- ***QC Samples—***Evaluate the implications of unacceptable QC sample results on the data usability for the associated samples. For example, consider the effects of observed blank contamination.
- ***Matrix—***Evaluate matrix effects (interference or bias). For example, consider the effects of observed matrix spike recoveries.
- ***Meteorological Data and Site Conditions—***Evaluate the possible effects of meteorological (e.g., wind, rain, and temperature) and site conditions on sample results. Review field reports to identify whether any unusual conditions were present and how the sampling plan was executed.
- ***Comparability—***Ensure that results from different data collection activities achieve an acceptable level of agreement.
- ***Completeness—***Evaluate the impact of missing information. Ensure that enough information was obtained for the data to be usable (completeness as defined in project quality objectives documented in the QAPP).
- ***Background—***Determine if background levels have been adequately established (if appropriate).

- ***Critical Samples*—**Establish that critical samples and critical target analytes, as defined in the QAPP, were collected and analyzed. Determine if the results meet criteria specified in the QAPP.

- ***Data Restrictions*—**Describe the exact process for handling data that do not meet project quality objectives (i.e., when measurement performance criteria are not met). Depending on how those data will be used, specify the restrictions on use of those data for environmental decision making.

- ***Usability Decision*—**Determine if the data can be used to make a specific decision considering the implications of all deviations and corrective actions.

- ***Usability Report*—**Discuss and compare overall precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity for each matrix, analytical group, and concentration level. Describe limitations on the use of project data if criteria for data quality indicators are not met.

Appendix A
Spectrum Analytical, Inc.
Certifications
and Quality Assurance Manual

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APPENDIX A-1
Spectrum Analytical, Rhode Island Certifications

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January 2013

Spectrum Analytical, Inc.

Statement of Qualifications

SPECTRUM ANALYTICAL, INC.

Featuring

HANIBAL TECHNOLOGY

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8405 Benjamin Road, Suite A · Tampa, FL 33634 · 1-888-497-5270 · FAX 813-889-7128
646 Camp Avenue · North Kingstown, RI 02852 · 401-732-3400 · FAX 401-732-3499
6437 Collamer Road · East Syracuse, NY 13057 · 315-214-5777· FAX 315-314-7938
www.spectrum-analytical.com

Table of Contents

Introduction

Spectrum Analytical, Inc. including its Divisions is committed to providing quantitative analysis for an extensive range of organic and inorganic compounds in soil, water, and air samples as well as petroleum products. We are dedicated to supply quality analytical data to consulting firms, industries, municipalities, universities, and the public sector in a timely manner.

To meet the evolving needs of our clients, we have dramatically expanded our headquarters in Agawam, MA and acquired the assets of Mitkem Laboratories in Warwick, RI as well as PEL in Tampa, FL, while making significant investments in progressive technology and maintaining stringent Quality Assurance/Quality Control (QA/QC) standards. In 2011 a New York satellite office was added to oversee the regional client service functions relating to bottle orders, courier service including sample pickup and bottle drop offs, sample login, project tracking and invoicing.

Spectrum's Agawam headquarters occupies two buildings, which cover over 25,000 square feet of laboratory space. The administrative headquarters, VOC and Air departments operate from a professionally designed state-of-the-art facility that is isolated from all other operational laboratories. This prevents cross-contamination commonly found in the environmental laboratory industry. Connected by all services, there is no interruption to communication and the transfer of samples and data between buildings is seamless. Certifications include MA DEP, NELAC and DOD.

Spectrum's Rhode Island Division's, 20,400 square foot laboratory space also separates the VOC department from other operational areas to eliminate cross-contamination. RI Division certifications include EPA CLP, DoD and NELAC.

The Florida Division of Spectrum Analytical, performs analyses in its fully equipped 14,000 square foot location also includes an isolated VOC Department from other laboratory areas. Methods are certified under NELAC, CLP, DoD, AFCEE and Navy protocols.

All three Divisions provide analytical services for soil, solid and hazardous wastes, ground water, and industrial/ municipal wastewater. Agawam matrices include air and potable water and the Florida and Rhode Island Divisions perform analysis on plant and animal tissue as well. Forensic geochemistry analysis and treatability bench studies are specialized services that are offered in addition to standard environmental tests.

As we continue to grow in size and capability we have never waivered from our commitment to build trusted relationships with our clients based on personal communication, responsiveness and availability during emergency situations. Volume discount prices, specialized analytical services, and expedited turnaround times underscore our dedication to customer satisfaction. The advanced analytical instrumentation and improved processes we employ reduce completion time and streamline internal scheduling to quicken our response time. We proactively work with agencies to modify and improve devised methods and help clients implement these methods while complying with ever-changing regulations.

Statement of Policy and Capabilities

Spectrum's successful business philosophy is based on three criteria: internal employee support, client support and satisfaction, and quality data.

Internal Employee Support - Spectrum's commitment extends not only externally to its clients and community, but also internally to support our employees. This commitment focuses on communication and has resulted in the formation of policies for performance and resources for employees. These policies enforce implementation of the quality assurance plan and correct deficiencies that may develop, without jeopardizing the quality of services. This commitment ensures unnecessary pressures are not added to the already existing demands that employees of Spectrum Analytical face to provide quality work.

Client Support - Spectrum is committed to providing its clients with prompt, reliable results at competitive prices. The commitment to support our clients can be demonstrated through the services provided, many at no additional charge. These services are listed below:

- Rush turnaround is always available. A Rush Analyses Request should be submitted a minimum of 24 hours prior to sample receipt in the laboratory. This request may be completed online through our web site. Emergency responses will be handled on a case- by-case basis. Spectrum provides 24-hour emergency response assistance. If services are needed outside of normal business hours, simply dial 413-789-9018, press option 4 to access our emergency response system and leave a brief message regarding the nature of the emergency. A member of our emergency response team will immediately process your call.

- Pre-cleaned, pre-preserved sample containers are supplied at no cost provided they return for laboratory analyses. Please allow a minimum of 48 business hours notice to prepare and process your request. Requisitions may be completed online through our web site. Spectrum will ship sample containers to your office at no additional charge provided sufficient notice is given for sample kit preparation and shipping or delivery. Additional notice is required for certified low-level air media.

- Spectrum provides analysis of trip blanks (VOCs and Gasoline Range Organics [GRO] only) and one site-specific MS/MSD free of charge.

- Data is delivered electronically at no additional charge. A specific EDD format can be provided, however this should be noted on the chain of custody along with a valid e-mail address.

- All laboratory reports will be posted online and will be securely accessible 24/7 through our eServices web page at www.spectrum-analytical.com/eservices/. All laboratory reports are posted online in pdf and other EDD formats as requested. An electronic notice is sent to the project manager listed on the Chain of Custody once the laboratory report is available.

- Electronic Data Deliverable (EDD) files are automatically generated by our LIMS, and can be used to import results into your own software and data management applications. Several EDD formats are available and Spectrum is happy to work with clients to provide customized EDD formats.

Certifications

State	Spectrum Analytical, Massachusetts Main Headquarters:	Spectrum Analytical, Rhode Island Division:	Spectrum Analytical, Florida Division:
Arkansas			✓
California			✓
Connecticut	✓	✓	
Delaware		✓	
Florida	✓	✓	✓
Kansas			✓
Louisiana			✓
Maine	✓	✓	
Massachusetts	✓	✓	
Montana	✓		
New Hampshire	✓	✓	
New Jersey	✓	✓	
New York	✓	✓	
North Carolina		✓	
North Dakota			✓
Pennsylvania	✓		
Rhode Island	✓	✓	
South Carolina			✓
Texas			✓
Utah	✓		
Vermont	✓		
Department of Defense - ELAP	✓	✓	✓
US EPA CLP program: Organic contract		✓	✓
US EPA CLP program: Inorganic contract		✓	
USDA	✓	✓	✓

Copies of our current certifications are available from our "Quality" web page at www.spectrum-analytical.com. There are many states that offer reciprocal certification with thirty days notice. Also, certification may be attained for an unlisted state on a project specific basis.

Corporate and Individual Ethics

Spectrum Analytical, Inc. knows its reputation depends upon the quality of the data and services produced as well as the integrity of the people who generate them. We recognize the need for an ethics program that is designed to establish meaningful context within the environmental laboratory. The objective is to provide an effective ethics program that involves training, managerial leadership and active dialogue between our staff.

We at Spectrum Analytical, Inc. believe that we share common goals and values. These goals and values include protection of the environment, quality of product, and personal integrity. Our corporate and individual ethics and data integrity program is outlined in our Quality Assurance Manual.

Confidentiality and Security

The protection of confidential business information and trade secrets is vital to the interest and success of this organization and its clientele. Confidentiality is an absolute condition of employment with Spectrum Analytical. At all times and occasions, it is imperative that all employees maintain confidentiality of any information concerning business practices, vendors, clients, site locations and analytical results.

Security also includes the use of telephones, computers, facsimiles, and any other electronic media systems. Passwords are assigned to each user and the use of all systems can be monitored. Each employee must agree to the non-disclosure terms set forth within our Comprehensive Quality Assurance Manual and Employee Handbook, and signed non-disclosure agreement forms are kept on file for each employee.

Quality Assurance / Quality Control

Spectrum follows well-defined EPA and NELAC standards for validation and data accuracy as the foundation of our QA/QC effort. The details of Spectrum's quality assurance program are documented in each Division's Quality Assurance Manual, which is available upon request. The primary QA/QC objective is to develop, implement and maintain procedures for sample receiving, sample preparation, laboratory analysis, data validation and reporting that provide scientifically valid, legally defensible data. Spectrum supports this objective with QA/QC procedures throughout the laboratory, including all ancillary departments.

Our use of reliable and technically sound instrumentation, the experience of our chemists, and our well-trained support staff are core components to our QA/QC program.

Organization and Responsibilities

Spectrum Analytical, Inc. employs a team of professionals who demonstrate and possess a high-level of training, along with technical experience in air methodologies, organic GC and GC/MS, and inorganic methods including wet chemistry and microbiology. Our staff utilizes their scientific and technical expertise to service the analytical and informational needs of our clients. These staff diversities enable Spectrum to produce high-quality data while maintaining efficiency and effective deliverables. Spectrum encourages its staff to revise and develop procedures that will improve the overall function within the laboratory with still adherence to all applicable QA/QC requirements. Current staff qualifications and work experience are on file and may be furnished upon request.

Organizational Structure

President-CEO
Oversees all aspects of business operations and development and ensures corporate policies are followed. Works closely with each Laboratory Director for budget planning and decisions regarding capital assets and staffing. Key component of the corporate marketing team.

Board of Directors

CFO
Responsible for managing the financial planning of the corporation.

Laboratory Director
Supervises laboratory operations, oversees all technical and administrative policies and procedures, as well as the enforcement and adherence to said policies by laboratory staff, provides final data and report validation, and supervises/approves methodology revision. Also, assists with marketing and planning the yearly budget and capital asset purchases.

Marketing
Responsible for securing MSA and Preferred Vendor agreements with clients and pursuing new contracts and bids, as well as ensuring each Division is aware of specific data deliverable requirements for new projects.

Accounting and Human Resource Department
Tracks all incoming vendor invoices, outgoing client invoices, payables and receivables while carrying out all human resource requirements and maintains vendor contracts.

Quality Assurance Department
Maintains overall laboratory quality assurance and certification status, conducts internal laboratory audits to assure compliance with all aspects of the Quality Assurance Manual, maintains QA/AC files and data base and performs data validation.

Deputy Directors
Oversees administrative and operational departments, assists with marketing, provides technical support and coordinates any operational issues with Laboratory Director. Oversees instrumentation maintenance and offers quality assurance support.

Quality Services Section
Oversees overall client service management including data reporting, invoice preparation, scheduling of courier service, laboratory data report publishing, and client communication.

Organic and Biochemistry Sections
Performs sample preparation and analysis in accordance with all applicable methodology and internal SOPs, processes data generated by laboratory instrumentation and maintains communication with QA Department regarding quality issues and data deliverable while adhering to Health and Safety and Chemical Hygiene Plans.

Sample and Courier Departments
Verifies the integrity of all samples received, inspects incoming Chains of Custody for completeness and accuracy, ensures all inter-company communication regarding incoming expedited TAT samples and short holding time samples, ensures data entry accuracy from Chain of Custody into LIMS database, communicates with clients regarding any COC deviations and prepares samples for subcontract laboratories (when necessary). Coordinates daily pick up schedule and oversees sample container inventory and preparation of sample kits.

Health and Safety Department
Implements the Chemical Hygiene Plan, enforces laboratory safety rules, coordinates staff safety training, and stores and disposes of samples and waste.

Information Systems Department
Maintains all network functions including the web-site and computer program development, as well as on-line services for clients.

Training

Spectrum Analytical's training program applies to all employees. All job functions are described in a formal job description, which is kept on file with the Human Resources Department. To be hired or promoted, an employee must meet all job description requirements. All hiring and subsequent changes in personnel are documented in the individual's personnel file. Training programs are provided for new employees or employees transferred to a new position. Additionally, certain positions require auxiliary training, including training videotapes, on-site training classes, or off-site attendance of specialized training or certification courses. Each analyst hired must perform an initial demonstration of capability before processing and reporting data; this is used to demonstrate the precision and accuracy of the individual.

Key Contacts for More Information

Spectrum Analytical, Massachusetts Main Headquarters:

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Project Experience

Spectrum Analytical, Massachusetts Main Headquarters:

Project Location: Bronx, New York
Client: Langan Engineering & Environmental Services
Description of Work: Analytical services were provided for a large scale, long duration remediation project in the Bronx. Analytical testing included a full suite of organic and inorganic parameters as well as hazardous waste characterization.

Project Location: Stamford, CT
Client: Loureiro Engineering Associates
Description of Work: Laboratory services for on-going investigation and remediation activities were conducted in accordance with the Connecticut Department of Environmental Protection's Reasonable Confidence Protocols (RCP) program. Analyses included a full array of organic and petroleum testing as well as testing for metals in soil/solid waste and aqueous sample matrices.

Project Location: Fort Montgomery, New York
Client: GES, Inc.
Description of Work: This assessment and remediation project for a major petroleum retail company has been ongoing since 2001. Spectrum Analytical has been providing laboratory services for this project that requires period sampling of 100 carbon treatment systems and approximately 75 residential tap locations. Samples are submitted for analysis of VOCs by EPA method 524.2, many times with a request for expedited results.

Project Location: Hartford, Connecticut
Client: Environmental Resources Management
Description of Work: Spectrum Analytical provided laboratory services for a large site investigation/ remediation project. The site was an Establishment as defined in the Connecticut Transfer Act and needed to comply with the Remediation Standard Regulations action levels. Spectrum provided all analytical services for sample matrices including soil, groundwater, drinking water and solder samples. The results for the majority of the samples were requested on an expedited turn-around and Spectrum met the client deadlines. Contaminants have included petroleum, VOC and SVOC compounds with metals analysis required for solder samples collected from interior drinking water piping network.

Contract Award: The State of Connecticut, DAS
Client: Various consulting firms and state agencies
Description of Work: Spectrum Analytical is an approved contractor under the State of Connecticut, DAS contract award no. 04PSX0173, and has provided analysis for various consulting firms and State agencies for many sites. This work consists of providing chemical analyses of water, soil, air and waste liquid. The laboratory furnishes all technical personnel, necessary labels, sample containers, and services, including courier service, in accordance with the terms of the contract.

Project Location: Yankee Nuclear Power Station
Contact: U.S. Environmental Protection Agency
Description of Work: Spectrum performed full organic and inorganic (non-radiological) testing in support of the environmental site closure investigations at Yankee Nuclear Power Station in Rowe, MA under the supervision of the US EPA.

International services
Client: AECOM Environment
Description of Work: Spectrum has conducted analytical services for several countries including Bolivia, Puerto Rico, Dominican Republic, Mexico and the Bahamas. This included VOC and TPH analysis of approximately 2500 samples over a 3 month time period for 72- hour RUSH results.

Spectrum Analytical, Rhode Island Division:

Project Location: Cornell Dubilier Superfund Site
Client: Sevenson Environmental
Description of Work: Provide 24-hour or same-day turnaround time analytical services for remediated soil, primarily for PCB, with somewhat longer (2-day) turnaround services for VOC, SVOC, Pesticides and Metals analyses. Analyze treated soil, extent of excavation soil, treated water and miscellaneous other site samples to maintain the rapid pace of this remediation. Final data packages are all delivered in Level 4-CLP type format full validation deliverables.

Project Location: EPA Contract Laboratory Program
Client: U.S. Environmental Protection Agency
Description of Work: Spectrum Analytical's Rhode Island Division, formerly Mitkem Laboratories, has been under contract with USEPA for volatile organics, semivolatile organics and pesticide/PCBs continuously since 1996 and for inorganics since 2010. Spectrum Analytical is one of few laboratories providing CLP lab services to the EPA for both organics and inorganics analyses. Results are reported on several turnaround times depending on project needs. All analyses are performed in strict compliance with the EPA SOM01 organic and ISM01 SOW requirements. On occasion modified analyses are performed to meet the needs of a specific unique technical scope of work. All data packages are subjected to contract compliance screening audit by the client. In a recent quarterly performance assessment, Spectrum Analytical was ranked as the top laboratory in the country in this program in terms of technical and data package compliance scores.

Project Location: NYDEC State Superfund Standby Contract
Client: Dvirka and Bartilucci Consulting Engineers
*Description of Work :*Spectrum provides analytical services under subcontract to Dvirka and Bartilucci Consulting Engineers to support their performance of hazardous waste investigation and environmental services to the New York State Department of Environmental Conservation. This involves the analysis of soil, sediment, surface water and waste material samples as needed. Upon completion of each sample delivery group (SDG), Spectrum provides full data packages in the EPA Contract Laboratory Program (CLP)-NYS ASP format and electronic data deliverables (EDD's). Under this contract, Spectrum provides analyses for a large list of parameters using EPA and NYSDEC Analytical Services Protocol (ASP) approved procedures. In addition to Dvirka & Bartilucci, Spectrum also provides analytical support to nine (9) other engineering firms under their NYSDEC State Superfund Standby Contracts.

Project Location: New York City DEP Water Supply Dependability Strategy
Client: CDM - Hazen & Sawyer Joint Venture, New York
Description of Work: Spectrum Analytical played a key role in providing laboratory analyses in support of the study of groundwater wells in Jamaica, Queens. Spectrum's Rhode Island Division initially was involved with sample analysis only, but our role in the program has expanded to include all three Spectrum laboratory facilities as well as the providing sample pick-up courier services, coordination with the third party sample collection contractor, determination of which samples are held, disposed, or requested for analysis, shipping samples to subcontractor laboratories, coordination the analytical services for a total of eight laboratories, including all three Spectrum facilities, collection of laboratory data reports, and reporting final hardcopy and electronic data.

Project Location: EPA Region I Superfund DAS Program
Client: Weston Solutions, Inc
Description of Work: Spectrum provides modified or non-routine analytical testing services to Weston Solutions, Inc. in support of their EPA Region I contract. These services are provided under the DAS (Delivery Analytical Services) program. This program supports non-routine testing requirements not addressed by standard CLP methods. Spectrum's participation has included special lists of metals and organic compounds, waste characterization analyses and rapid turnaround time report delivery. All testing requires full QC documentation, reports are provided in full CLP-type data deliverables packages and electronic data deliverables in SEDD-ADR formats as well as a client specific Excel file. This work has included a major PAH/PCB analysis program

involving over 1000 samples and all three of the Spectrum laboratory locations and other programs with a significant portion of the analyses on a 24-hour turnaround or 3 day turnaround basis.
Spectrum also provides non-routine testing support to other EPA engineering contractors in Region I (Nobis, Metcalf & Eddy), Region II (Weston), VIII (CH2M-Hill) and VIII (CDM), as well as to previous contractor in Region I, Tetra Tech NUS.

Project Location: Various Naval Sites in the East and Northeast United States
Client: Tetra Tech NUS
Description of Work: Spectrum has completed work for Tetra Tech NUS under a number of their Navy CLEAN (Comprehensive Long-term Environmental Action Navy) Analytical Support contracts. The analytical services provided by Spectrum have included both EPA SW846 and other analyses. Routine volatile organic, semivolatile organic, pesticide/PCB, metals, explosives and wet-chemistry analyses have been performed on water, soil, sediment and biological tissue samples. For all work conducted under these contracts, Spectrum provided full data packages in PDF and hardcopy forms that followed CLP formats. In addition, the EDD's were created in the specific format requested by TTNUS.

Project Location: RIRRC, Central Landfill, Johnston RI, OU-1 Superfund Site.
Client: Rhode Island Resource Recovery Corporation
Description of Work: Spectrum has provided soil, water, air and compost monitoring services to the Rhode Island Resource Recovery Corporation (RIRRC) since 1995. Spectrum provides services in support of numerous RIRRC programs including monthly industrial wastewater pretreatment monitoring, routine groundwater monitoring, Superfund site RI/FS support analyses, compost testing as well as non-routine or emergency analysis programs.

Spectrum Analytical, Florida Division:

Project Location: Air Force Plant 6
Client: CH2M HILL, Inc.
Description of Work: Spectrum provides analytical services for an on-going U.S. Federal Government prime contract for Air Force Plant VI in Marietta, Georgia. Analytical testing includes analysis using SW846 organic, inorganic and waste characterization methods; other methods are put on-line when requested by the project teams conducting field testing. Spectrum provides AFCEE data package deliverables as well as electronic data in an ASCII, comma-delimited file format.

Project Location: Cape Canaveral and Patrick AFB
Client: 3E
Description of Work: Spectrum supports Long term Monitoring, OTM, Remediation, Munitions response and Space Launch Demolition for an on-going U.S. Federal government prime contract for Cape Canaveral/Patrick AFB in Cape Canaveral, Florida. Analytical testing methods include analysis using SW846 organic, inorganic and waste characterization methods. Spectrum provides AFCEE ERPIMS deliverables and 2^{nd} 45^{th} Space Wing GIS deliverables for this contract.

Project Location: MacDill AFB
Client: Earth Tech AECOM
Description of Work Spectrum is providing analytical services for an on-going U.S. Federal Government prime contract for MacDill AFB and the U.S. Army Corps of Engineers. The laboratory requirements for these programs entails performing analyses, meeting all quality control criteria, and reporting analytical data for samples collected during all phases of the work effort. Samples include solid waste and aqueous matrices. Analyses requested include SW846, MEE, and RSK175 methods. Data packages are provided both electronically and in hard copy format. All information is also available on the online web-based dms.

APPENDIX A-2
Spectrum Analytical, Rhode Island
Quality Assurance Manual

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SPECTRUM ANALYTICAL, INC.
Featuring
HANIBAL TECHNOLOGY
Rhode Island Division

QUALITY ASSURANCE PLAN
2012

Approved By:

Digitally signed by Hanibal C. Tayeh
Date: 2012.10.09 14:40:39 -04'00'

Hanibal C. Tayeh, Ph. D.
President, and CEO — 10/09/2012 Date

Yihai Ding
Laboratory Director — 10/09/2012 Date

Sharyn B. Lawler
Quality Assurance Director — 10/09/2012 Date

EFFECTIVE DATE: 10/26/2012

646 Camp Ave. North Kingstown Rhode Island 02852 401-732-3400 · FAX 401-732-3499
www.spectrum-analytical.com

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Appendices

3.0 INTRODUCTION

Spectrum Analytical, Inc. Featuring Hanibal Technology Rhode Island Division (formerly MITKEM and referenced as Spectrum Analytical, Inc. RI Division throughout this document going forward) is an environmental testing laboratory dedicated to providing high quality analytical data and exceptional customer service.

Opened in 1994, as Mitkem Corporation, and purchased by Spectrum Analytical, Inc. in 2007, Spectrum Analytical, Inc. RI Division's laboratory facility is designed for high throughput and efficient laboratory operations. Separate secure areas are dedicated to sample receipt and storage. Spectrum Analytical, Inc. RI Division has individual sample preparation laboratories for organic and inorganic analyses and individual instrumentation rooms for extractable organics, volatiles, metals and wet-chemistry analyses.

Spectrum Analytical, Inc. RI Division recognizes the importance of controlling in-house cross contamination. The organic preparation area and the volatile organic instrument room are in separate areas, at opposite ends of the building to minimize solvent contamination of the volatile analysis. The air handling system in the volatiles laboratory is completely isolated from the remainder of the facility. A floor plan of the facility is included (Figure 3-1).

Spectrum Analytical, Inc. RI Division has placed a priority on obtaining and operating a large fleet of the latest, most sophisticated Hewlett-Packard, Thermo Scientific and Perkin-Elmer instruments. This emphasis on instrumentation enables the lab to operate at a high level of analytical efficiency.

Spectrum Analytical, Inc. RI Division specializes in performing laboratory analyses using the newest US EPA Contract Laboratory Program (CLP) *SOM* Organic and *ISM* Inorganic methods, as well as providing CLP-format data reports for virtually any test we perform. Spectrum Analytical, Inc. RI Division provides CLP-format reporting for EPA CLP, SW-846, MCAWW and Standard Methods analyses. Much of this work is performed by the laboratory under Department of Defense Quality Systems Manual (QSM) and ISO-17025 guidelines. Spectrum Analytical, Inc. RI Division has the flexibility to provide project-specific custom method modifications to meet the needs of a unique client or analytical requirement.

Spectrum Analytical, Inc. RI Division has participated in numerous environmental laboratory programs for both state and federal agencies including: the United States Navy, the United States Army Corps of Engineers, and the Air Force Center for Environmental Excellence. In addition Spectrum Analytical, Inc. RI Division is currently providing laboratory services under the United States Environmental Protection Agency Contract Laboratory Program. Spectrum Analytical, Inc. RI Division has been a contractor to the EPA under the CLP program continuously for over 15 years.

Spectrum Analytical, Inc. RI Division is a division of Spectrum Analytical, Inc. of Agawam, Massachusetts. Spectrum Analytical, Inc is an environmental laboratory company with laboratory locations in Agawam, MA, North Kingstown, Rhode Island and Tampa, Florida, providing analyses of soil, water and air samples for a wide variety of private and government clients.

This Quality Assurance Plan (QAP) describes the policies, organization, objectives, and quality control activities. It also specifies quality assurance functions employed at Spectrum Analytical, Inc. RI Division and demonstrates our dedication to the production of accurate, consistent data of known quality. This QAP is developed by following the guidelines discussed in the EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/R-5, Reissued May 2006: EPA Requirements for Quality Management Plans, EPA QA/R-2, Reissued May 2006: Department of Defense (DOD QSM) Quality Systems Manual for Environmental Laboratories Version 4.2: and the National Environmental Laboratory Accreditation Conference (NELAC) standards, June 5, 2003 (Effective July 1, 2003)/ The NELAC Institute (TNI) Standards.

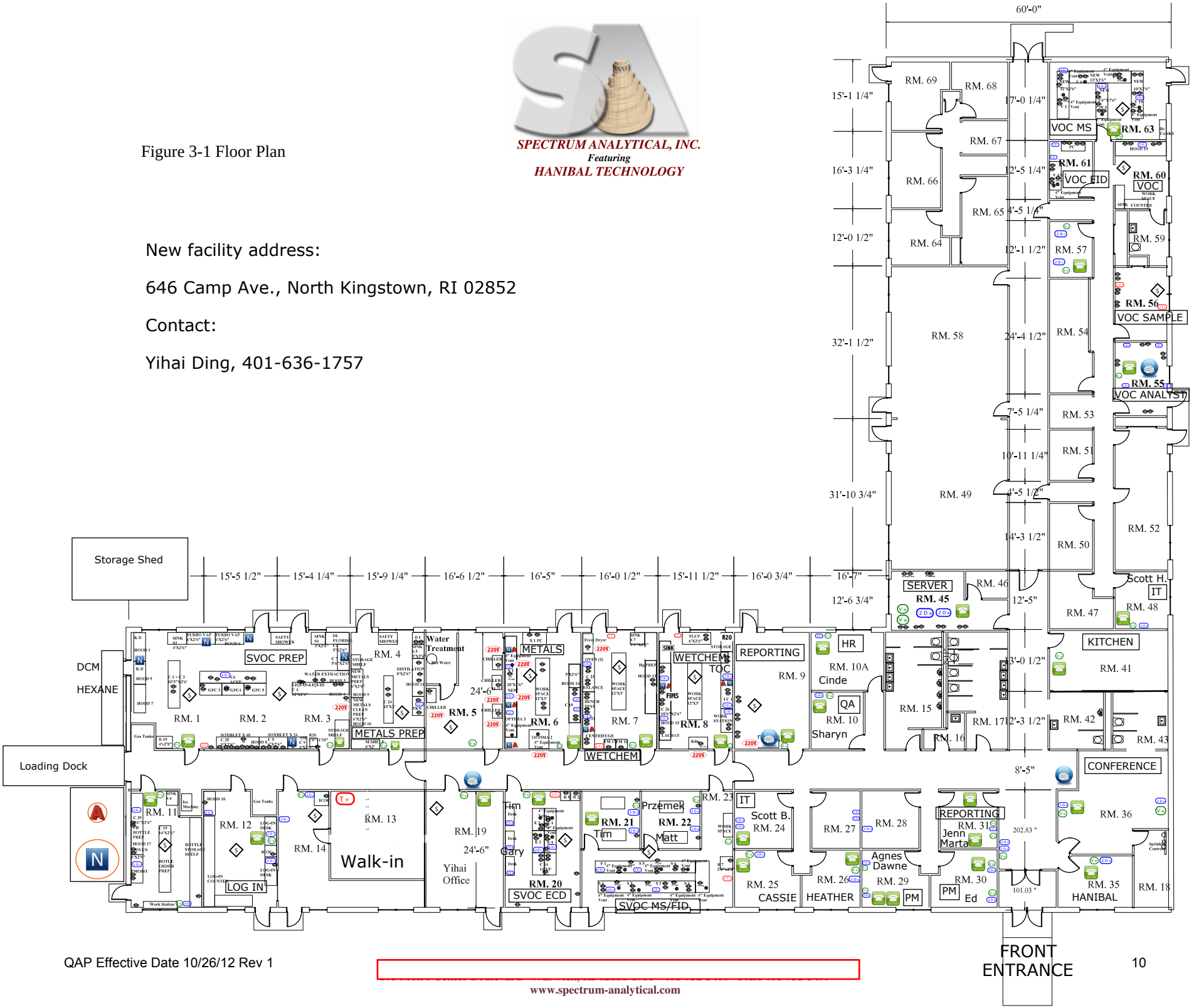
Figure 3-1 Floor Plan

New facility address:

646 Camp Ave., North Kingstown, RI 02852

Contact:

Yihai Ding, 401-636-1757



4.0 QUALITY ASSURANCE POLICY STATEMENT

Spectrum Analytical, Inc. RI Division is firmly committed to the production of valid data of known quality through the use of analytical measurements that are accurate, reproducible and complete. To ensure the production of such data, Spectrum Analytical, Inc. RI Division has developed a comprehensive Quality Assurance/Quality Control Program that operates throughout the entire organization.

Spectrum Analytical, Inc. RI Division Management considers Quality Assurance/Quality Control to be of the highest importance in the success of its Analytical Testing Laboratory and therefore fully supports the staff in the implementation and maintenance of a sound and thorough Quality Assurance Program.

Spectrum Analytical, Inc. RI Division's corporate success is based on its participation in the most rigorous and quality-focused environmental testing programs, such as the EPA Contract Laboratory Program, US Department of Defense programs, NELAC, and other nationwide and state-specific certification and approval programs. These programs require consistent application of the QA/QC procedures described in this document. Spectrum Analytical, Inc. RI Division's ability to demonstrate and document that analyses were performed in this manner is one of the foundations of its business. The other foundation of its business is to provide superior levels of customer service, above and beyond the norm for laboratories performing at this level of quality.

Spectrum Analytical, Inc. RI Division's approach to customer service is to aggressively meet or exceed customer expectations, particularly in terms of turnaround time for results. While the production of rapid turnaround time data may require lab employees to "go the extra mile" for the customer, without quality, the data are useless. Spectrum Analytical, Inc. RI Division constantly strives to manage its business to rapidly provide data to meet all the requirements of its quality program.

- Spectrum Analytical, Inc. RI Division management works to insure: that employees understand the primary importance of quality in its day to day operations,
- that employees will not be subjected to pressure to sacrifice quality for turnaround, financial or other considerations,
- that employees understand the importance of their ethical responsibilities in terms of data manipulation, falsification or other illegal or improper actions,
- that the company avoids involvement in activities that diminish its competence, impartiality, judgment or operational integrity.
- that employees maintain all client information in a confidential manner, and
- that employees understand that any short-term gain realized by disregarding the QA/QC program will be more than wasted by the serious penalties for these actions.
- That the laboratory has the technical personnel to identify occurrences of departure from the quality system and to initiate actions to prevent or minimize such departures.

All employees receive training in these issues as part of the initial orientation process, and are required to acknowledge that they understand their responsibilities in these areas. These issues are also discussed among all laboratory staff at company meetings and re-training sessions. The QA Officer, Technical Director and other senior management are readily available to all staff through their daily presence, "open door" policy and approachable manner. This allows any employee to readily discuss any questions, concerns or issues that may occur.

Quality Control is defined as an organized system of activities whose purpose is to demonstrate that quality data are being produced through documentation. Quality Assurance is more broadly defined as a system of activities designed to ensure that the quality control program is actually effective in producing data of the desired quality.

Quality Control is included as part of Quality Assurance. In supporting government regulatory and enforcement proceedings, a high degree of attention to quality is essential. Thorough application of quality control principles and routine quality assurance audits is required.

The basic components of the Spectrum Analytical, Inc. RI Division's QA/QC Program are control, evaluation and correction.

Control ensures the proper functioning of analytical systems through the implementation of an orderly and well-planned series of positive measures taken prior to and during the course of analysis including quality control practices, routine maintenance and calibration of instruments, and frequent validation of standards.

Evaluation involves the assessment of data generated during the control process. For example, precision and accuracy are determined from the results of duplicates and spikes, and other check samples. Long-term evaluation measures include performance and systems audit conducted by regulatory agencies, as well as the lab's quality assurance department.

Correction includes the investigation, diagnosis and resolution of any problems detected in an analytical system. Proper functioning of the system may be restored through method re-evaluation, analysis of additional check samples, trouble-shooting and repair of instrumentation or examination and comparison with historical data. Corrective actions are documented and reviewed to make sure they are implemented.

Certain situations may occur when there are occasional departures or exceptions from documented policies and procedures or standard specifications due to client or project specific protocols, unusual sample matrix, or special non-target analyte or non-routine analyses. Spectrum Analytical, Inc. RI Division's policy is to fully document all such procedures and their associated QC, and notify the client or regulatory agency. If the situation is to continue, a Standard Operating Procedure will be written and implemented.

5.0 QUALITY ASSURANCE MANAGEMENT, ORGANIZATION AND RESPONSIBILITY

Quality Assurance at Spectrum Analytical, Inc. RI Division is a company-wide function that depends on:

(1) cooperative working relationships at all levels within the laboratory and

(2) Multi-level review through all working levels of responsibility.

Responsibilities for QA/QC functions begin with the bench scientist and extend to the chief executive officer.

The primary level of quality assurance resides with the bench scientist. After completion of the documented training program, his/her responsibilities include:

- complying with all aspects of formally approved analytical methods and SOPs,
- carefully documenting each step of the analytical process,
- conscientiously obtaining peer review as required,
- promptly alerting laboratory supervisors and/or QA staff members to problems or anomalies that may adversely impact data quality, and
- participation in corrective actions as directed by the laboratory supervisor or QA Director.

The Manager of each laboratory department is responsible for ensuring thorough oversight of the quality of the data generated by the department supervisors, technicians and/or analysts. The Department Manager implements and monitors the specific QC protocols and QA programs with the laboratory to ensure a continuous flow of data meeting all control protocols and Spectrum Analytical, Inc. RI Division QA requirements. The Department Manager's responsibilities include providing the technicians and/or analysts with adequate resources to achieve the desired quality of performance.

The Spectrum Analytical, Inc. RI Division organizational structure is shown in the Organization Chart (Figure 5-1).

Spectrum Analytical, Inc. RI Division's lines of communication flow upward on the Organizational Chart. The open door policy allows all employees' access to anyone on the organization chart. If an employee has an issue with his/her immediate supervisor, he or she may, at any time, speak with someone in management higher up in the Organizational Chart.

Implementation of the entire Quality Assurance Program is the responsibility of the QA Director. While interacting on a daily basis with laboratory staff members, the QA Director remains independent of the laboratories and reports directly to the Laboratory

Director. The QA Director evaluates laboratory compliance with respect to the QA program through informal and formal systems and performance audits as described in Section 13. Remedial action, to alleviate any detected problems, is suggested and/or discussed with the appropriate parties and implemented when necessary.

With input from the appropriate staff members, the QA Director writes, edits and archives QA Plans, QC protocols, and Standard Operating Procedures (SOPs) in accordance with US EPA approved methodologies, and GLP procedures. If site-specific or project-specific QA Plans and/or QC protocols are required, these will be generated as needed.

An essential element of the QA program is record keeping and archiving all information pertaining to quality assurance including QA/QC data, pre-award check sample results, performance test sample results, scores, and follow-up; state certifications of the laboratory; external and internal audits with resolution of EPA and other audit team comments, recommendations and reports. The QA Director also plays an important role in the corrective action mechanism described in Section 16.

In addition, the QA Director works with laboratory staff and management to continuously upgrade procedures and systems to improve the laboratory's efficiency and data quality.

Ultimately, the success of the QA program depends on the cooperation and support of the entire organization. Spectrum Analytical, Inc. RI Division's most valuable resource is its staff of dedicated professionals who take personal pride in the quality of their performance.

Laboratory management works to ensure the competence of all who operate equipment, perform tests and calibrations, evaluate data and sign reports. When employees are in training, appropriate supervision will be provided until the employee has demonstrated the appropriate level of understanding, training, and skill.

Spectrum Analytical, Inc. RI Division's personnel job descriptions:

Responsibilities of each staff area in the laboratory include:

Technician / Preparation Laboratory Areas:

- Analysis of samples through compliance with all aspects of formally approved analytical methods and laboratory SOPs.
- Carefully documenting each step of the analytical process.
- Noting in the appropriate logbook area any unusual occurrences or sample matrix problems.
- Conscientiously obtaining peer review as required.

- Promptly alerting laboratory supervisor, Department Managers and/or QA staff members to problems or anomalies that may adversely impact data quality.
- Routine housekeeping duties for their laboratory area.

Analyst / Instrument Laboratory Areas:

- Analysis of samples through compliance with all aspects of formally approved analytical methods and laboratory SOPs.
- Routine maintenance of instrumentation.
- Preparation of analytical standards and spiking solutions which are documented and traceable to their original source.
- Carefully documenting each step of the analytical process.
- Noting in the appropriate logbook area any unusual occurrences or sample matrix problems.
- Conscientiously obtaining peer and Department Manager review as required.
- Promptly alerting the appropriate Department Manager and/or QA staff members to problems or anomalies that may adversely impact data quality.
- Documenting the initial review of analysis data to determine compliance with established company QA/QC protocols and any project-specific QA criteria, and noting any unusual occurrences or discrepancies on the data review checklist.
- Routine housekeeping duties for their laboratory area.

Data Reporting Specialists:

- Assemble CLP-format data reports by organizing data report forms and raw data in proper order to allow for technical data review.
- Enter data into LIMS or other data reporting computer programs, and print report forms as appropriate.
- Provide non-technical typographical review of data entered into computer systems by other individuals.
- Deliver data reports to customers by FAX or electronic mail.
- Paginate, photocopy, scan, save to CD (bookmark if required) and archive copies of customer reports or other documentation to be retained by the laboratory, or prepare paperless reports.
- Ship, or organize for courier delivery, final data reports to customers.
- Assist the QA Director in management of the document control system.
- Assist Project Managers with bottle order requests and shipment of coolers.
- Assist Project Managers in other tasks as required.

Laboratory Department Manager/Supervisors:

- Oversight of supervisors (where applicable), technicians and/or analysts in their laboratory areas.

- Monitors the status of all work in their laboratory area to insure compliance with holding time and turnaround time requirements.
- Training new scientists in the appropriate procedures and methods in the laboratory.
- Works with Laboratory Director and the QA staff to review, revise and implement SOPs.
- Insures adequate resources to perform the needed tasks by working with administrative personnel to order needed supplies.
- Insures all supplies and reagents meet the QC requirements of their intended task prior to their use in the laboratory.
- Insures all staff are using proper safety protocols.
- Works with Laboratory Director on the annual review of personnel performance.
- Interviews prospective new employees to insure they have the minimal level of qualifications, experience, education and skills necessary to perform their tasks, as well as the appropriate work ethic and social skills necessary for proper teamwork and productivity.
- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Documents any non-compliance or other unusual occurrences noted during sample analysis and data review such that these can be included in the report narrative and explained to the client.

Data Reviewer:

- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Generates paperless CLP and CLP-like data packages.
- Documents any non-compliance or other unusual occurrences noted during sample analysis and data review such that these can be included in the report narrative and explained to the client.
- Compiles narrative.
- Assist Laboratory Director, Supervisors and Department Managers in other tasks as required.

Laboratory Director:

- Works with Department Managers to coordinate laboratory areas in the completion of analytical projects.
- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Works with QA Director to implement new SOPs and to annually review and revise existing SOPs.
- Works with the QA Director, Department Managers and Supervisors to develop and implement corrective action when needed.

- Works with management and supervisory staff to continuously improve the quality and efficiency of all company procedures.
- Assists Department Managers in the annual review of personnel performance.
- Supervises all Management, QA and Supervisory staff to insure compliance with company QA policies and other company procedures.
- Provides technical assistance to all areas of the laboratory staff.
- Acts as technical consultant for chemistry related issues that arise in the lab.
- Provides assistance with instrument optimization or performance issues as needed.
- Offers input on the purchase and operation of new instrumentation.
- Trains other analysts in procedures and methodologies.

Director of Business Development

- Pursues new contracts/projects as well as clients.
- Works with Spectrum Marketing to prepare Bids.
- Ensures laboratory is aware of specific requirements of new projects/contracts.
- Works with clients to insure all questions and concerns are addressed and answered.
- Works with clients to insure their understanding of complex technical issues.
- Works with Quality Services Department staff to continuously improve the quality and efficiency of all company procedures.

Data Reporting Supervisor:

- Works with Laboratory Director, Department Managers and Supervisors to prioritize and coordinate laboratory areas in the timely completion of analytical projects.
- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Writes project report narratives to document any unusual occurrences noted during sample analysis.
- Works with management and supervisory staff to continuously improve the quality and efficiency of all company procedures.
- Works with Laboratory Director on the annual review of personnel performance.

Project Manager:

- Works with the client to completely understand the requirements of all incoming work.
- Evaluates the client’s requirements as compared to the abilities of the laboratory as stated in Standard Operating Procedure (SOP) #110.0023 Project Management.
- Works with the Data Reporting staff to continuously improve the quality and efficiency of all company procedures.

- Communicates the customer's requirements to all laboratory staff working on the project.
- Works with the customer to determine the number and type of sample containers required for the project.
- Works with the Sample Custodian to resolve and communicate to the client any problem or discrepancies with incoming samples.
- Maintains open, responsive and continuous communication with the customer.
- Follows up with the client to assess level of satisfaction, and insure all project goals have been accomplished.
- Assist Business Development and Marketing Staff in other tasks as required.

Information Technology Director:

- Oversees the operations of the three divisions of Spectrum Analytical, Inc. (MA, FL and RI). The IT Director's role is technical guidance, IT long term planning, coordination/communication between the divisions, oversees and makes the necessary decision to support the overall IT function.

Information Technology Manager:

Primary function is to oversee the operations of the Spectrum Analytical, Inc. RI Division's IT department.

- Oversee the operations of the network, including servers and workstations.
- Plan for hardware and software updates
  1) Support users IT needs.
  2) Support client IT needs.
  3) Oversee security of network
- Development and expansion of LIMS.
  1) Program new functionality into LIMS including program based protocols requirements
  2) hard copy reports
  3) electronic reports
  4) processing of data to web site
  5) tracking of data
  6) maintenance of LIMS
  7) security of LIMS
- Generate and troubleshoot more complex EDDs
- Provide backup for the Information Technology Specialist when out and support when it is needed.

Secondary function is to work with the other divisions to try and make transfer of information as seamless as possible. Lend technical support to other divisions and help to bring technical help from other divisions to Spectrum Analytical, Inc. RI Division IT department.

Information Technology Specialist:

- Primary duty is to generate and review EDDs using EDD module.
  a) Generate and validate EDDs using EDD specific tools (CRA, Tetra Tech, CH2M Hill, etc…).
  b) Generate all SEDD files for the EPA SOM contract, and work with the chemists to resolve any defects, if possible.
- Perform server room duties.
  a) Monitor the servers and troubleshoot (if needed)
  b) Perform backup/archive of data on servers
  c) File grooming at the end of the month
  d) Monitor event logs of the servers for issues.
  e) Monitor status of centralized anti-viral program (AVG). Includes monitoring AVG status of workstations
  f) Monitor centralized Windows System Update Server (WSUS). Includes monitoring WSUS status of workstations.
- Handles user issues with printer/scanner/copier systems from Ikon. Based on evaluation, schedule service calls or replaces consumable parts.

Quality Assurance Director:

- Implements the entire QA program.
- Interacts on a daily basis with laboratory staff.
- Evaluates compliance with the QA program through formal and informal reviews of data and processes.
- Implements the corrective action system.
- Maintains a master list of all SOPs and monitors review schedules.
- Works with Department Managers and Supervisors to implement new SOPs and to annually review and revise existing SOPs.
- Controls all master and controlled-copies of SOPs and QAP as per SOP #80.0012; Production of Standard Operating Procedures.
- Posts to intranet, and archives all old and edited revisions of SOPs and QA manual as per SOP# 80.0012; Production of Standard Operating Procedures.
- Interfaces with certification authorities and agencies to maintain existing certifications and programs, and obtain new certifications.
- Maintains records of employee training and certification as per SOP# 80.0016; Training Procedures and Tracking.
- Instructs laboratory personnel on ethics in the workplace.
- Oversees analytical trends that need to be evaluated and corrected.
- Oversees the implementation of MDLs and control limit studies.
- Directs the internal audit program as per SOP# 80.0006; Internal Audits.
- Coordinates all external audit corrective action reports and investigations.
- Maintains certification of NIST thermometers and weights.

- Schedules annual hood inspections and balance calibrations.

In Spectrum Analytical, Inc. RI Division's organizational structure, the Laboratory Director has the ultimate authority for all chemistry-related aspects of the company.

The QA Director reports directly to the Laboratory Director. She has the authority within the management system to bring any issue to the highest levels of the company management and ownership, as well as to halt the release of data she believes to be questionable or suspend the performance of an analysis she believes to be unreliable.

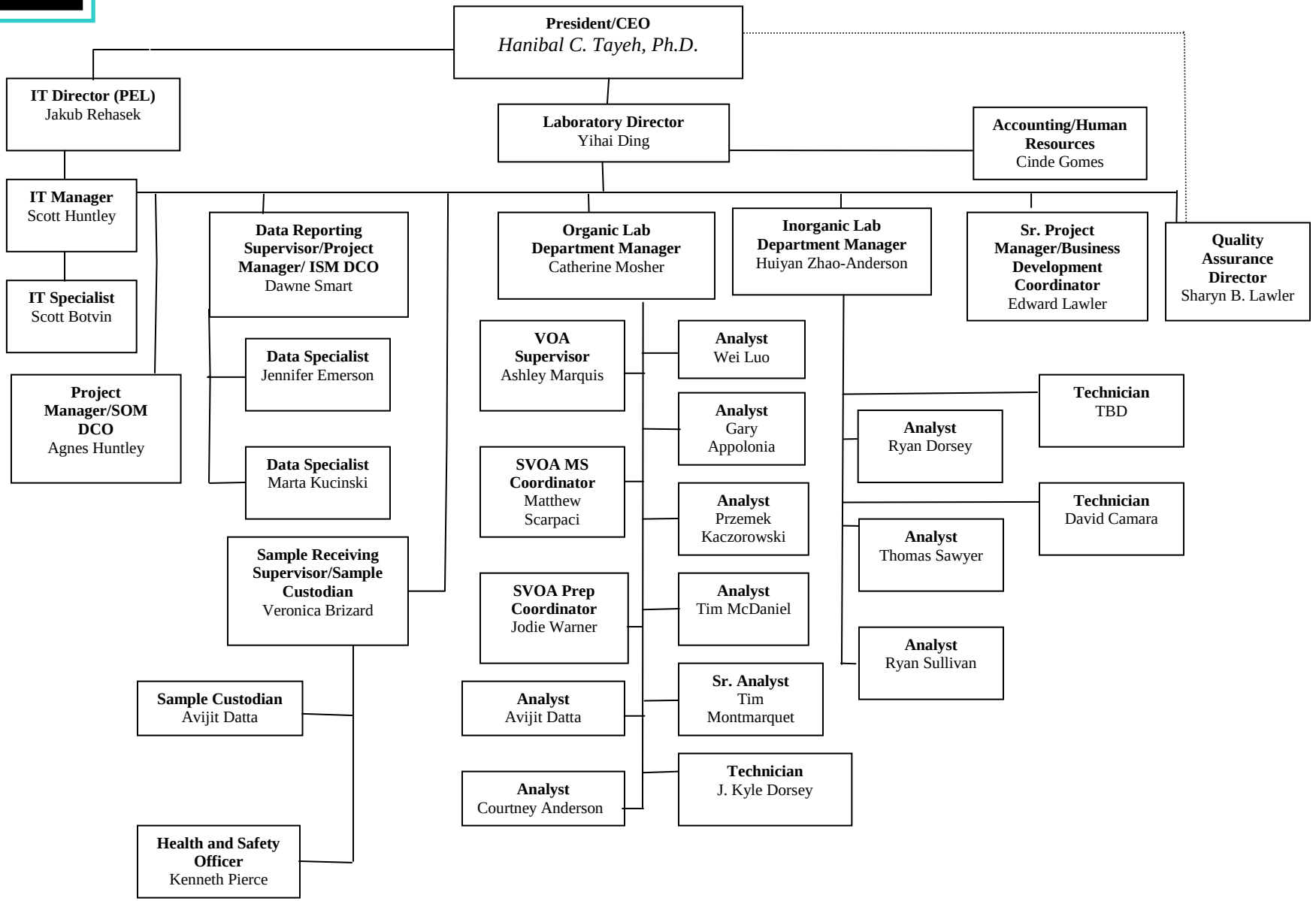
The Director of Business Development works with the project managers and marketing staff and with the Department Managers and Supervisors to prioritize and coordinate work within the laboratories.

The personnel training records are located in the QA department on-site as well as additional training documents being saved in pdf form on the Spectrum network. All individual training is documented including new employee training, individual training, annual retraining procedures, and Health and Safety training.

Figure 5-1
SPECTRUM ANALYTICAL, INC. RI Division's Organizational Chart

Spectrum
Analytical RI

Organizational Chart



6.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, REPRESENTATION, COMPLETENESS AND COMPARABILITY AND QA REPORTING

As part of the evaluation component of the overall QA Program, laboratory results are compared with the data quality indicators defined as follows:

- Precision: the agreement of reproducibility among individual measurements of the same property usually made under identical conditions.

- Accuracy: the degree of agreement of a measurement with the true or accepted value.

- Representation: the degree to which data accurately and precisely represent a characteristic of a population, parameter variations of a sample of a finite process condition, or of a finite environmental condition.

- Completeness: a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions.

- Comparability: an expression of the confidence with which one laboratory data set can be compared with another laboratory data set in regard to the same property and laboratory sample population.

Quality Assurance objectives may vary by project and requested parameters. The accuracy, precision, and representation of data will be functions of the origins of the sample material, the procedures used to analyze sample and generate data, and the specific sample matrices involved in each project. Quality control practices utilized in the evaluation of these data quality indicators include blanks, replicates, spikes, standards, check samples, calibrations and surrogates. The process for quantifying or assessing the above indicators for data quality is addressed in Section 15.

6.1 Precision and Accuracy:

For each parameter analyzed, the QA objectives for precision and accuracy will be determined from:

- Published historical data;
- Method validation studies;
- Spectrum Analytical, Inc. RI Division’s experience with similar samples and/or;
- Project-specific requirements, such as those stipulated by the USEPA in the CLP protocols and control documents.

6.2 Representation:

Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. In most cases, representation is achieved by mixing the laboratory sample well before removing a portion for analysis. On occasion, multi-phase laboratory samples may require that each phase be analyzed individually and reported in relation to its proportion in the whole sample.

6.3 Completeness:

The completeness goal is 100% in all cases and includes:

- Analysis of all samples;
- Generation and analysis of all required QC samples;
- Sufficient documentation of associated calibration, tuning and standardization;
- Records of data reduction processes, including manual calculations.

While the laboratory staff is responsible for achieving the completeness objective stated above, assigning each project a specific project manager whose functions include sample management and tracking ensures completeness.

6.4 Comparability:

To assure comparability, Spectrum Analytical, Inc. RI Division employs established and approved analytical methods (e.g. USEPA protocols), consistent analytical bases (dry weight, volume, etc.) and consistent reporting units (mg/Kg, µg/L, etc.). Where data from different samples must be comparable, the same sample preparation and analysis protocols are used for all of the samples of interest.

6.5 QA Reporting

General QA procedures require that an MS/MSD or DUPLICATE/MS be reported with each sample batch up to 20 samples. In addition, each batch requires a method blank (MB) and laboratory control sample (LCS).

An acceptance criterion for the MB depends upon the method criteria. In-house control limits dictate the acceptability of the LCS in many methods. Several methods have set LCS control limits. A high bias LCS is considered acceptable if the analyte is not present in the samples above the reporting limit. A low bias LCS will require re-extraction (if sample volume allows) and re-analysis.

DUP, MS, and MSD recoveries and calculated RPDs are specified in the analytical methods. Recoveries outside the limits require some form of corrective action, whether that includes a post-digestion/distillation spike, re-extraction, re-analysis and/or notification to the client in the project narrative.

LIMS will flag any QA samples outside method criteria on the reporting forms. Formal written corrective action reports are required for any incident that does not meet method criteria and cannot be remedied or explained by the laboratory. The QA Officer signs off on any corrective actions and can also track QA trends in this manner.

7.0 SAMPLING PROCEDURES

For most projects, outside sampling teams deliver or send samples to Spectrum Analytical, Inc. RI Division's. When sampling by Spectrum Analytical, Inc. RI Division's personnel is required, the sampling team follows the sampling procedures outlined in the EPA *Test Methods for Evaluating Solid Wastes*, SW-846, 3rd Edition, or procedures found in the EPA "Handbook for Sampling and Sample Preservation of Water and Wastewater".

Appropriately prepared sample containers are supplied by Spectrum Analytical Inc., RI Division at clients' request. When required, preservatives are added to the sample containers. Tables 7-1 through 7-3 provide the Spectrum Analytical, Inc. RI Division Recommended Container, Preservation Techniques and Holding Times. Additional sample volumes may be required if additional QC functions are to be performed.

Holding times for SW846, CLP Methods, Standard Methods and certain USEPA methods are different and are presented in Tables 7-1 to 7-3. Holding times for most methods are calculated from the date of sample collection. Holding times for CLP methods are calculated from the Validated Time of Sample Receipt (VTSR). It should be noted that the CLP analysis program combines chemical analyses and contract compliance procedures in one document. For laboratory analysis and contract compliance purposes, holding times are calculated from VTSR, while post-analysis data usability and validation (generally performed by the client or a third party) compares holding times to the SW-846 method holding times calculated from date of sample collection.

Representative portions of samples are taken for analysis by following Spectrum Analytical, Inc. RI Division's SOP 110.0039 Standard Operating Procedure for Sub-Sampling.

Table 7-1

Recommended Container, Preservation Techniques and Holding Times For SW-846 Analyses

Analytes	Method	Containers	Required* Volume	Preservation	Holding Times
Volatile Organics					
Solid	8260, 5030	Amber glass jar with Teflon lining	Minimal head-space in jar	4°C	14 days
Solid [a]	8260, 5035	40mL vial or Encore with Teflon lining	5.0gram ± 0.5	4°C, unpreserved	48 hours
				DI Water -10 to –20^{0}C	14 days
				Sodium bisulfate -10 to –20^{0}C, 4^{0}C	14 days
				Methanol 4^{0}C	14 days
Aqueous	8260, 5030	40mL VOA Vials with Teflon septum	40mL	4°C HCl, pH<2	14 days
Semivolatile Organics					
Solid	3540, 3550 8270	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days
Aqueous	3510, 3520 8270	Amber glass bottles with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days
Polychlorinated Biphenyls					
Solid	3540, 3550 8082	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days
Aqueous	3510, 3520 8082	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days
Organochlorine Pesticides					
Solid	3540, 3550 8081	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days
Aqueous	3510, 3520 8081	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days
Chlorinated Herbicides					
Solid	8151	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days
Aqueous	8151	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days

Table 7-1 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times
For
SW846 Analyses

Analytes	Method	Containers	Required* Volume	Preservation	Holding Times
Total Petroleum Hydrocarbons					
Gasoline Range Organics, including Maine-GRO**					
Solid	8015, 5030 ME 4.1.17	Amber glass jar With Teflon lining	Minimal head-space in jar	4°C	14 days
Solid [a]	8015, 5035	40mL vial or Encore with Teflon lining	5.0gram ± 0.5	4°C, unpreserved	48 hours
				4°C, Methanol	14days
Aqueous	8015, 5030 ME 4.1.17	40mL VOA vials With Teflon septum	40mL	4°C HCl, pH<2	14 days
Diesel Range Organics, including Maine-DRO					
Solid	3540, 3550 8015 ME 4.1.25	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days
Aqueous	3510, 3520 8015 ME 4.1.25	Amber glass bottle with Teflon lining	1L	4°C H_2SO_4, pH<2	Extraction within 7 days Analysis within 40 days
Total Metals except Mercury and Chromium (VI)					
Solid	3050 6010	Amber glass jar with Teflon lining	10g	4°C	180 days
Aqueous	3005, 3010	Polyethylene bottle	100mL	HNO_3, pH<2	180 days
Chromium (VI)					
Solid	3060, 7196	Amber glass jar with Teflon lining	10g	4°C	Digestion within 30 days Analysis within 96 hours
Aqueous	7196	Polyethylene bottle	25mL	4°C	24 hours
Mercury					
Solid	7471	Amber glass jar	10g	4°C	28 days
Aqueous	7470	Polyethylene bottle	100mL	4°C HNO_3, pH<2	28 days
Cyanide					
Solid	9012	Amber glass jar with Teflon lining	10g	4°C	14 days
Aqueous	9012	Polyethylene bottle	50mL	4°C NaOH, pH≥12	14 days
Flashpoint					
Aqueous	1010	Amber glass bottle	30mL	4°C	28 days

Table 7-1 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times
For
SW846 Analyses

Analytes	Method	Containers	Required* Volume	Preservation	Holding Times
Chloride					
Aqueous	9056	Polyethylene bottle	50mL	4°C	28 days
Nitrate					
Aqueous	9056	Polyethylene bottle	50mL	4°C	48 hours
Nitrite					
Aqueous	9056	Polyethylene bottle	50mL	4°C	48 hours
Orthophosphate					
Aqueous	9056	Polyethylene bottle	50mL	4°C	48 hours
Sulfates					
Aqueous	9056	Polyethylene bottle	50mL	4°C	28 days

Table 7-2

Recommended Container, Preservation Techniques and Holding Times For CLP/ASP Analyses

Analytes	Method	Containers	Required* Volume	Preservation	Holding Times
Volatile Organics					
Solid	CLP/ASP	Amber glass jar with Teflon lining	Minimal head-space in jar	4°C	10 days from VTSR
Aqueous	CLP/ASP	40mL VOA vials with Teflon septum	40mL	4°C HCl, pH<2	10 days from VTSR
	CLP Low	40mL VOA vials with Teflon septum	40mL	4°C HCl, pH<2	10 days from VTSR
Semivolatile Organics					
Solid	CLP/ASP	Amber glass jar with Teflon lining	30gram	4°C	10 days from VTSR Analysis within 40 days
Aqueous	CLP/ASP	Amber glass bottle with Teflon lining	1L	4°C	5 days from VTSR Analysis within 40 days
	CLP Low	Amber glass bottle with Teflon lining	1L	4°C	5 days from VTSR Analysis within 40 days
Organochlorine Pesticide/PCB					
Solid	CLP/ASP	Amber glass jar with Teflon lining	30gram	4°C	10 days from VTSR Analysis with 40 days
Aqueous	CLP/ASP	Amber glass bottle with Teflon lining	1L	4°C	5 days from VTSR Analysis within 40 days
	CLP Low	Amber glass bottle with Teflon lining	1L	4°C	5 days from VTSR Analysis within 40 days
Cyanide					
Solid	CLP/ASP	Amber glass jar	10gram	4°C	12 days from VTSR
Aqueous	CLP/ASP	Polyethylene bottle	50mL	4°C NaOH, pH>12	12 days from VTSR
Total Metals except Mercury					
Solid	CLP/ASP	Amber glass jar	10gram	4°C	180 days from VTSR
Aqueous	CLP/ASP	Polyethylene bottle	100mL HNO_3, pH<2	4°C	180 days from VTSR

Table 7-2 (cont'd)

Recommended Container, Preservation Techniques and Holding Times
For
CLP/ASP Analyses

Analytes	Method	Containers	Required* Volume	Preservation	Holding Times
Mercury					
Solid	CLP/ASP	Amber glass jar	10gram	4°C	26 days from VTSR
Aqueous	CLP/ASP	Polyethylene bottle	100mL	4°C HNO_3, pH<2	26 days from VTSR

Table 7-3

Recommended Containers, Preservation Techniques and Holding Times for Other Analyses

Analytes	Method	Containers	Required* Volume	Preservation	Holding Times
Volatile Organics					
Aqueous	624	40mL VOA vials with Teflon septum	40mL	4°C HCl, pH<2	14 days
Semivolatile Organics					
Aqueous	3510, 3520 625	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days
Organochlorine Pesticide/PCB					
Aqueous	3510, 3520 608	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days
EDB/DBCP					
Aqueous	8011	40mL VOA vials with Teflon septum	35mL	4°C HCl, pH<2	28 days
MA Extractable Petroleum Hydrocarbons (EPH)					
Solid	3540, 3550 MADEP	Amber glass jar with Teflon lining	10gram	4°C	Extraction within 7 days Analysis within 40 days
Aqueous	3510, 3520 MADEP	Amber glass bottle with Teflon lining	1L	4°C HCl, pH<2	Extraction within 14 days Analysis within 40 days
MA Volatile Petroleum Hydrocarbons (VPH)					
Solid	MADEP	Amber glass jar with Teflon lining	10gram	4°C 10mL Methanol	14 days
Aqueous	MADEP	40mL VOA vial with Teflon lining	40mL	4°C HCl, pH<2	14 days
Total Metals excluding Mercury					
Aqueous	200.7, 200.8	Polyethylene bottle	100mL	HNO_3, pH<2	180 days
Mercury					
Aqueous	245.1	Polyethylene bottle	100mL	HNO_3, pH<2	28 days
Cyanide					
Aqueous	335.4	Polyethylene bottle	50mL	NaOH, pH>12	14 days

Table 7-3 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times
for
Other Analyses

Analytes	Method	Containers	Required Volume*	Preservation	Holding Times
Chloride	E300.0	Polyethylene bottle	50mL	4°C	28 days
COD					
Aqueous	SM5220D	Amber VOA vial	40mL	4°C H_2SO_4, pH<2	28 days
Color					
Aqueous	SM2120B	Polyethylene bottle	50mL	4°C	Immediate
Nitrate					
Aqueous	E300.0	Polyethylene bottle	50mL	4°C	48 hours
Nitrite					
Aqueous	E300.0	Polyethylene bottle	50mL	4°C	48 hours
Orthophosphate					
Aqueous	SM4500-P, E E300.0	Polyethylene bottle	50mL	4°C	48 hours
Total phosphate					
Aqueous	SM4500-P B5,E	Polyethylene bottle	50mL 50mL	4°C H_2SO_4, pH<2	28 days
Phenols					
Aqueous	SM5530B E420.1	glass	250mL	4°C H_2SO_4, pH<2	28 days
Sulfates					
Aqueous	SM426 15th Ed. SM4500-SO4 E, E300.0	Polyethylene bottle	50mL	4°C	28 days
Sulfide Total					
Aqueous	SM4500-S-D	Polyethylene bottle	50mL	4°C NaOH, pH>12 ZnAc	28 days
Reactivity					
Solid	Chapter 7 SW846	Amber glass jar	10gram	4°C	28 days
Aqueous	Chapter 7	Polyethylene bottle	250mL	4°C	28 days
Total Organic Carbon (TOC)					
Solid	Lloyd Kahn Walkley-Black	Amber glass jar	10g	4°C	14 days

Table 7-3 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times
For
Other Analyses

Analytes	Method	Containers	Required* Volume	Preservation	Holding Times
Total Organic Carbon					
Aqueous	SM5310B	40mL VOA vials	40mL	4°C H_3PO_4, pH<2	28 days
TKN					
Aqueous	SM4500Norg C	Polyethylene bottle or Amber glass bottle	50mL	4°C H_2SO_4, pH<2	28 days
Total Solids (TS)					
Aqueous	SM2540B	Polyethylene bottle	200mL	4°C	7 days
Total Dissolved Solids (TDS)					
Aqueous	SM2540C	Polyethylene bottle	200mL	4°C	7 days
Total Suspended Solids (TSS)					
Aqueous	SM2540D	Polyethylene bottle	200mL	4°C	7 days
Settleable Solids					
Aqueous	SM2540F	Polyethylene bottle	200mL	4°C	48 hours
Chromium (VI)					
Aqueous	SM3500 Cr+	Polyethylene bottle	25mL	4°C	24 hours
Alkalinity					
Aqueous	SM2320B	Polyethylene bottle	100mL	4°C	14 days
Ammonia					
Aqueous	SM4500NH3B	Polyethylene bottle	100mL	4°C H_2SO_4, pH<2	28 days
Oil & Grease					
Aqueous	1664	Amber glass bottle with Teflon lining	1L	4°C HCl, pH<2	28 days

* These represent minimum required volume. Additional sample volumes should be collected to minimize headspace loss for volatile analysis. Additional sample aliquots are also required to perform QA/QC functions (e.g. spikes, duplicates), % moisture for solid samples and sample re-analysis (if needed).

[a] For Massachusetts analyses, the Volatile Organics soil samples are preserved in Methanol in the field.

EPA SW-846 Method 5035 provides several options for preservation of soil samples for volatile organics. Certain projects have not adopted these options to-date, and continue to recommend the collection of unpreserved soil sample aliquots for volatiles analysis. Spectrum Analytical Inc., RI Division's preference for low-level analysis is to collect approximately 5 grams of soil into 5mL of organic-free DI water and to preserve by freezing within 48hours of collection. A separate container with approximately 5 grams of

soil into 5mL of methanol is also collected for potential medium-level analysis. A separate container of unpreserved soil also must be collected to perform percent moisture analysis.

** Maine GRO soil analysis requires a medium level methanol extraction. A 10 gram sample and 10mL methanol volume is used.

8.0 SAMPLE CUSTODY

8.1 Chain of Custody:

Samples are physical evidence collected from a facility or the environment. In hazardous waste investigations, sample data may be used as evidence in (EPA) enforcement proceedings. In support of potential litigation, laboratory chain-of-custody procedures have been established to ensure sample traceability from time of receipt through the disposal of the sample.

A sample is considered to be in the custody under the following conditions:

- It is in an authorized person's actual possession, or
- It is in an authorized person's view, after being in that person's physical possession, or
- It was in an authorized person's possession and then was locked or sealed to prevent tampering, or
- It is in a secure area.

Chain-of-custody originates as samples are collected. Chain-of-custody documentation accompanies the samples as they are moved from the field to the laboratory with shipping information and appropriate signatures indicating custody changes along the way.

Laboratory chain-of-custody is initiated as samples are received and signed for by the Sample Custodian or his/her designated representative at Spectrum Analytical, Inc. RI Division. Documentation of sample location continues as samples are signed in and out of the designated storage facility for analysis in the several laboratory departments, using the Internal Chain of Custody (IntCOC) barcode system. After analysis, any remaining sample is held in the designated storage area to await disposal. Spectrum Analytical Inc., RI Division's policy is to hold spent samples for a period of at least thirty days from submittal of final report, unless other arrangements are agreed upon with the client. USEPA samples and empty containers are held for 60 days.

8.2 Laboratory Security:

Samples and all data generated from the analyses of samples at Spectrum Analytical, Inc. RI Division are kept within secure areas during all stages of residence, including the periods of time spent in preparation for analysis, while undergoing analysis, and while in storage.

The entire laboratory is designated as a secure area. The doors to the laboratory are under continuous surveillance, are kept locked after regular business hours

and may only be accessed by key or keypad entry. Only authorized personnel are allowed to enter the secure areas. The laboratory facility and IT office are only accessed through keypad entry. A Spectrum Analytical, Inc. RI Division staff member must accompany visitors to the laboratory.

8.3 Duties and Responsibilities of Sample Custodian:

Duties and responsibilities of the Sample Custodian include:

8.3.1 Receiving samples.

8.3.2 Inspecting and documenting sample shipping containers for presence/absence and condition of:

8.3.2.1 Custody seals, locks, “evidence tape”, etc.;

8.3.2.2 Container breakage and/or container integrity, including air space in aqueous samples, or proper preservation for soil samples for Volatiles analysis.

8.3.3 Recording condition of both shipping containers and sample containers (cooler temperature, bottles, jars, cans, etc.).

8.3.4 Signing documents shipped with samples (i.e. air bills, chain-of-custody record(s), Sample Management Office (SMO) Traffic Reports, etc.)

8.3.5 Verifying and recording agreement or non-agreement of information on sample documents (i.e. sample tags, chain-of-custody records, traffic reports, air bills, etc.). If there is non-agreement, recording the problems, contacting the project manager for direction, and notifying appropriate laboratory personnel. (Client’s corrective action directions shall be documented in the case file.)

8.3.6 Initiating the paper work for sample analyses on laboratory documents (including establishing sample workorder files) as required for analysis or according to laboratory standard operating procedures.

8.3.7 Label samples with laboratory sample identification numbers and cross-referencing laboratory numbers to client numbers and sample tag numbers.

8.3.8 Scanning samples into the ICOC system.

8.3.9 Placing samples and spent samples into appropriate storage and/or secure areas.

8.3.10 Where applicable, making sure that sample tags are removed from the sample containers and included in the workorder file.

8.3.11 Where applicable, accounting for missing tags in a memo to the file or documenting that the sample tags are actually labels attached to sample containers or were disposed of, due to suspected contamination.

8.3.12 Monitoring storage conditions for proper sample preservation and prevention of cross-contamination.

8.3.13 Sending shipping containers with prepared sample bottles and sample instructions to clients who request them.

8.3.14 Calibrating the non-contact infrared temperature gun quarterly.

8.3.15 Disposal of samples after a specified time period determined by contract or client request.

8.4 Sample Receipt:

The Sample Custodian or his/her designated representative receives sample shipments at Spectrum Analytical, Inc. RI Division. Unless the shipment is a continuation of a previous workorder, a new workorder file is started for the sample.

The cooler is inspected for the following (if applicable) and findings are documented on the Sample Login Form (Figure 8.4-1) for USEPA CLP samples, and on the Sample Condition Form (Figure 8.4-2) for all other samples:

- Custody seal (conditions and custody number)
- Air bill (courier and air bill #)

The cooler is then opened and the following items are checked (in order). Make sure the hood is turned on when the cooler is opened.

- Chain of custody (COC) records (or traffic report). These are usually taped to the inside of the cooler cover.
- Radioactivity using the Geiger counter, which continuously monitors the receiving area for radiation
- Cooler temperature using the non-contact infrared temperature gun. Record the temperature of a temperature blank if available, using a calibrated thermometer. Record each temperature on the COC.

The Sample Custodian will perform the following:

- Remove the sample containers and arrange them in the same order as documented in the chain of custody report.
- Inspect condition of the sample containers.
- Assign laboratory sample ID and cross-reference the laboratory ID to the client ID.
- Remove tags and place in the workorder file.
- Check preservative and document in the Sample Condition Form (Figure 8.4-2) if needed. If additional preservative is needed, it is added at this time.
- Check for air bubbles in aqueous samples and for proper preservation and immersion of soil samples designated for volatile organic analysis.
- Ensure peer review occurs for proper cross-referencing and labeling of sample containers.

Any discrepancies or problems are noted in the Sample Condition Notification Form (Figure 8.4-3).

The sample custodian conveys the information to the project manager who will in turn inform the client, or may directly inform the client of the discrepancies.

Samples can be rejected at Spectrum Analytical, Inc. RI Division for any of the following reasons:

1. Complete and proper documentation was not sent with the samples.
2. Sample labels cannot be identified because indelible ink was not used during the sampling procedure.
3. Hold times had already been exceeded when samples arrived at the laboratory.
4. Inadequate sample volume.
5. Potential cross-contamination has occurred among samples.
6. Samples are inadequately preserved.
7. The samples or shipping container is badly destroyed during shipping.
8. The samples are potentially radioactive.
9. The samples represent untreated fecal waste for which Spectrum Analytical Inc., RI Division employees are currently not inoculated against.

In all instances, the client is contacted initially before any action is taken at Spectrum Analytical, Inc. RI Division.

The Sample Custodian signs the Sample Receipt Form and originates a file folder for the set of samples. The following forms are included in the file: the Sample Receipt Form, chain of custody records, shipping information, and an orange

Sample Condition Notification Form if any problems or discrepancies need to be addressed.

When the Sample Custodian is not available to receive samples, another lab staff member will sign for the sample container. The time, date and name of the person receiving the container are recorded on the custody records. In addition, the cooler temperature is measured and recorded on the Sample Condition Form. The samples are then stored in the centralized walk-in refrigerator in the sample receipt area. The sample receipt area is located in the secure central storage facility of the laboratory. VOA samples are stored in the VOA analysis laboratory. The samples are officially received and documented by the Sample Custodian or designee before the next business day.

At times, samples will be sent to another lab for analysis not performed at Spectrum Analytical, Inc. RI Division. These subcontracted analyses are performed by laboratories certified to perform the analyses. The use of a subcontractor laboratory is discussed with the client prior to sending samples, per Spectrum Analytical, Inc. RI Division's Project Management Standard Operating Procedure.

These samples are packed to prevent breakage and stored in a cooler in the walk-in or stored in the small refrigerator in the central storage facility. The samples are either hand delivered to a local sub-contract lab, or shipped with sufficient coolant to maintain a 4 degree temperature by air courier under Spectrum Analytical, Inc. RI Division's chain-of-custody (Figure 8.4-4).

8.5 Sample Log-in Identification:

8.5.1 Sample Identification:

To maintain sample identity, each sample received at Spectrum Analytical, Inc. RI Division is assigned unique sample identification (Sample ID) numbers. Samples are logged into the laboratory via the Laboratory Information Management System (LIMS).

After inspecting the samples, the Sample Custodian logs each sample into the LIMS, which assigns a lab Sample ID Number. These Numbers are assigned sequentially in chronological order. Spectrum Analytical Inc., RI Division Sample Identification Numbers appear in the following format: **YXXXX-NNF**

In which: Y – represents the current year with A for 2002, B for 2003, C for 2004, etc.
XXXX – represents a four-digit work order number that is assigned sequentially to each submittal of samples
NN – represents the sample number within the group or workorder.

F – represents the fraction. All sample portions that are received in identical bottles with identical preservatives are grouped into one fraction.

For example, the first fraction of the fifth sample of the 20th workorder of 2003 would have the number: B0020-05A

The Sample ID Number is recorded on the Sample Login Form (Figure 8.4-1) for USEPA CLP samples, and on the Sample Condition Form (Figure 8.4-2) for all other samples. Information on these forms cross-reference the Sample ID Numbers with SDG numbers, sample tag numbers and/or other client identifiers. Each sample is clearly labeled with its lab Sample ID Number by the Sample Custodian. The same sample ID Number appears on the LIMS status report, on each sample preparation container and extract vial associated with the sample.

8.5.1.1 Sample Extract Identification:

As described in Section 8.5.1, a sample extract is identified with the same unique sample identification number as the sample from which it derives

8.5.2 Sample Login:

The sample login system at Spectrum Analytical, Inc. RI Division consists of computerized entry using LIMS (Figure 8.5-1). The information recorded onto the Workorder Report includes:

- Workorder number
- Client name
- Project name and location
- Final data report format
- Date of receipt
- Date sample collected
- Due date, fax and/or hardcopy
- EDD requirements
- Comments or notes on the workorder
- Lab Sample Identification numbers
- Client Sample Identification numbers
- Sample matrix
- Analyses required
- Case number, where used by the client
- SDG number, where used by the client

8.5.3 Sample Information:

After sample information is properly recorded and the samples have been properly logged into the LIMS, bottle labels are generated and applied to the sample containers. The Sample Custodian notifies the Project Manager or peer or supervisor to review the sample bottle labeling. This person reviews all the information associated with the samples. He/she verifies (by initialing) the correctness of the information on the Sample Condition Form or Sample Log-In Form. Sample login information is available through the LIMS to all appropriate laboratory staff. The Sample Custodian then scans the samples into the IntCOC system and posts the samples.

The Sample Custodian initiates a red workorder file. This file contains the original Sample Log-In Form or Sample Condition Form, air bills, SMO traffic reports, sample tags, workorder reports and all correspondence with the Client or SMO or others. The red workorder file is forwarded to the Project Manager for review of the login paperwork, and for updating status of the workorder in the LIMS. Once the login information is thoroughly reviewed for correctness, the red workorder file is stored in the data reporting area. Analytical data are placed in this as analyses are completed and data are reviewed.

8.6 Sample Storage and Disposal:

Samples at Spectrum Analytical, Inc. RI Division are stored in a central storage facility or in satellite designated areas, (see SOP 30.0003 Sample Receipt Storage Tracking and Disposal). After sample receipt and login procedures are completed, the Sample Custodian places the samples in the centralized walk-in refrigerator. Volatile Organic sample aliquots are released to the volatile organic lab with documentation (Figure 8.6-1).

The central storage facility is for samples only; no standards or reagents are to be stored there. Access to the centralized sample storage facility is limited by keypad entry at all times. All sample storage areas are within the secure laboratory facility.

All sample/extract refrigerators are maintained at 4°C ± 2°C. Standards are kept in freezers maintained at -10 to -20°C. The temperature is recorded electronically using temperature probes that are affixed inside all refrigerator and freezer units (see SOP #80.0020 Temperature Monitoring Systems).

When analysis is complete, any remaining sample is retained in the designated storage facility until it may be removed for disposal (see SOP 30.0024 Sample Disposal). Broken and damaged samples are promptly disposed in a safe manner. Unless there is a specific request by the client, excess, unused sample aliquots are stored for at least 30 days after the submission of compliant data (USEPA is 60 days for samples and empty containers). The samples are then disposed after such

period. USEPA and NYS ASP extracts are stored under refrigeration for at least one year. Other extracts are stored under refrigeration for up to three months, unless there is a specific agreement with the client. After such time, the extracts are disposed. All disposals are performed in a manner compliant with federal and state regulations. International samples require special disposal procedures associated with the USDA Soil Permit (see SOP #30.0024 Sample Disposal).

8.6.1 Extract Transfer:

The extracts generated during the preparation for the organic analyses are transferred from the Organic Prep Lab to the Analysis Labs. The transfer of extracts for Semivolatiles, TPH, Pesticides and PCBs, are documented electronically in the Prep Batch Log with the storage location (refrigerator ID).

Metals analysis samples that are transferred from the prep area to the analysis room are also documented in the Prep Batch Log with the storage location (ICP or Hg lab).

There is no extract transfer that occurs with either Wet Chemistry or VOA samples.

8.6.2 Extract Storage:

Semivolatile, Pesticide/PCB, and TPH extracts, which are contained in crimp top vials or screw cap vials with Teflon lined septa, are stored at 4°C ± 2°C. Semivolatile and Pesticide/PCB extracts are stored in refrigerators in the Semivolatiles Analysis room. They are catalogued numerically by workorder number that approximates chronological order, according to date of receipt. USEPA CLP extracts are stored separately within the refrigerator from sample extracts of other clients.

Excess Pesticide extracts, not analyzed, are stored in screw cap vials with Teflon lined septa in the Organic Prep Lab. In most instances, they consist of the remaining 8-9 mL aqueous and soil sample extracts and are stored chronologically by workorder.

8.7 Sample Tracking:

When a sample is removed from storage, the analyst must scan each jar or bottle taken, using the IntCOC program and their user ID. When the sample(s) are returned to the central storage facility, the analyst must scan the samples back into the system using the IntCOC program and their user ID, and return the physical samples to their original storage location. In addition to the individual's initials, the date and time is recorded. This system maintains the location of the sample at any point in time.

Chain-of-custody of a sample ensures that the sample is traceable from the field, where it was taken, through laboratory receipt, preparation, analysis and finally disposal. The primary chain-of-custody documents are used to locate a sample at any point in time.

1. The chain-of-custody form from the field describes the origin and transportation of a sample;

2. The ICOC document acceptance of a sample by Spectrum Analytical Inc., RI Division; and

3. The ICOC documents which analyst has custody of the sample after removal from storage.

4. The sample preparation logs and/or extract transfer logs document when the extracts or digestates were received by the analytical labs and where they are stored in the refrigerator.

Figure 8.4-1
USEPA CLP Sample Login Form

SAMPLE LOG-IN SHEET
FORM DC-1

Lab Name					Page _____ of _____
Received By (Print Name)					Log-in Date
Received By (Signature)					
Case Number		Sample Delivery Group No.			Mod. Ref. No.
Remarks:			Corresponding		
		EPA Sample #	Sample Tag #	Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal Nos.	_____				
3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists	Present/Absent*				
4. Airbill	Airbill/Sticker Present/Absent*				
5. Airbill No.	_____				
6. Sample Tags	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on Chain-of-Custody				
7. Sample Condition	Intact/Broken*/ Leaking				
8. Cooler Temperature Indicator Bottle	Present/Absent*				
9. Cooler Temperature	_____				
10. Does information on TR/COCs and sample tags agree?	Yes/No*				
11. Date Received at Laboratory	_____				
12. Time Received	_____				
Sample Transfer					
Fraction	Fraction				
Area #	Area #				
By	By				
On	On				

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

SAMPLE LOG-IN SHEET

Lab Name: Spectrum Analytical Inc., Rhode Island Division		Page __ of ___
Received By (Print Name)		Log-in Date
Received By (Signature)		
Case Number	Sample Delivery Group No.	Mod. Ref. No.

Remarks:	
1. Custody Seal(s)	Present/Absent* Intact/Broken
2. Custody Seal NOs.	_____ _____
3. Traffic Reports/Chain of Custody Records or Packing Lists	Present/Absent*
4. Airbill	Airbill/Sticker Present/Absent*
5. Airbill No.	_____ _____
6. Sample Tags Sample Tag Numbers	Present/Absent* Listed/Not Listed on Traffic Report/Chain of Custody Record
7. Sample Condition	Intact/Broken*/ Leaking
8. Cooler Temperature Indicator Bottle	Present/Absent*
9. Cooler Temperature	_____
10. Does information on Traffic Reports/Chain of Custody Records and sample tags agree?	Yes/No*
11. Date Received at Lab	_____
12. Time Received	_____

Sample Transfer	
Fraction	Fraction
Area#	Area#
By	By
On	On

	EPA Sample #	Aqueous/ Water Sample pH	Corresponding		Remarks: Condition of Sample Shipment, etc.
			Sample Tag #	Assigned Lab #	
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

Figure 8.4-2
Sample Condition Form

SPECTRUM ANALYTICAL, INC. RI DIVISION

Sample Condition Form

Received By:	Reviewed By:	Date:	Spectrum RI Work Order #:
Client Project:		Client:	

		Lab Sample ID		Preservation (pH)					VOA Matrix	Soil Headspace or Air Bubble ≥ 1/4"
				HNO_3	H_2SO_4	HCl	NaOH	H_3PO_4		
1) Cooler Sealed	Yes / No									
2) Custody Seal(s)	Present / Absent									
	Coolers / Bottles									
	Intact / Broken									
3) Custody Seal Number(s)										
4) Chain-of-Custody	Present / Absent									
5) Cooler Temperature										
IR Temp Gun ID										
Coolant Condition										
6) Airbill(s)	Present / Absent									
Airbill Number(s)										
7) Samples Bottles	Intact / Broken / Leaking									
8) Date Received										
9) Time Received										

Preservative Name/Lot No.:

VOA Matrix Key:

US = Unpreserved Soil	A = Air
UA = Unpreserved Aqueous	H = HCl
M = MeOH	E = Encore
N = NaHSO4	F = Freeze

See Sample Condition Notification/Corrective Action Form yes / no

Form ID: QAF.0006

Rad OK yes / no

Figure 8.4-3
Sample Condition Notification Form

Spectrum Analytical, Inc. RI Division Sample Condition Notification

Project#:_____ **Date of Receipt:**_____
Client:_____ **Received By:**_____
Client project #/name:_____

Unusual Occurance Description:

Client Contacted:

Contacted via: Phone/Fax/E-mail
Date:_____ **Time:**_____
Contacted By:_____
Name of person contacted:_____

Client Response:

Responded via: Phone/Fax/E-mail
Date:_____
Name of person responding:_____
Responding to:_____

Action Taken: _____

Form ID: QAF.0005

Figure 8.4-4
Spectrum Analytical, Inc. RI Division Chain-of-custody Form

SPECTRUM ANALYTICAL, INC.
Featuring
HANIBAL TECHNOLOGY

Page _____ of _____

CHAIN OF CUSTODY RECORD

☐ 11 Almgren Drive
Agawam, MA 01001
(413) 789-9018

☐ 8405 Benjamin Road, Ste A
Tampa, FL 33634
(813) 888-9507

☐ 175 Metro Center Blvd
Warwick, RI 02886
(401) 732-3400

Special Handling:

TAT- Indicate Date Needed: _____
· All TATs subject to laboratory approval. Min. 24-hour notification needed for rushes.
· Samples disposed of after 60 days unless otherwise instructed.

Report To: _____
_____
_____
_____
Telephone #: _____
Project Mgr. _____

Invoice To: _____
_____
_____
_____
P.O. No.: _____ RQN: _____

Project No.: _____
Site Name: _____
Location: _____ State: _____
Sampler(s): _____

1=Na_2S2O_3 2=HCl 3=H_2SO_4 4=HNO_3 5=NaOH 6=Ascorbic Acid 7=CH_3OH
8= $NaHSO_4$ 9= Deionized Water 10=H_3PO_4 11= _____ 12= _____

List preservative code below:

QA/QC Reporting Notes:

DW=Drinking Water GW=Groundwater WW=Wastewater
O=Oil SW= Surface Water SO=Soil SL=Sludge A=Air
X1= _____ X2= _____ X3= _____

G=Grab C=Composite

Lab Id:	Sample Id:	Date:	Time:	Type	Matrix	Containers: # of VOA Vials	# of Amber Glass	# of Clear Glass	# of Plastic		Analyses:								

QA/QC Reporting Level
☐ Level I ☐ Level II
☐ Level III ☐ Level IV
☐ Other _____

State-specific reporting standards:
_____

Relinquished by:	Received by:	Date:	Time:	Temp°C

☐ EDD Format _____
☐ E-mail to _____

Condition upon receipt:
☐ Ambient ☐ Iced ☐ Refrigerated ☐ DI VOA Frozen ☐ Soil Jar Frozen

Figure 8.5-1
Workorder Information Form

Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division

WorkOrder: L1458

Client ID: MITKEM_WARWICK
Project: INTERNAL TESTING
WO Name: INTERNAL TESTING
Location: WATER_TESTING, WW, 6/2012
Comments: Internal test

Case:
SDG:
PO: INTERNAL TESTING

HC Due: 07/12/12
Fax Due:
Fax Report: ☐

Report Level: LEVEL 2
Special Program:
EDD:

Lab Samp ID	Client Sample ID	Collection Date	Date Recv'd	Matrix	Test Code	Samp / Lab Test Comments	HF	HT	MS	SEL	Storage
L1458-01A	WW-6/28-G	06/28/2012 08:05	06/28/2012	Aqueous	E624	/				Y	VOA
L1458-01B	WW-6/28-G	06/28/2012 08:05	06/28/2012	Aqueous	E625	/ Needs benzidine, 1,2-diphenyhydr, n-nitrosodimethl				Y	Disposed
L1458-01C	WW-6/28-G	06/28/2012 08:05	06/28/2012	Aqueous	E335.4	/					Disposed
L1458-02A	WW-6/28-C	06/28/2012 15:00	06/28/2012	Aqueous	E200.7	/ Cd, Cr, Cu, Pb, Ni, Ag, Zn				Y	Disposed
L1458-02B	WW-6/28-C	06/28/2012 15:00	06/28/2012	Aqueous	SM5220	/					Disposed

HF = Fraction logged in but all tests have been placed on hold

HT = Test logged in but has been placed on hold

Figure 8.6-1
Volatiles Receiving Logbook Form

Spectrum Analytical, Inc. RI Division : VOLATILE SAMPLES RECEIVING LOGBOOK

VOA Log-In Date	Workorder	Client ID	Sample Numbers	Relinquished by:	Received by:	Pres. Used	F/R	Returned to R1

Logbook ID 90.0191-04/12

Reviewed By:

"Preservative Used" Key

UA = Unpreserved Aqueous **H** = HCL **A** = Air **M** = MeOH **E** = Encore

US = Unpreserved Soil **N** = $NaHSO_4$ **F** = Freeze **T** = Trace, HCL

9.0 CALIBRATION PROCEDURES AND FREQUENCIES

All purchased equipment, materials, and services must meet specific method requirements, standard requirements, or project specific requirements. These requirements are documented in the individual analytical or project SOPs.

9.1 Instruments:

Specific calibration and check procedures are given in the analytical methods referenced in Section 10. The frequencies of calibration and the concentrations of calibration standards are determined by the cited methods and any special project or contract-specific requirements. Standard calibration curves of signal response versus concentration are generated on each analytical instrument used for a project, prior to analysis of samples. A calibration curve of the appropriate linear range is established for each parameter that is included in the analytical procedure employed and is verified on a regular basis with check standards as specified in the appropriate CLP Protocols. For non-CLP work, Spectrum Analytical, Inc. RI Division adheres to the calibration criteria specified by SW-846 and/or Standard Methods for both organic and inorganic analyses. Where requested, other method specific calibration criteria are used. Refer to the individual Standard Operating Procedures listed in Figure 11.7-1 of this QAP for the specific calibration and check procedures as well as concentration and frequency requirements.

For organic analyses whenever possible, unless otherwise specified in the individual methods, the initial calibration standards (ICAL), continuing calibration verification standards (CCV), laboratory control sample spike (LCS) and matrix spike (MS) will all be from the same source. The initial calibration verification (ICV) standards are prepared from a separate source. Refer to the Standard Operating Procedures listed in Figure 11.7-1 of this QAP for the specific calibration source and procedural requirements of each method. The following are examples of calibration procedures for various instrumental systems:

GC/ECD and GC/FID – An initial calibration is performed using five different concentration levels for each parameter of interest for SW-846 analyses. The initial calibration is done on each column and each instrument, and is repeated each time a new column is installed or whenever a major change is made to the chromatographic system.

Initial calibration verification (ICV), near mid level concentration for all analytes, is performed immediately after the calibration. If the ICV does not meet method specific criteria, a new calibration curve is generated and an ICV is analyzed. If repeated ICV failures are encountered, the system is checked to find the cause of these failures, and the problem is corrected. For certain GC/FID analyses (i.e. GRO /DRO), the instrument is calibrated using individual compounds while the laboratory control sample or ICV uses a product (diesel or gasoline).

Continuing calibration verification (CCV), near a mid-level concentration for all analytes, is run at intervals determined by sample number or time allowed, as required by the individual methods. If CCV values are determined outside the upper limit of the method specified range and if no analytes were detected in the samples, the run will be accepted as valid and 'Non Detects' reported for the sample. If an analyte is detected and the CCV is out at the high end, the problem will be identified and corrected and the affected samples will be re-analyzed with a compliant CCV.

If a CCV value is out of the method specified limits at the lower limit, the cause of the problem will be identified and corrected, and all samples affected by the out of control CCV will be rerun with a compliant CCV.

For CLP-type analyses, the continuing calibration takes place at the beginning of the analytical sequence and once every twelve (12) hours throughout the analytical sequence, and again at the end of the sequence. The percent difference in calibration factors for each standard must not exceed the criteria specified by the method.

If a CCV fails to meet criteria limits, a new calibration curve will be generated and all samples affected will be re-analyzed.

GC/MS – For CLP methods, a minimum of five-level calibration (four-level for select semivolatile compounds) is carried out for each analyte per system before analysis of samples take place.

Continuing calibrations, near midpoint levels, are analyzed every twelve hours of instrument analysis time for CLP analyses.

Re-calibration takes place whenever a major change occurs in the system, such as a column change in the GC or a source cleaning of the mass spectrometer or when the continuing calibration fails to meet method specific requirements.

Tunes are performed once every twelve (12) hours of instrument run time for all CLP-type and SW846 analyses. The GC/MS system is tuned to USEPA specifications for bromofluorobenzene (BFB) or decafluorotriphenylphosphine (DFTPP) for volatile and semivolatile analyses, respectively. Extended tune time is allowed in CLP SOM protocols where an ending CCV is acceptable as an opening CCV.

More detailed instrument and method-specific calibration procedures and criteria are described in the individual analysis SOPs.

ICP/AES and ICP/MS – Instrument calibration, for each wavelength used, occurs at the start of each analysis. The calibration curve is constructed per method specification.

An initial calibration verification and initial calibration blank (ICB) are analyzed before analysis of samples. If the ICV and ICB do not meet method specific criteria for an analyte, the analyte is re-analyzed with a new calibration.

During the analysis, a continuing calibration verification (CCV) and continuing calibration blank (CCB) is analyzed at least every ten (10) samples or two hours depending on method. If either the CCV or CCB fails to meet method specific criteria for an analyte, the source of the problem is investigated. If it can be determined that the failed CCV and/or CCB is not representative (such as for instrument carryover from previous sample or from an empty autosampler tube), the CCV and/or CCB are re-analyzed and the reason for the failure documented. If a failure still occurs, further corrective action is performed, and the analyte is re-analyzed with a new calibration.

The CCV is obtained from a source independent from that of the standards. The CCV concentration for the different analytes are at method specified levels.

The Flow Injection Mercury System (FIMS) - Instrument calibration occurs at the start of each analysis. The calibration curve is constructed per method specification.

An initial calibration verification (ICV) and initial calibration blank (ICB) are analyzed before analysis of samples. If the ICV and ICB do not meet method specific criteria for Mercury, re-calibration and reanalysis are required.

During the analysis, a continuing calibration verification (CCV) and continuing calibration blank (CCB) is analyzed at least every ten (10) samples. If either the CCV or CCB fails to meet method specific criteria for Mercury, the source of the problem is investigated. If it can be determined that the failed CCV and/or CCB is not representative (such as for instrument carryover from previous sample or from an empty autosampler tube), the CCV and/or CCB are re-analyzed and the reason for the failure documented. If a failure still occurs, further corrective action is performed, and the analyte is re-analyzed with a new calibration.

The CCV is obtained from a source independent from that of the standards. The CCV concentration for Mercury is at method specified levels.

Other instrumentation:

IC- The Ion Chromatograph is calibrated each day of use. Calibration verification is analyzed at the beginning, end, and at least every 10 samples. The verification standard is from an independent source. If the calibration verification does not

meet method specific criteria for an analyte, it is re-analyzed once. If failure still occurs, a new calibration curve is established and any affected samples are reanalyzed.

pH- the meter is calibrated at two pH levels (4.0 and 10.0) before analyses of samples. The pH 7.0 buffer is analyzed as an LCS and recovery is calculated.

Lachat 8000- automated flow-through spectrophotometer is calibrated per method specification before the analyses of samples.

An initial calibration verification and initial calibration blank (if required) are analyzed before analysis of samples. If the ICV and/or ICB do not meet method specific criteria for an analyte, re-calibration must occur.

During the analyses, continuing calibration verification and continuing calibration blanks are analyzed at least every ten (10) samples. If either the CCV or CCB fails to meet specified criteria for an analyte, the source of the problem is investigated. If it can be determined that the failed CCV and/or CCB is not representative (such as for instrument carryover from previous sample or from an empty autosampler tube), the CCV and/or CCB are re-analyzed and the reason for the failure documented. If a failure still occurs, further corrective action is performed, and the analyte is re-analyzed with a new calibration.

The CCV is obtained from a source independent from that of the standards. The CCV concentration for the different analytes are at method specified levels.

SpecGenesys- manual spectrophotometer is calibrated per method specification.

Calibration curve calibration verification is analyzed at the beginning, end, and at least every 10 samples. The verification standard is from an independent source. If the calibration verification does not meet method specific criteria for an analyte, it is re-analyzed once. If failure still occurs, a new calibration curve is established and any affected samples are reanalyzed. Calibration curves are established at least quarterly.

Annual calibration and preventative maintenance is required by an outside vendor unless calibration can be performed in-house using a calibration kit.

Balances: are calibrated by an outside source on an annual basis.

The balances are calibrated externally each day of use by a lab technician with NIST traceable Class “1” or “2” weights. The weights are certified by an outside service on a regular basis, not to exceed five years.

Thermometers are calibrated once a year against a NIST-verified thermometer or as they are replaced. Digital thermometers are verified quarterly. The NIST-verified thermometers are certified by an outside certified service annually.

Gel Permeation Chromatography is used to clean samples according to CLP and client requirements. GPCs are calibrated using a calibration standard provided by Ultra Scientific, Cat. # CLP-340. Once a successful calibration is achieved it is valid for a period of seven days.

9.2 Standards and Reagents:

Standard reference materials used for routine calibration, calibration checks, and accuracy are obtained from commercial manufacturers. These reference materials are traceable to the source and readily compared to EPA references. All standards come with a Certificate of Analysis which is kept on record in the appropriate laboratories. When a chemical standard can not be purchased in solution form, a neat source may be bought. The lab must attempt to obtain the highest purity available. If the lab can not find a neat source with at least 97% purity, the laboratory must document why. In addition, the impurity correction factor must be used when calculating the standard concentration. See SOP #80.0001, Standard Preparation, Equivalency and Traceability, for more details. While most standards are traceable to NIST; however, certain projects, especially those involving pesticide registration, may necessitate the use of reference standards supplied by the client. New standards are also routinely validated against known standards that are traceable to EPA or NIST reference materials.

Organic Preparatory Lab Surrogate and Matrix spikes are prepared in the appropriate instrument labs and then QA'd by diluting the standard and analyzing it on the GC or GC/MS. Criteria for the diluted spike analysis must meet the method or in-house criteria. If acceptable, the spike is able to be used. If unacceptable, another standard is prepared and the same steps repeated. Data from the QC analysis is retained in the laboratory for reference and traceability.

Primary, intermediate and working standards are all named using specific nomenclature as designated in the QA Department SOP# 80.0001, Standard Preparation, Equivalency and Traceability.

Standards are dated and labeled upon arrival. Any material exceeding its shelf life as described by the methods in QAP Section 10 is discarded and replaced. Standards are periodically analyzed for concentration changes/degradation and inspected for signs of deterioration such as color change and precipitate formation. Standards Logbooks, which contain all pertinent information regarding the source and preparation of each analytical standard, are maintained by each of the laboratory departments in the LIMS.

See individual analytical SOPs (listed in Figure 11.7-1), sections 7 and 8 for standards preparation procedures.

Solvents are tested for purity prior to use to ensure there is no external source of contamination. For organic solvents, each lot number of solvent is QC'd prior to use. This is accomplished by concentrating an aliquot of solvent or extracting with reagent media (such as sodium sulfate) in the same manner as the samples and analyzing it for contamination by GC/MS. Any detectable analyte could render the solvent or reagent unsuitable for use. Supervisors make the final decision as to the suitability of the solvent or reagent, and whether the lot may be used for standard or sample preparation.

Chemicals and Reagents are stored in the respective laboratories during use. Backup supplies are stored in the stockroom. Reagent grade chemicals are used in all tests. All dry chemicals and reagents are given a 5-year expiration period unless designated otherwise by the manufacturer. Sometimes the viability of the reagent does not remain throughout the entire 5-year period (as determined through investigation following poor results in a preparation method blank or bench analysis, for example). In this case, the chemical or reagent is readily discarded. Acids/caustics are given a 3-year expiration period unless designated otherwise by the manufacturer. Solvents are given a 1-year expiration period unless designated otherwise by the manufacturer.

Chemicals and reagents are logged into the laboratory and each bottle is given a unique ID. The ID is based upon the date of its arrival at the laboratory. The only exceptions include cases/cycletainers of solvents and cases of acids. For solvents and acids, the boxes/cases are labeled with received date to insure first in/first out usage. All other chemicals and reagents are named using specific nomenclature as designated in the QA Department SOP # 80.0013, Reagent Purchasing and Tracking.

When a bottle is opened in the laboratory, it is inspected to ensure it meets the requirements of the method. The analyst records his or her initials on the bottle along with the date opened and the ID. Any applicable certificates of analysis (COA) are scanned and archived. They may also be stored in the individual laboratories or in the QA Department.

10.0 ANALYTICAL PROCEDURES

Spectrum Analytical, Inc. RI Division uses the methods specified in Tables 10-1 through 10-6 unless otherwise specified by the client. Spectrum Analytical, Inc. RI Division performs analyses on non-potable waters, groundwater and soil/solids. The RI Division does not perform regulatory potable (drinking) water analyses with the exception of trace metals by EPA 200.8, or environmental lead (paint chips, wipes, etc. for RIDOH compliance) testing. Associated Standard Operating Procedures related to these analytical procedures can be found in Figure 11.7-1 of this QAP.

Table 10-1
Potable Water Analytical Methods

Parameter	Method Description	Method Reference
Metals	ICP-MS	200.8

Table 10-2
Non-potable Water Analytical Methods

Parameter	Method Description	Method Reference
Metals	ICP-AES	200.7
Mercury	Cold Vapor	245.1
Cyanide	Midi-distillation Automated	EPA 335.4
Alkalinity	Titration	SM2320B
Anions Chloride Sulfate Nitrate Nitrite Orthophosphate Bromide Fluoride	Ion Chromatography	EPA 300.0
Volatile Fatty Acids Acetic Butyric Lactic Propionic Pyruvic	Ion Chromatography	EPA 300.0 Mod
pH	Electrode	SM4500 H+ B
Sulfate	Turbidimetric	SM4500-SO4 E.
Ammonia	Distillation/Titration	SM4500-NH3 B, C
Total Kjeldahl Nitrogen	Digestion Distillation/Titration	SM4500- Norg C SM4500- NH3 B, C
Orthophosphate	Ascorbic, Manual	SM4500-P E
Total phosphate	Persulfate, Manual	SM4500-P B5 & E

Table 10-2
Non-potable WaterAnalytical Methods (cont.)

Parameter	Method description	Method Reference
Chemical Oxygen Demand	Spectrophotometric(Closed Reflux)	SM5220-D
Total Organic Carbon	Combustion	SM5310 B
Phenols	Distillation, 4-AAP, Direct Photometric	SM5530 B E420.1
Total Dissolved Solids	Gravimetric	SM2540 C
Total Solids	Gravimetric	SM2540 B
Total Suspended Solids	Gravimetric	SM2540 D
Total Settleable Solids	Imhoff cones	SM2540 F
Hexavalent Chromium	Diphenyl Carbazide Colorimetric	SM 3500Cr B
Volatile Organics		
Halocarbons	Purge & Trap, GC/MS	624
Aromatics	Purge & Trap, GC/MS	624
Semivolatile Organics	Extraction, GC/MS	625
Organochlorine Pesticides/ PCBs	Extraction, GC/ECD	608
Oil & Grease (HEM, SGT)	Extraction, Gravimetric	1664A

Table 10-3
SW-846 Inorganic Analytical Methods

Parameter	Method Description	Method Reference
Metals		
Aqueous	Acid digestion	Method 3005A/3010A
	ICP/AES	Method 6010C
	ICP/MS	Method 6020A
Solid	Acid digestion	Method 3050B
	ICP/AES	Method 6010C
	ICP/MS	Method 6020A
Mercury		
Aqueous	Permanganate digestion Cold Vapor analysis	Method 7470A
Solid	Permanganate digestion Cold Vapor analysis	Method 7471B
Hexavalent Chromium		
Aqueous	Colorimetric	Method 7196A
Solid	Acid Digestion Colorimetric	Method 3060A/7196A
Cyanide		
Aqueous	Midi-distillation Automated	Method 9012B
Solid	Midi-distillation Automated	Method 9012B
pH		
Solid	Electrode	Method 9045D
Ignitability (Flashpoint)		
Aqueous	Pensky-Martens closed cup	Method 1010A
Solid	Pensky-Martens closed cup	Method 1010A Mod.
Reactive Cyanide		
Solid & Aqueous	Distillation Automated	SW 846 7.3.3.2

Table 10-3
SW-846 Inorganic Analytical Methods (cont.)

Parameter	Method Description	Method Reference
Reactive Sulfide Solid & Aqueous	Distillation Colorimetric	SW 846 7.3.4.2
Anions Chloride Sulfate Nitrate Nitrite Orthophosphate Bromide Fluoride	Ion Chromatography	SW 846 9056A
Total Organic Carbon	Combustion	SW 846 9060A
Toxicity Characteristic Leaching Procedure (TCLP)		
Aqueous	Leachate by Filtration	Method 1311
Solid	Leachate Generation	Method 1311
Synthetic Precipitation Leaching Procedure (SPLP)		
Aqueous	Leachate by Filtration	Method 1312
Solid	Leachate Generation	Method 1312

Table 10-4
SW-846 Organic Analytical Methods

Parameter	Sample Preparation	Sample Analysis
Volatile Organic Compounds		
Aqueous	Method 5030B	Method 8260C
Solid	Method 5035A	Method 8260C
1,2-Dibromo-3-chloropropane 1,2-Dibromomethane	Micro extraction GC\ECD Analysis	Method 8011
Semivolatile Organic Compounds		
Aqueous	Method 3510C Method 3520C	Method 8270D
Solid	Method 3540C Method 3550B Method 3545 Method 3570	Method 8270D
Organochlorine Pesticides		
Aqueous	Method 3510C Method 3520C	Method 8081B
Solid	Method 3540C Method 3550B Method 3545 Method 3570	Method 8081B
Polychlorinated Biphenyls (Aroclors and Congeners)		
Aqueous	Method 3510C Method 3520C	Method 8082A
Solid	Method 3540C Method 3550B Method 3545 Method 3570	Method 8082A
Total Petroleum Hydrocarbons		
Aqueous	Method 3510C Method 3520C	Method 8015B,D
Solid	Method 3540C Method 3550B	Method 8015B,D

Table 10-4
SW-846 Organic Analytical Methods (cont.)

Parameter	Sample Preparation	Sample Analysis
Herbicides		
Aqueous	Method 8151A	Method 8151A
Solid	Method 8151A	Method 8151A
Toxicity Characteristic Leaching Procedure (TCLP)		
Aqueous	Method 1311	
Solid	Method 1311	
Synthetic Precipitation Leaching Procedure (SPLP)		
Aqueous	Method 1312	
Solid	Method 1312	
Gel Permeation Chromatography (GPC)		
Aqueous	Method 3640A	
Solid	Method 3640A	
Florisil Cleanup		
Aqueous	Method 3620B	
Solid	Method 3620B	
Silica Gel Cleanup		
Aqueous	Method 3630C	
Solid	Method 3630C	
Sulfur Cleanup		
Aqueous	Method 3660B	
Solid	Method 3660B	
Sulfuric Acid Cleanup		
Aqueous	Method 3665A	
Solid	Method 3665A	

Table 10-5
CLP-Type Analytical Methods

Parameter	Method Reference
USEPA CLP Organics	OLM04.3, SOM01.2
USEPA CLP Inorganics	ILM05.4, ISM01.3
USEPA Low Level Organics	OLC03.2
NYS-ASP CLP Organics	ASP 2000/2005 SOW
NYS-ASP CLP Organics	ASP 2000/2005 SOW

Table 10-6
Other Analytical Methods

Parameter	Method Reference
Volatile Petroleum Hydrocarbons	
Aqueous	MADEP VPH 1.1
Solid	MADEP VPH 1.1
Extractable Petroleum Hydrocarbons	
Aqueous	MADEP EPH 1.1
Solid	MADEP EPH 1.1
Extractable Total Petroleum Hydrocarbons	
Aqueous	CT ETPH 99-3
Solid	CT ETPH 99-3
Diesel Range Organics	
Aqueous	ME 4.1.25
Solid	ME 4.1.25
Gasoline Range Organics	
Aqueous	ME 4.2.17
Solid	ME 4.2.17

10.1 Analytical References

1. Analysis of Extractable Total Petroleum Hydrocarbons (ETPH) Using Methylene Chloride Gas Chromatograph/Flame Ionization Detection, Environmental Research Institute, University of Connecticut, March, 1999

2. Analytical Services Protocol, Volume 1-8, New York State Department of Environmental Conservation, 2003.

3. Annual Book of ASTM Standards. Part 31-Water. American Society for Testing and Materials, Philadelphia, PA, 1981.

4. Chemical Characteristics of Marine Samples, API Publications No. 4307, API, Washington, D. C.

5. Federal Register. Vol. 72, No. 47, March 12, 2007.

6. Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100).

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9. The EPA 600 Series. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Appendix A, 40 CFR Part 136, Federal Register, Vol. 49, No. 209, 1984.

10. Methods of Soil Analysis. Part 2, Chemical and Microbiological Properties, Second Edition, American Society of Agronomy, Inc., Soil Science Society of America, Inc., Madison, WI, 1982.

11. Standard Methods for the Examination of Water and Wastewater, 18th Edition, APHA, Washington, D. C., 1992.

12. Standard Methods for the Examination of Water and Wastewater, 20th Edition, APHA, Washington, D. C., 1998.

13. Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, SW-846, 3rd Edition Final Updates I through IV. Office of Solid Waste and Emergency Response, USEPA, Washington, D. C., 1998. Status table found at http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/methstat.pdf

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15. USEPA Contract Laboratory Program. Statement of Work for Inorganic Analysis, USEPA ILM05.4, and ISM01.2.

16. Maine Health and Environmental Testing Laboratory. Modified GRO and DRO Methods, Method 4.2.17 and 4.1.25, September 6th 1995.

17. EPA Methods and Guidance for Analysis of Water, Version 2.0. includes MCAWW Methods and most current EPA Methods @ http://www.epa.gov/ost/methods/

11.0 DATA COLLECTION, REDUCTION, VALIDATION AND REPORTING

11.1 Data Collection:

Most of the lab's data is uploaded into the LIMS systems directly from the instruments. The exception is the GC's and GC/MS's in which data is first processed in Target and then uploaded into the LIMS.

Either the instrument analyst or data reporting group will upload the data into the LIMS. The person who performs the upload does a technical review to ensure recoveries of CCVs, MS, MSD, and LCS all seem to be correct. A completeness review is done at this time to ensure all applicable samples have been uploaded for all the necessary analytes.

Next, an employee with a technical background will perform the QA process of the uploaded data. This person is either a supervisor or someone with extensive experience in environmental chemistry. Corrections to the run are made at this step if necessary. When the review is complete, this technical person authorizes the data to be reported by "QA-ing" the run in the LIMS. For a more detailed view of the LIMS uploading/review procedure, see SOP # 110.0028, Data Validation/Self Inspection Procedures.

11.2 Data Reduction:

Instrument printouts, computer terminal displays, chromatograms, strip chart recordings and physical measurements provide raw data that are reduced to concentrations of analytes through the application of the appropriate calculations.

Equations are generally given within the analytical methods referenced in Section 10. Data reduction may be performed automatically by computerized data systems on the instrument, manually by the analyst, or by PCs using verified spreadsheets and/or data base software.

11.3 Data Verification:

The verification process requires the following checks to be made on data before they are submitted to the client:

- A completeness inspection is required which ensures that all required data are included in the data packages submitted to the client and that the appropriate signatures are present on the data packages.

- A contract compliance screening to ensure that contractual requirements have been satisfied.

- A consistency check to ensure that nominally identical or similar data appearing in different places within a data package are consistent with respect to value and units.

- All manual integrations are properly performed and documented.

- A correctness check to ensure that reported data have been calculated correctly or transcribed correctly.

11.4 Data Validation:

Data validation is an essential element of the QA evaluation system. Validation is the process of data review and subsequent acceptance or rejection based on established criteria.

The following analytical criteria are employed by Spectrum Analytical, Inc. RI Division in the technical evaluation of data:

- Accuracy requirements.
- Precision requirements.
- Detection limits requirements.
- Documentation requirements.

As in the case of EPA/CLP procedures, data acceptance limits may be defined within the method. As one means of tracking data acceptability, quality control charts are plotted for specific parameters determined in similar, homogeneous matrices. Control limits for non-CLP methods are statistically determined as analytical results are accumulated unless provided by method or program.

Upon completion of the evaluation, the evaluator dates and initials the data review checklist as described in Section 11.5 below.

11.5 Data Interpretation and Reporting:

Interpretation of raw data and calculation of results are performed by a scientist experienced in the analytical methodology. Upon completion of data reduction, the scientist signs for the reported results on the data review checklist. For GC/ECD, GC/FID and GC/MS, a technical peer review is performed using the data processing software prior to form generation.

The laboratory supervisor is responsible for the data generated in that department. The supervisor or other senior technical staff performs an independent review of data and completed report forms. Members of the QA staff also check the results on selected sets of data (usually 10%).

11.5.1 Report Formats:

Spectrum Analytical, Inc. RI Division uses a flexible data reporting system where final report format is based on the requirements of the client. The two most common types of data reports generated by the Spectrum Analytical Inc., RI Division are Level 2 or "commercial-format" and Level 4 or "CLP-format". The lab adapts its data report format, wherever possible, to meet customer requirements. Occasionally reports are generated that are a compromise between "commercial" and CLP-format deliverables or are designed to meet the needs of a particular regulatory format or sampling program.

Drinking water Metals samples have special reporting requirements and client notification criteria for results exceeding the MCL. Clients are notified via facsimile or e-mail of all samples that exceed any EPA maximum contaminant level (MCL), maximum residual disinfectant level or reportable concentration within 24 hours of obtaining valid data. Drinking water Metals analyses are reported using a custom reporting format that will list the associated MCL and certification status for each element. Additionally, the requirement for the 24 hour MCL exceedence report will be highlighted in the comment section of the Subcontract Work Order for any subcontracted potable water samples.

Commercial data reports are generated using the LIMS. All instrumental analysis data are uploaded from instruments to the LIMS by electronic data transfer. Non-instrumental analysis data or sample preparation data are manually entered into the LIMS. All manual data entry steps are double-checked to insure they are correct, and instrumental data are spot-checked to insure the proper functioning of the data upload system. All data receive a 100% review before they are released to the client as final.

CLP data reports are generated using specialized CLP report modules in the LIMS for all inorganic and most organic analyses. These reports also undergo a 100% review before they are released to the client in their final form.

Records are maintained for all data, even those results that are rejected as invalid.

11.6 Levels of Data Review:

Spectrum Analytical, Inc. RI Division employs five (5) levels of data review. These are based on requirements outlined in several government and other environmental analysis programs including the U. S. Army Corps of Engineers, Air Force Center for Environmental Excellence (AFCEE), Naval Facilities Engineering Service Center (NFESC), HAZWRAP, Department of Defense

ELAP (QSM), EPA Contract Laboratory Program (SOM/ISM), as well as commercial engineering firm programs.

The data review and evaluation process is structured to insure that all data reported to customers has been thoroughly reviewed and approved using a multi-step process designed to identify and correct any error. At any step in the data evaluation and review process, the reviewer has the responsibility and authority to return any data not meeting requirements back to the previous step for re-analysis or correction. No reports are released to the client as final data without successfully passing through each step in the data evaluation and review process. The steps of the data review process are documented, generally using a checklist. Several checklists are used, depending on the type and format of analysis data being reviewed. Any data released prior to the completion of the full review process are released with the statement that the data is preliminary pending final review. The word "Preliminary" is automatically printed on the bottom of all data sheets that are generated prior to completion of data review.

The five levels of data review are detailed in SOP # 110.0028 Data Validation/Self Inspection Procedures. A Flow chart of the data review process follows in Figure 11.6-1.

11.7 Document Control:

All login sheets, Chains-of-Custody (COC) and Sample Condition Forms (SCF) and other sample transmittal documentation are generated in Sample Receiving. A red Workorder File is initiated to contain all workorder-specific hard copy documents. Samples are signed in/out of the sample receiving area by analysts. In the Prep lab, samples and all pertinent information is recorded into logbooks. Once samples are moved to the instrument lab, the transfer of extracts is documented in the electronic transfer logbook (ICOC). In the instrument lab, the analysis of extracts is recorded in the instrument run log. All analysis data, including ICAL, CAL and raw data are acquired using computer-controlled instruments, and stored on the hard drive of the computer performing data acquisition. Data are automatically copied to the company file server after acquisition. Organics analysis data are processed using Thru-Put Systems' Target software. This system creates a folder on the file server for each analysis fraction for each work order or SDG. This folder contains raw data, processed analysis results, instrument tune, initial calibration and continuing calibration results as well as a copy of the data processing method used. This allows for long-term archiving and complete reconstruction of the data at any time in the future. Organic data files are also uploaded into LIMS so reporting forms can be printed. The raw data are printed electronically and arranged with all appropriate sample-preparation and instrument run logbook page copies for technical review.

Inorganic data files are uploaded into LIMS and reporting forms are printed electronically. The original instrument data files and the processed SDG are

stored on the file server where they can later be archived by the LIMS Administrator. PDF printouts for reporting forms, instrument data output and all associated preparation logbook page copies are assembled for technical data review through a custom reporting system, Package Maker.

Spectrum RI is primarily utilizing a paperless reporting system with the exception of our EPA CLP reports which require a hard copy report.

See SOP # 110.0029, Electronic Data Management for a detailed description of data management activities used to support laboratory activities.

Following technical review and generation of the report narrative, results go into the workorder file in data reporting. The original copy or electronic pdf version (dependent on client requirements) of the report is sent to the client. Spectrum offers our clients secure access to their pdf reports and EDDs via our website eServices portal. All other information associated with the report, including data review checklists are kept in the red workorder file. The non-reported data (NRD) is scanned into the optical file database for long-term archiving. As documents are scanned into the database they are recorded for permanent storage on hard drives within the fileserver. The archived electronic data is kept for a minimum of ten (10) years or according to contract/program requirements. Prior to the use of the optical file database, hardcopy reports and NRD were shipped to an offsite storage area where they will remain for a minimum of ten (10) years. After this time, these older files will be destroyed.

11.7.1 Logbooks:

All logbooks are issued and controlled by the QA Department. Logbooks are given a unique ID that includes the mm/yy the logbook was printed. Laboratory personnel must sign for the logbook when it has been released by the QA Department. When logbooks are complete, the analyst returns them to the QA Department for archiving unless still needed for reference in the lab. A new logbook is released. The archived logbooks are stored in an on-site storage box for approximately 4-6 months and then are stored in an off-site storage facility or may remain on-site depending on storage space. Refer to SOP # 80.0040, Logbook Use, Review, and Control for more detail. In addition, refer to SOP # 110.0027, Documentation Policy and Procedures for details on Spectrum Analytical, Inc. RI Division's Logbook policies. Logbooks are archived for a minimum of ten (10) years or according to contract/program requirements.

11.7.2 Workorder/Data Files:

Spectrum Analytical, Inc. RI Division is a secured, limited access building. The doors are secured with a keypad entry system. All hard copy information pertaining to the analysis of samples is maintained and stored in a workorder file folder. This information includes all login

sheets, COC, SCF, bench sheets and printed analytical data. Electronic data are also stored by laboratory workorder number on the company file server, and in the optical file database of completed reports and NRD as mentioned in section 11.7. File folders containing any remaining workorder information are stored in an off-site storage facility or may remain on-site for a total of 10 years.

The off-site storage facility referred to in the above sections is a locked storage area. Access is limited to the Laboratory Director or his designee and request to retrieve a file will be made to this person.

In the event Spectrum Analytical, Inc. RI Division changes ownership, the maintenance, control, storage and eventual disposal at the end of the appropriate time period, of all records, including client data and QA/QC files, will transfer to the new owners.

In the event Spectrum Analytical, Inc. RI Division decides to cease operations, clients will be notified prior to the cessation of operations and their files/records will be made available to them. Within a designated time period after notification, the client will be responsible for taking custody and the future maintenance of their records. If the client determines they do not want to maintain the records, these will be disposed of properly.

11.7.3 Standard Operating Procedures (SOPs):

SOPs are prepared by the Lab Supervisor and laboratory personnel in conjunction with the QA Director. The QA Director/Staff downloads a copy of the current SOP to the network at Public on 'Bernoulli'. The SOPs can be found in Q:\QA_SOPs. In addition a .pdf file of the SOP is located in Q:\QA_PUBLIC\PDF-MITKEM SOPs. A list of the current SOPs in use at Spectrum Analytical, Inc. RI Division is given in Figure 11.7-1.

The laboratory staff revises the SOPs by making changes to the document that is then reviewed by the department supervisor only if the supervisor is not the party responsible for the revisions. Any additional changes are made at this point.

The QA Department is notified that revisions are completed. The QA Director/Staff moves the revised copy of the SOP to the QA directory, QA Safety/SOPs Needing QA Revision. The QA Director makes changes to the document to include revision number and date and title clarification, if necessary. Changes from the last revision are clearly marked using 'Track Changes' in Microsoft Word.

The QA Director prints a searchable pdf copy of the SOP. At this time, hard copies of several pages are printed for original signatures of the Laboratory or Technical Director, and the QA Director. The effective date is then added to the SOP and the signed pages are scanned and inserted into the pdf document. If an older version of the SOP exists, it is moved to its archive location. The new version will be moved into the Spectrum Analytical, Inc. RI Division Intranet SOP Database as the only version accessible by laboratory personnel. Each analyst who performs any duties related to the SOP must review the new version and enter electronically that he or she has read and understands the material there.

SOP review/revisions occur on an annual basis. The procedure for preparing, reviewing, approving, revising and distributing SOPs as well as the SOP Revision Schedule are described in SOP No. 80.0012.

Minor changes to the SOP between revision dates can be done as needed. Minor changes are recorded in the Revision Record that is a part of the master copy. Edits are clearly marked. This allows readers quick access to the changes.

11.7.4 Quality Assurance Manual:

The lab will review the QA Manual annually at a minimum. Past versions of the QA Manual are maintained and archived by the QA Director in the same manner as SOPs. Edits to the QA Manual are made by the QA Director in conjunction with the laboratory management. Spectrum Analytical, Inc. RI Division will amend the QAP and any affected SOPs within 14 days when technical changes (or any of the circumstances outlined in the USEPA SOW for SOM or ISM, Exhibit E, section 5.3.2) occur. The revised QAP with visible markups will be sent to the USEPA as per section 5.3.2.1.

11.7.5 Method Updates:

In most cases it is the laboratory's policy to implement new revisions of frequently used methods within six months of the date the method revision is promulgated or published as a final method (non-CLP methods, for CLP methods see below). The QA Director, Deputy Director for Quality Services, Technical Director and Laboratory Director make the final decision on when a method revision will be adopted by the laboratory. Additionally, if a client specifically requests or mandates that an "older" method, Spectrum Analytical, Inc. RI Division will advise the client that it is not the most recent method. If the client still insists upon the older method, the lab will comply and make a note in the narrative.

When the laboratory is in the middle of a client's project, the lab will continue using the same revision for the entire sampling event unless advised otherwise by the client. Consequently, once the laboratory has formally adopted a new method revision, both the old and new revision may be in use at the same time, depending on the project.

If a client should not specify which methods to be used, the methods employed by the laboratory shall be fully documented and validated. Additionally, the methods shall be published in a reputable technical journal or text or by a reputable technical organization or instrument manufacturer.

Revisions to USEPA CLP methods are required to be implemented within 14 days of notification when the EPA modifies the technical requirements of the statement of work, or the contract. At this same time, the QAP will be amended as necessary as noted in section 11.7.4.

Laboratory-developed methods can be used as long as they have been documented and validated by qualified personnel. In all cases the client should be notified.

Figure 11.6-1
Data Review Flow Diagram

Spectrum Analytical, Inc. RI Division
Review Process Flow Diagram

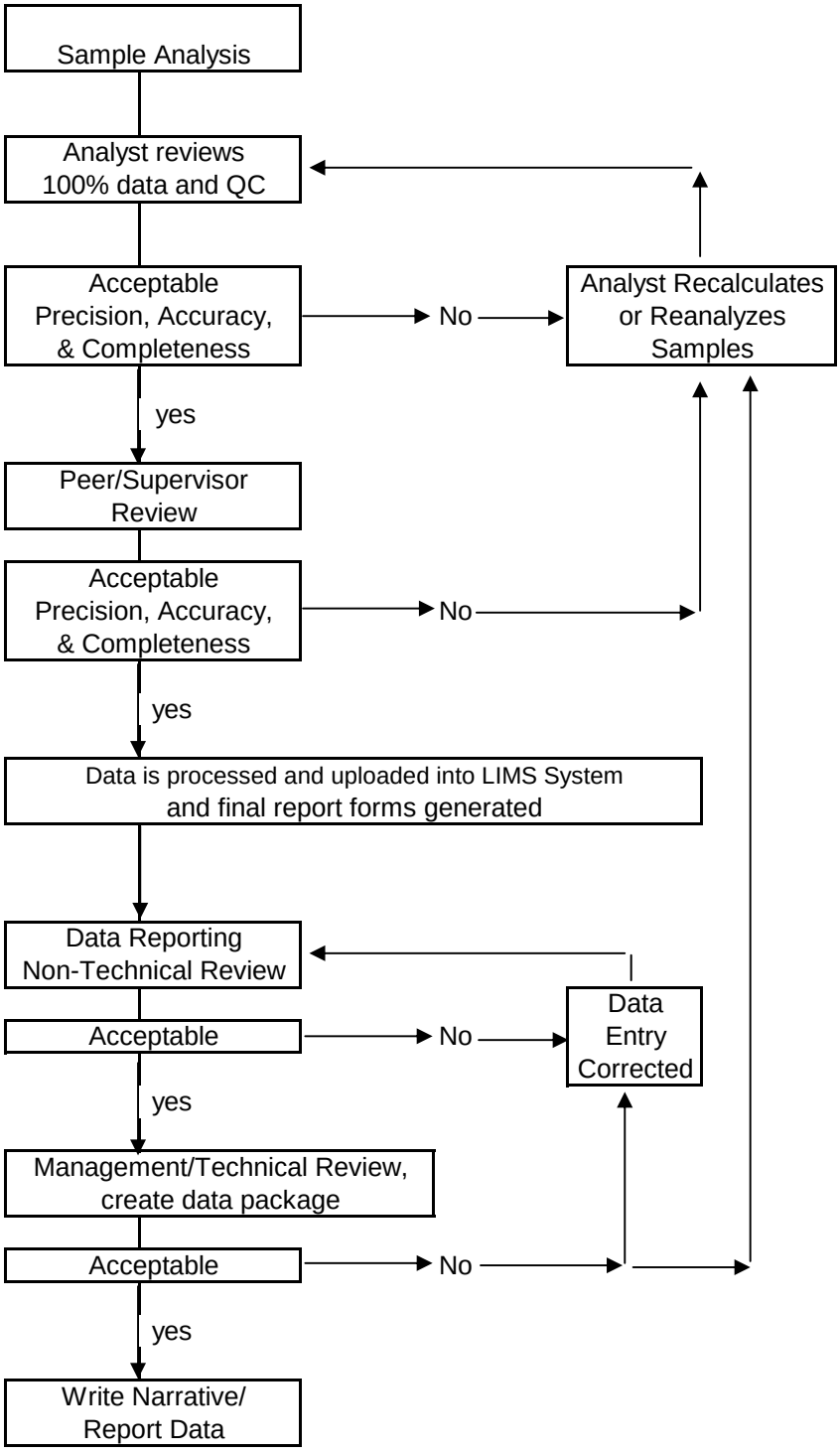


Figure 11.7-1
Standard Operating Procedures (SOPs)

Spectrum Analytical, Inc. Rhode Island Division

Standard Operating Procedures (SOPs)

Master List

SOP #	Title
10.0016	Assembly of Inorganic CLP and CLP-type Reports
10.0017	Assembly of Organic CLP and CLP-type Reports
10.0018	Assembly of Commercial Data Reports
10.0021	Data Report Options
10.0036	EPA/SOM Organic Data PDF Bookmarking
10.0037	EPA/ISM Inorganic Data PDF Bookmarking
20.0003	Logging Workorders into Omega
20.0005	Level 2 LIMS report preparation
30.0002	Bottle order preparation
30.0003	Sample Receipt, Storage, Tracking and Disposal
30.0024	Sample and Waste Disposal
30.0030	ICOC Procedures using IntCOC program
50.0004	Glassware Cleaning - Organics
50.0027	Organic Preparation of Aqueous/Soil Samples for Chlorinated Herbicides by SW-846 Method 8151A
50.0030	SOM01.2 Sulfur Cleanup
50.0031	SW-846 Method 3665A Acid Cleanup
50.0032	Gel Permeation Chromatography by SW-846 Method 3640A
50.0033	SW-846 Method 3620B Florisil Cleanup
50.0034	SW-846 Method 3630C Silica Gel Cleanup
50.0035	Oil&Grease (HEM&SGT) by Method 1664 Revision A
50.0036	SW-846 Method 3660B Sulfur Cleanup

Spectrum Analytical, Inc. Rhode Island Division

Standard Operating Procedures (SOPs)

Master List

SOP #	Title
50.0050	Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid (Method 3520)
50.0051	Organic Preparation of Aqueous Samples by Separatory Funnel (Method 3510)
50.0052	Organic Preparation of Soil Samples by Sonication (Method 3550)
50.0053	Organic Preparation of Soil Samples by Soxhlet (Method 3540)
50.0054	Organic Extract Filtration and Concentration Techniques
50.0060	Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid for Pesticides/Aroclors for SOM01.2
50.0061	Organic Preparation of Aqueous Samples by Separatory Funnel for Pesticides/Aroclors for SOM01.2
50.0062	Organic Preparation of Solid Samples by Sonication for Pesticides/Aroclors for SOM01.2 by Method 3550B
50.0063	Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid for Semivolatiles for SOM01.2
50.0064	Organic Preparation of Solid Samples by Sonication for Semivolatiles for SOM01.2
50.0100	Preparation of Soil Samples by MSE by Method 3570
50.0101	Preparation of Soil Samples by PFE by Method 3545
50.0102	Percent Lipid Determination in Tissue Samples
60.0002	Pesticide/PCB Analysis by EPA Method 608
60.0003	Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron Capture Detector Analysis by SW846 Method 8082A
60.0006	Determination of Pesticides by Gas Chromatography/Electron Capture Detector Analysis by SW846 Method 8081B
60.0007	EDB/DBCP by EPA Method 504.1 and SW-846 8011

Spectrum Analytical, Inc. Rhode Island Division

Standard Operating Procedures (SOPs)

Master List

SOP #	Title
60.0034	Determination of Chlorinated Herbicides by Gas Chromatography/Electron Capture Detector Analysis by
60.0048	Aroclor Analysis GC/ECD by USEPA SOW SOM01.2
60.0049	Pesticide Analysis GC/ECD by USEPA SOW SOM01.2
60.0050	Total Petroleum Hydrocarbons by GC-FID using EPA SW-846 Methods 8015/State Methods
60.0053	PCB Congeners by SW-846 Method 8082 (MOD)
60.0054	PCB Homologs by E680 GC/MS SIMS (MOD)
70.0011	Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8270D
70.0030	Screeening for Semivolatile Organic Analysis by Gas Chromatography/Mass Spectrometry for SOM01.2
70.0033	SIM Analysis by GC/MS (Modified EPA Method 8270D)
70.0035	Semivolatile Organic Analysis by SIM Gas Chromatography/Mass Spectrometry for SOM01.2
70.0048	Semivolatile Organic Analysis by Gas Chromatography/Mass Spectrometry for SOM01.2
70.0051	Semivolatile Organics by GC/MS for Aqueous Samples by EPA Method 625
80.0001	Standard Equivalency/Traceability
80.0002	Client Complaint Policies
80.0004	QA Data Pkg Review
80.0005	Method Detection Limit Determination
80.0006	Internal Audit Procedures
80.0007	Corrective Action Procedures

Spectrum Analytical, Inc. Rhode Island Division

Standard Operating Procedures (SOPs)

Master List

SOP #	Title
80.0009	Newly Implemented Methods (Demonstration of Acceptable Performance)
80.0010	Control Chart Generation and Use
80.0012	The Production of Standard Operating Procedure
80.0013	Reagent Purchasing & tracking
80.0016	Training Procedures and Tracking
80.0020	Temperature Monitoring Systems
80.0030	Labware Volume Verification
80.0040	Logbook Use, Review, and Control
80.0050	Performance Testing Procedures
90.0012	Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8260C
90.0035	Low/Med Volatile OrganicsAnalysis GC/MS by USEPA SOM01.2
90.0036	Trace Volatile OrganicsAnalysis GC/MS for USEPA SOM01.2
90.0038	Gasoline Range Organics by GC/FID using Methods SW-846 8015 and Maine 4.2.17
90.0040	Trace Volatile OrganicsAnalysis GC/MS using SIM for USEPA SOM01.2
90.0052	Volatile Organics by GC/MS for Aqueous Samples by EPA Method 624
90.0060	Methane, Ethane, and Ethene by GC/FID Method RSKSOP-175
100.0001	Glassware Cleaning - Inorganics
100.0002	Alkalinity (by Standard Method 2320)
100.0003	Sample Preparation of Aqueous Samples by Acid Digestion ICP (3005/3010)
100.0004	Total Cyanide by Automated Colorimetric with Midi-distillation by SW846 9012B

Spectrum Analytical, Inc. Rhode Island Division

Standard Operating Procedures (SOPs)

Master List

SOP #	Title
100.0005	Determination of Metals and Trace Elements in Water and Waste by ICP - Atomic Emission Spectrometry by EPA Method 200.7
100.0006	ICAP 3000XL/4300DV Operation
100.0007	Aqueous sample Prep E200.8
100.0010	Nitrite Analysis by Standard Method 4500-NO2 B
100.0011	pH Value by Standard Methods 4500-H+ B
100.0012	Mercury Analysis in Aqueous Samples by Flow Injection Analysis System for Atomic Analysis by Method 7470A/7471B
100.0013	Total and Ortho Phosphate using Ascorbic Acid Method by Standard Method 4500-P E
100.0014	Mercury (Manual Cold Vapor Technique) by EPA Method 245.1
100.0015	The Preparation of Waste Samples for reactive Cyanide and Sulfide; Determination of Reactive Cyanide by Automated Colorimetric Method and Reactive Sulfide by Spectrophotometric Method SW-846 Methods 7.3.3.2 and 7.3.4.2
100.0016	Preparation of Soil Samples for Sulfide Analysis by Modified SW-846 Method 9031
100.0017	Inorganic Analysis of Sulfates in Aqueous Samples by SM 426 C 15th Ed and SM4500 SO4 E-.
100.0018	Inorganic Analysis of Sulfides in Aqueous Samples (Methylene blue method)
100.0019	Total Dissolved Solids Dried at 180°C by Standard Method 2540 C
100.0020	Total Solids Dried at 103-105°C by Standard Method 2540 B
100.0021	Total Suspended Solids Dried at 103-105°C by Standard Method 2540 D
100.0022	TKN Distillation and Determination by Manual Spectrophotometric Analysis by Standard Method 4500-N
100.0023	Color Analysis by Visual Comparison by Modified Standard Methods 2120B
100.0024	Flashpoint Analysis by SW846 Method 1010A
100.0025	Total Organic Carbon by Methods SW-846 9060A and SM5310B

Spectrum Analytical, Inc. Rhode Island Division

Standard Operating Procedures (SOPs)

Master List

SOP #	Title
100.0026	Settleable Solids by Standard Method 2540 F
100.0027	Paint Filter Liquids Test by SW-846 Method 9095A
100.0028	Carbon Dioxide (CO2) and Forms of Alkalinity by Calculation by Standard Method 4500-CO2 D
100.0029	Ferrous Iron Analysis by Standard Method 3500-Fe B, Phenanthroline Method
100.0030	Phenols Analysis by EPA Method 420.1 and Standard Method 5530 B & D, Cleanup and Direct Photometric Method
100.0032	Total Volatile Solids for Solids by SM 2540 E, E160.4; Fixed and Volatile Solids Ignited at 550 C
100.0033	Total Cyanide by Auto-Colorimetric with Midi-Distillation by EPA Method 335.4
100.0053	ISM01.3 ICP-AES Analysis
100.0054	ISM01.3 ICP-MS Analysis
100.0055	Mercury Preparation and Analysis by ISM01.3
100.0056	Cyanide Preparation and Analysis by ISM01.3
100.0100	Sample Preparation of Soils by Acid Digestion for ICP/MS (3050B/6020A)
100.0103	AVS and SEM
100.0104	Sample Preparation of Soils by Acid Digestion for ICP/AES (3050B/6010C)
100.0106	Chemical Oxygen Demand Determination SM5220D
100.0110	Determination of Metals in Water and Wastes by Inductively Coupled Argon Plasma Mass Spectrometry by SW846 Method 6020A
100.0111	Determination of Metals in Water and Wastes by Inductively Coupled Argon Plasma Atomic Emission Spectrometry by SW846 Method 6010C
100.0112	pH in Soil Samples by SW846 9045D/SOM1.2
100.0113	Determination of Metals and Trace Elements in Water by ICP - MS by EPA Method 200.8
100.0121	ICP Aqueous Preparation by ISM01.3

Spectrum Analytical, Inc. Rhode Island Division

Standard Operating Procedures (SOPs)

Master List

SOP #	Title
100.0122	Prep of Soil, Wipe/Air Filter for ICP Analysis by ISM01.3
100.0201	Ammonia Distillation & Determination SM4500-NH3 B&C
100.0208	Inorganic Analysis of Hexavalent Chromium in Soil Samples by SW846 Methods 3060A & 7196A
100.0209	Mercury Speciation…SW846 Method 3200
100.0308	Inorganic Analysis of Hexavalent Chromium in Aqueous Samples by SM 3500 Cr +6 B
100.0400	Inorganic Anions by IC EPA 300.0 and 9056A
100.0410	TOC in Soil by Lloyd-Kahn and SW-846 9060
100.0420	Volatile Fatty Acids by IC using EPA 300.0 (modified)
100.0430	Walkley Black TOC in Soil
100.0440	Total, Fixed and Volatile Solids in solid/semisolid samples by SM2540G
110.0006	Thermometer Calibration
110.0007	Balance Calibration
110.0008	Manual Integration of GC, IC and GC/MS Chromatograms
110.0012	Laboratory Security
110.0013	North Carolina Samples
110.0021	Bids and Proposals
110.0023	Project Management
110.0025	Toxicity Characteristic Leaching Procedure by SW846 Method 1311
110.0026	Handling of Evidentiary Materials
110.0027	Documentation Policy and Procedures
110.0028	Data Validation-Self Inspection Procedures
110.0029	Electronic Data Management
110.0031	Synthetic Precipitation Leaching Procedure by SW-846 Method 1312
110.0032	ASTM Leachate Procedure D3987-06

Spectrum Analytical, Inc. Rhode Island Division

Standard Operating Procedures (SOPs)

Master List

SOP #	Title
110.0034	Sample Data Control for Inorganic CLP (ILM/ISM)
110.0035	Sample Data Control for Organic CLP (SOM)
110.0038	Percent Solids Determination as Required for Various SW-846 and EPA Methods
110.0039	Sub-Sampling for Soil and Solid Samples
110.0040	Instrument Maintenance
110.0041	Multiple Extraction Procedure by SW846 EPA Method 1320
110.0043	Standard Elutriate Preparation
110.0060	Tissue Sample Preparation

12.0 LABORATORY QUALITY CONTROL CHECKS

Spectrum Analytical, Inc. RI Division's analytical procedures are based on sound quality control methodology, which derives from three primary sources:

1. Specific EPA and other approved analytical methods, and

2. "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" (EPA 600/4-79-019).

3. Standards for Good Laboratory Practice.

In the application of established analytical procedures Spectrum Analytical, Inc. RI Division employs, at a minimum, the QC protocols described in the references found in the Analytical Methods section of this document. Specific projects may require additional quality control measures, due to such factors as difficult sample matrices or use of innovative techniques. For those projects Spectrum Analytical, Inc. RI Division will recommend and implement, subject to client approval, QC measures to produce data of known quality.

Each of the Spectrum Analytical, Inc. RI Division laboratory departments have an individual QC program, which includes, but is not limited to, the practices described below.

12.1 Method Detection Limit Determination/Verification:

Method Detection Limits are developed annually for certain inorganic and many organic analyses. Per NELAC Standards, MDLs are not required where target analytes are not reported below the lowest calibration standard concentration. For these analyses, results are only reported within the calibration range, and MDLs are not appropriate or needed. The reporting limit for these compounds is the concentration of the lowest standard in the calibration. For certain inorganic analyses and most organic analyses, Spectrum Analytical, Inc. RI Division typically reports analytes below the lowest level of the calibration range, but above the MDL, as estimated and are qualified with the "J" flag. Spectrum Analytical, Inc. RI Division reports estimated values below the calibration range for those analyses where results are able to be confirmed as in dual column confirmation, or by two concurrent determinative tests such as retention time and mass spectra as in GC/MS analyses. For these analyses MDLs are determined or verified annually, depending on program requirements.

MDLs are determined for all test methods where required by specific program or state regulations. Methods analyzed for the State of Massachusetts which do not detail MDL requirements within the published method, require preparation and analysis of the MDL samples over a minimum of three days. This is believed to

better mirror real world samples and day to day variability of preparatory and analytical steps.

In addition, to address special project requirements, MDLs can be determined for those tests which are not routinely reported below calibration range. If a client requests results to be reported below the calibration range without an MDL study, this is clearly identified in the workorder narrative.

Following an MDL study, the determined limits are verified by the analysis of an MDL Verification Standard. This standard is analyzed at approximately 2 to 3 times the calculated MDL for single analyte tests or 1-4 times the calculated MDL for tests with multiple analytes. This spike concentration is also referred to as the Limit of Detection in Department of Defense Quality Systems Manual (DoD QSM). DoD QSM requires quarterly verification of the LOD. For more details refer to SOP 80.0005 Determination of Method Detection Limits.

12.2 Personnel Training:

Chemists who begin their employment at Spectrum Analytical, Inc. RI Division are to be instructed under the lab's Safety Training Program within the first month. The Safety Training Program includes laboratory basics, safety video and testing, and MSDS instruction.

Before performing any analyses, a chemist is required to read the appropriate protocols and SOPs. The chemist is required to sign off on all documents read in the electronic SOP database located on our lab Intranet.

The new analyst must become familiar with the laboratory equipment and the analytical methods, and begins a training period during which he or she works under strict supervision. Independent work is only permitted after the chemist successfully completes an accuracy and precision study.

The accuracy and precision study is also commonly referred to as a Demonstration of Capability exercise. Upon the successful completion of the Initial Demonstration of Capability exercise, the QA Department issues a Demonstration of Capability Certificate (IDOC) which is signed by both the QA Director and Laboratory Director.

Demonstration of Capability studies requires the acceptable mean recovery of 4 LCS samples for each matrix or the acceptable analysis of a blind spike sample such as a Performance evaluation sample. Acceptance limits are established by the method. It is necessary to pass the study whether for extraction and/or analysis.

Annually thereafter the employee must perform an acceptable demonstration of capability study to document continued acceptable performance in his/her

particular preparatory or analytical method specialty. This is referred to as the On-going DOC. All DOCC documentation is filed in the employee's personnel folder, which is stored in the QA Department/or in the electronic personnel folder as the system has transitioned to a paperless filing system for DOCC.

Initial and on-going personnel training include data integrity training. The 4 required elements of the data integrity system include: 1) data integrity training, 2) signed data integrity documentation, 3) in-depth, periodic monitoring of data integrity, and 4) data integrity procedure documentation.

Data integrity training topics will include the need for honesty and full disclosure in all analytical reporting, how and when to report integrity issues and what those issues could be. Employees will understand that infractions of data integrity procedures can result in an investigation that could lead to serious consequences which include immediate termination, and civil or criminal prosecution. At the start of employment all new employees read, discuss and sign a Confidentiality, Ethics and Data Integrity Agreement. Annually, an on-going integrity training session is held. An attendance sheet will be generated for every integrity session. These sheets are filed in the QA Office under "Training". Another option for the annual training session is having all staff review refresher materials online and documents their having done so. This is done within the framework of the SOP database on the lab's intranet.

Data integrity procedures are reviewed and updated annually by senior management.

Training for the EPA Statement of Work occurs according to the above requirements. In addition, analysts are required to read the CLP Statement of Work as a part of the documentation training.

12.3 Control Charts:

For organic and inorganic analyses, the recoveries of analytes in the lab control samples are plotted on control charts. These charts are used to establish control and warning limits.

12.3.1 Control limits are calculated ,compared, and/or updated at least annually from the LCS, MS/MSD, and Surrogate data points for each analyte and matrix using the following equations:

$$Average(\bar{x}) = \frac{\left[\sum_{i=1}^{n} x_i\right]}{n}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(x_i - \bar{x}\right)^2}{n-1}}$$

In which:

SD = Standard Deviation
N = number of data points

Warning Limits = Average ± 2 * SD

Control Limits = Average ± 3 * SD

12.3.2 Control limits must be approved by the QA Director and by the Laboratory Director prior to adoption by the laboratory. In the event that limits are wider than method recommended limits, the method recommended limits may be adopted and the analytical procedure will be re-evaluated and/or re-determined to identify possible causes. Additionally, in the event that control limits are tighter than 15% from the average, the lab may adopt a control limit of ±15% from the average. If in the experience of the laboratory, statistical control limits are unreasonably wide or narrow, alternative limits may be used until appropriate statistical limits are developed. Alternative limits are based on sources such as DoD QSM published guidelines, EPA limits from the specific test method or from similar methods, laboratory experience with the method or other sources.

12.3.3 Control charts are plotted in EXCEL using the LIMS system.

Data from each laboratory is uploaded into the LIMS. The compounds, recoveries, and date analyzed for each test are recorded in the system. In order for LIMS generated control limits to be valid, all data, including data not meeting existing recovery criteria, must be uploaded. A control chart is then printed for review by the QA Director and by the Lab Supervisor. Out of control situations noted on the control chart are discussed with the Supervisor or Laboratory Director by the QA Director.

An example control chart is presented as Figure 12.3-1. LCS data must be reviewed and evaluated daily against the Control Limits to establish that the system is in control.

12.3.4 The following situations constitute an out of control situation on a control chart:

- One data point above or below the Control Limit line.
- Two consecutive data points above or below the Warning Limit line.
- Six or more consecutive data points above the Average Line or six or more consecutive data points below the Average Line. This situation suggests a trend and suggests the procedure has been changed in some way (for better or worse). The cause for this trend must be investigated.

12.4 General QC Protocols:

12.4.1. Organics Laboratory:

- Trip blanks and holding blanks, when applicable, are analyzed to detect contamination during sample shipping, handling and storage.
- Method blanks, at a minimum of one in every 20 samples, are analyzed to detect contamination during analysis.
- Volatile organic method blanks are analyzed once during each analytical sequence.
- One blank spike (Laboratory Control Sample or LCS) consisting of an analytical sample of laboratory water, anhydrous sodium sulfate, or Ottawa sand with every batch of 20 or fewer samples, is analyzed to determine accuracy.
- Sample spikes and spike duplicates, as requested, are analyzed to determine accuracy and the presence of matrix effects. The Relative Percent Difference (RPD) is also determined for matrix spike/matrix spike duplicates to measure precision. The criteria followed are stated in the individual methods. For batches without a sample duplicate (for example, if insufficient sample volume is provided), a duplicate blank spike (LCSD) is performed to provide for precision measurement.

- Performance evaluation samples from EPA and state agencies are analyzed to verify continuing compliance with EPA and NELAC QA/QC standards.

- Surrogate standards are added to samples and calculations of surrogate recoveries are performed to determine matrix effect and extraction efficiency.

- Internal standards for GC/MS analysis are added to sample extracts to account for sample-to-sample variation.

- Analysis of EPA traceable standards (ICV) to verify working standard accuracy and instrument performance.

- Initial multi-level calibrations are performed to establish calibration curves.

- Instrument calibration is established or verified with every analytical sequence.

- Tuning of GC/MS systems once every 12 hours for CLP and SW-846 methods or 24 hours for methods 624/625 to method specifications is implemented for consistency in data generation.

- Quarterly analysis of LOD and/or LOQ check samples to verify low level detection and reporting limits for Department of Defense QSM programs.

- Annual Verification of MDL for NELAC/TNI.

When QC limits are not met during an analytical run, the source of the problem must be investigated. Following an evaluation of the data, those samples affected must be re-analyzed after the problem has been solved. If QC limits continue to be out of control, the instrument must be checked and/or a service call made and/or further corrective action implemented.

12.4.2. Inorganic Laboratory:

- Trip blanks are analyzed when applicable, to detect contamination during sample shipping, handling and storage.

- Method blanks are analyzed at a minimum of one every 20 samples, to detect contamination during analysis.

- One matrix spike of an analytical sample or laboratory water or soil is made and spike recoveries are calculated with every batch up to 20 samples to determine accuracy. Duplicate samples are analyzed and the RPD between the sample and duplicate is calculated for every batch up to 20 samples. If insufficient volume of sample is received, a note is made in the appropriate preparation logbook.

- Performance evaluation samples from EPA and state agencies are analyzed to verify continuing compliance with EPA and NELAC QA/QC standards.

- Metals analysis instruments are calibrated for every analytical run.

- Analysis of EPA traceable standards (ICV) to verify working standard accuracy and instrument performance.

- QC/LCS checks samples are analyzed during every analytical batch of up to20 samples in order to document accuracy.

- Quarterly analysis of LOD and LOQ check samples to verify low level detection and reporting limits for Department of Defense QSM programs.

- Annual Verification of MDL for NELAC/TNI.

When QC limits are not met during an analytical run, the source of the problem must be investigated. Following an evaluation of the data, those samples affected must be re-analyzed after the problem has been solved. If QC limits continue to be out of control, the instrument must be checked and/or a service call made and/or further corrective action implemented.

12.5. Lab Pure Water used for method blanks and dilutions:

Spectrum Analytical, Inc. RI Division uses several systems to generate analyte-free water for use in the laboratory. These systems generate high quality, analyte free water dedicated to the needs of specific analyses.

12.5.1. For inorganic analyses the wet chemistry and metals labs use a US Filter mixed-bed deionization system followed by particle and carbon filters. This is followed by a polishing system using Barnstead E-Pure cartridges optimized for removal of inorganic constituents. Purity is monitored using an in-line electrical resistivity meter with integral cell. Finished Inorganic reagent water is tested for conductance on a routine basis (at least annually), through the use of an external conductivity meter.

12.5.2. For organic analyses, the extractable organics laboratory uses a Barnstead E-Pure system optimized for removal of organic constituents. As organic contaminants are not measured by a resistivity meter, this is not a relied-upon method to monitor the quality of organic analyte-free water. Instead, laboratory method blanks are used, typically several per working day, to monitor the acceptability of the water for its intended use. Any analyte detected above (half of) the reporting limit is investigated. If this can be traced to the water purification system as its source, maintenance is performed on the water purification system. The volatile organics laboratory uses a Whirlpool Model WHER25 Reverse Osmosis Drinking water system to provide analyte free water.

Figure 12.3-1
Example Control Chart

Test Code: SW8081_W Analyte: 4,4´-DDD

SampType	Sample ID	Analysis Date	Batch ID	Low Limit	High Limit	% Recovery
LCSD	LCSD-65227	3/23/2012	65227	25	150	94.0
LCS	LCS-65227	3/23/2012	65227	25	150	88.6
LCS	LCS-65354	4/3/2012	65354	25	150	93.9
LCS	LCS-65320	4/3/2012	65320	25	150	91.4
LCSD	LCSD-65320	4/3/2012	65320	25	150	84.5
LCS	LCS-65743	4/26/2012	65743	25	150	92.8
LCS	LCS-65925	5/7/2012	65925	25	150	91.6
LCS	LCS-66030	5/14/2012	66030	25	150	75.4
LCS	LCS-66116	5/15/2012	66116	25	150	93.2
LCSD	LCSD-66116	5/15/2012	66116	25	150	92.7
LCS	LCS-66132	5/16/2012	66132	25	150	92.8
LCS	LCS-66631	6/12/2012	66631	25	150	94.4
LCSD	LCSD-66631	6/12/2012	66631	25	150	99.1
LCS	LCS-66758	6/18/2012	66758	25	150	90.8
LCSD	LCSD-66767	6/18/2012	66767	25	150	82.5
LCSD	LCSD-66758	6/18/2012	66758	25	150	79.9
LCS	LCS-66767	6/18/2012	66767	25	150	89.5
LCS	LCS-66817	6/19/2012	66817	25	150	92.8
LCSD	LCSD-66817	6/19/2012	66817	25	150	92.6
LCS	LCS-66801	6/20/2012	66801	25	150	99.0
LCSD	LCSD-66801	6/20/2012	66801	25	150	98.6
LCS	LCS-66899	6/28/2012	66899	25	150	80.5
LCSD	LCSD-66899	6/28/2012	66899	25	150	83.1
LCSD	LCSD-67208	7/19/2012	67208	25	150	84.8
LCS	LCS-67208	7/20/2012	67208	25	150	89.5
LCS	LCS-67206	7/20/2012	67206	25	150	80.6
LCS	LCS-68027	9/13/2012	68027	25	150	96.9
LCS	LCS-68082	9/13/2012	68082	25	150	100.3

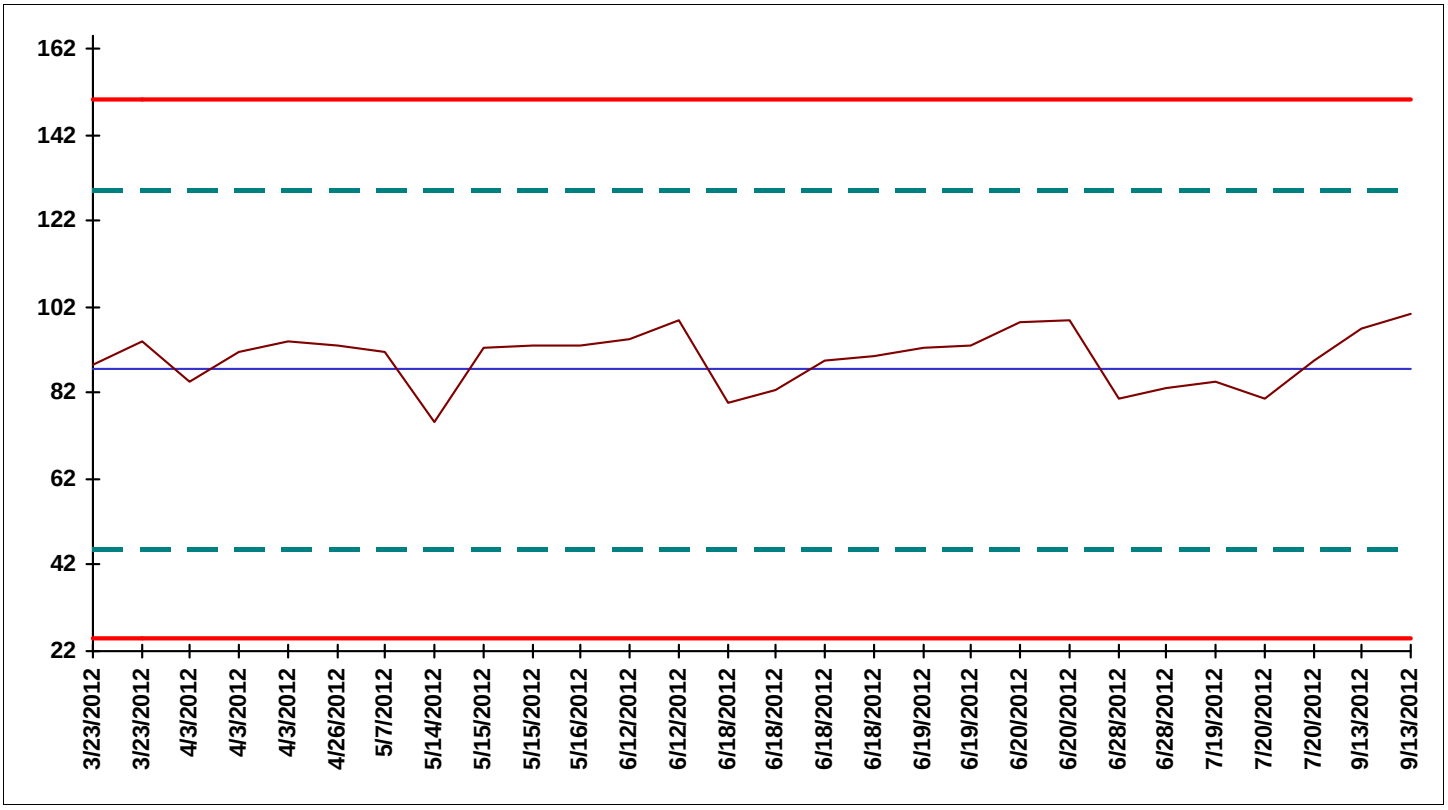
Spectrum Analytical, Inc. Featuring Hanibal Technology

REC QUALITY CONTROL CHART

Date: ***24-Sep-12***

Test Code: SW8081_W Analyte: 4,4´-DDD

SampType	Sample ID	Analysis Date	Batch ID	Low Limit	High Limit	% Recovery



13.0 QUALITY ASSURANCE SYSTEMS AUDITS, PERFORMANCE AUDITS AND FREQUENCIES, PEER REVIEW

The Spectrum Analytical, Inc. RI Division Quality Assurance staff performs routine internal audits of the laboratory. The frequency of such audits depends on the workload in house but is done annually, at a minimum. These audits entail reviewing laboratory logbooks and all appropriate operations to ensure that all laboratory systems including sample control, analytical procedures, data generation and documentation meet contractual requirements and comply with good laboratory practices.

13.1 System Audits:

The QA Director audits each individual laboratory annually in order to detect any sample flow, analytical or documentation problems and to ensure adherence to good laboratory practices as described in Spectrum Analytical, Inc. RI Division's Standard Operating Procedures and Quality Assurance Plan. A checklist used in an internal systems audit is presented in Figure 13.1-1.

Areas covered by the internal audit include logbook documentation and review, standard traceability, standard storage and expiration dates, method criteria adherence, instrument maintenance records, SOP review, and knowledge of the analysts. Often, deficiencies that have been noted during "outside" audits will also be reviewed.

Upon the completion of the internal audit, a formal audit report is presented to the laboratory supervisor who is given a specific timeframe to respond in writing to the deficiencies. The QA Department will do a follow up audit to check that at least the major deficiencies have been corrected. The follow-up audit occurs within 30-45 days from the date of the audit response.

13.2 Performance Audits:

Spectrum Analytical, Inc. RI Division participates in external Performance Test (PT) studies under the National Environmental Accreditation Program (NELAP) through the New Jersey Department of Environmental Protection (Primary Accreditation Authority). The QA department administers the Performance Evaluation Samples for Wastewater/Solid Waste (WW/SHW). Additionally, performance samples are administered for test methods not certified through the New Jersey program, such as specific state methods. PT samples are handled (i.e., managed, analyzed, and reported) in the same manner as real environmental samples utilizing the same staff, methods as used for routine analysis of that analyte, procedures, equipment, facilities, and frequency of analysis. When analyzing a PT sample, a laboratory shall employ the same calibration, laboratory quality control and acceptance criteria, sequence of analytical steps, number of replicates and other procedures as used when analyzing routine samples. PT

samples are reported electronically via the vendor's website (ERA, RTC…), and results are sent directly to all applicable state or agency certification programs.

Clients also send performance evaluation samples (PES) to Spectrum Analytical, Inc. RI Division as part of their own quality control program. Spectrum Analytical, Inc. RI Division is blind to the true values of the PES. The USEPA CLP program provides quarterly blind (QB) studies for all tests and matrices. The lab is informed of their performance after the study has been graded through an Individual Laboratory Summary Report. When results in any section are less than 90.0%, the lab is required to complete a formal corrective action report to the EPA.

Spectrum Analytical, Inc. RI Division also participates in external electronic data QA monitoring audits and data package audits through the USEPA CLP program. On request, the Spectrum Analytical, Inc. RI Division CLP Project Manager submits instrument data tapes and all applicable documentation for tape audits, including a copy of the data package. All original documentation generated during sample analyses may be requested. The results of the tape audit are sent to Spectrum Analytical, Inc. RI Division in report format in the same manner as an on-site audit (see below). A formal response is required.

Several times a year outside agencies (federal, state, or private) may schedule an audit at Spectrum Analytical, Inc. RI Division in order to check the laboratory's processes. Most often these audits begin and end with a meeting between auditors and laboratory management. Each individual laboratory is examined. The QA Director and/or Senior Management Staff are most likely to remain with the auditors at all times during the audit.

Sometime after the audit, the lab receives a formal audit report to which it must respond. The audit report is initially reviewed by the QA Director who copies and distributes the report to each laboratory supervisor. The supervisors are required to respond in writing to the findings that pertain to his or her department. The QA Officer compiles the formal response that could be tweaked several times before the auditing authority accepts the results. A specific timeframe is set by the individual agency involved.

The QA Officer then sends a memo to each supervisor to detail what needs to be done in each department within a specific timeframe. The QA Department then follows up with the labs to ensure procedures have been modified and the corrective actions are in place.

Internally, performance is monitored on a daily basis at Spectrum Analytical, Inc. RI Division through the use of surrogate and internal standards, and LCS and MS/MSD samples. Check samples from independent commercial sources are employed routinely in each of the Spectrum Analytical, Inc. RI Division laboratory departments and ensure continuing high-level performance. The QA

Director may distribute internal blind PE samples to each laboratory department as needed. These blind PE samples can also be used to show on-going analyst proficiency in lieu of 4 LCS studies.

13.3. Peer Review:

Peer review is used as a vital quality control tool within all areas of the laboratory, and at all levels. Peer review allows defects in the acquisition, evaluation and reporting of sample data to be identified before moving on to the next step in the process of preparing and analyzing samples. Several steps of peer review are included at Spectrum Analytical, Inc. RI Division to prevent and catch mistakes, whether caused by human error or a system malfunction. As soon as samples enter the laboratory they are logged into the LIMS system and given unique sample identifiers that correspond to the client's IDs listed on the chain of custody. The individual jars or bottles are labeled and the technician employs a peer review of this labeling process. A project manager or peer technician visually inspects each jar or bottle for proper identification and matching lab/client IDs. Once the samples are sent into the labs for test preparation, they again undergo peer review as they are set up for extraction, digestion or distillation... This time the samples are inspected to confirm the samples at the bench match the identifications written into the lab preparation logbooks. Once the concentrated extract, digestate or distillate is ready for analysis and set up on the analytical instrument, an analyst will perform another peer review of the autosampler set up to avoid any misplacements of sample vials. In some lab areas this review may occur after instrument analysis, to verify all sample data were acquired electronically. Every analytical instrument sequence (GC/ECD, GC/FID, GC/MS, ICP/MS, ICP/AES, CVAA, FIA, IC) undergoes a technical peer review by a qualified analyst to verify positive and false positive results as well as manual integrations. Data reports are also reviewed at length according to the 5 level review processes described in Section 11 of the QAP as well as in SOP No. 110.0028 Data Validation/Self Inspection Procedures. At each point in the process, the peer review is documented.

Figure 13.1-1
QA Systems Audit Checklist

Quality Assurance Department
Spectrum Analytical, Inc. RI Division
.

Quality Review of Laboratory Department

Auditor:
Date:

Purpose

The Quality Review is a necessary tool to assess a department's quality and service functions. Each department will undergo a review of their process and procedures to evaluate their needs and areas of possible improvement. Each department will be tracked for quality, safety, compliance, reoccurring errors and process improvement.

Process

Each department will be broken down into several categories or areas of review. Each category will be reviewed and assessed for compliance. The categories will include at a minimum:

Personnel Training and Knowledge
Equipment
SOP Updates and Review
Logbook Review and Control
Chemicals/Standard Storage and Preparation
Sample Procedures and Method Compliance
QA/QC Procedures
Corrective Actions in process

Each category will be reviewed and a listing of any deficiency or findings will be documented for response and correction. The department Supervisor (s) will be required to respond to each deficiency or finding within 30 days of receipt of this report. All deficiencies or findings must have its correction(s) documented. For example, logbook deficiencies will require a photocopy of the correction(s). All other responses will require a written response or adequate explanation. Deficiencies will be tracked for reoccurrence. All documentation should be forward to the QA department for evaluation. A follow up audit may be scheduled.

Findings:

Personnel Training and Knowledge

.

Equipment

SOP Updates and Review

Logbook Review and Control

Chemicals/Standard Storage and Preparation

Sample Procedures and Method Compliance

QA/QC Procedures

Corrective Actions in process

Items marked with an* asterisk will require a written response by the lab supervisor or his designee to the QA Dept. This response must be submitted to the QA Department by *mm/dd/yyyy*. The response can be entered directly into this document in a different font color. Please note date that the CA was completed.

Auditor _____ Date _____

14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a routine practice at Spectrum Analytical, Inc. RI Division for all instrumentation. Scheduled preventive maintenance minimizes instrument downtime and subsequent interruption of analysis.

Only those equipment items meeting or exceeding applicable performance requirements are used for data collection. This includes items such as laboratory balances as well as major analytical instruments such as ICPs, ICP/MS, GCs and GC/MSs. All major instrumentation and equipment, as well as backup alternatives, are listed in Appendix A. Spectrum Analytical, Inc. RI Division SOP No. 110.0040, Instrument Maintenance, describes routine maintenance in detail. Individual analytical standard operating procedures describe maintenance as well (See Figure 11.7-1 for SOP listing).
When new software is purchased or developed, it is loaded onto one workstation with copies of data that have been previously processed using older software, and known to be correct. The data is then reprocessed using the new software and then the new results are compared to the original results for defects. If the software was purchased and found to contain a defect, the vendor is contacted and a solution and/or patch are requested. If the software was developed in-house, the problems are identified and corrected. This process is applicable to all software including enhancements made to customize the LIMS and network servers.

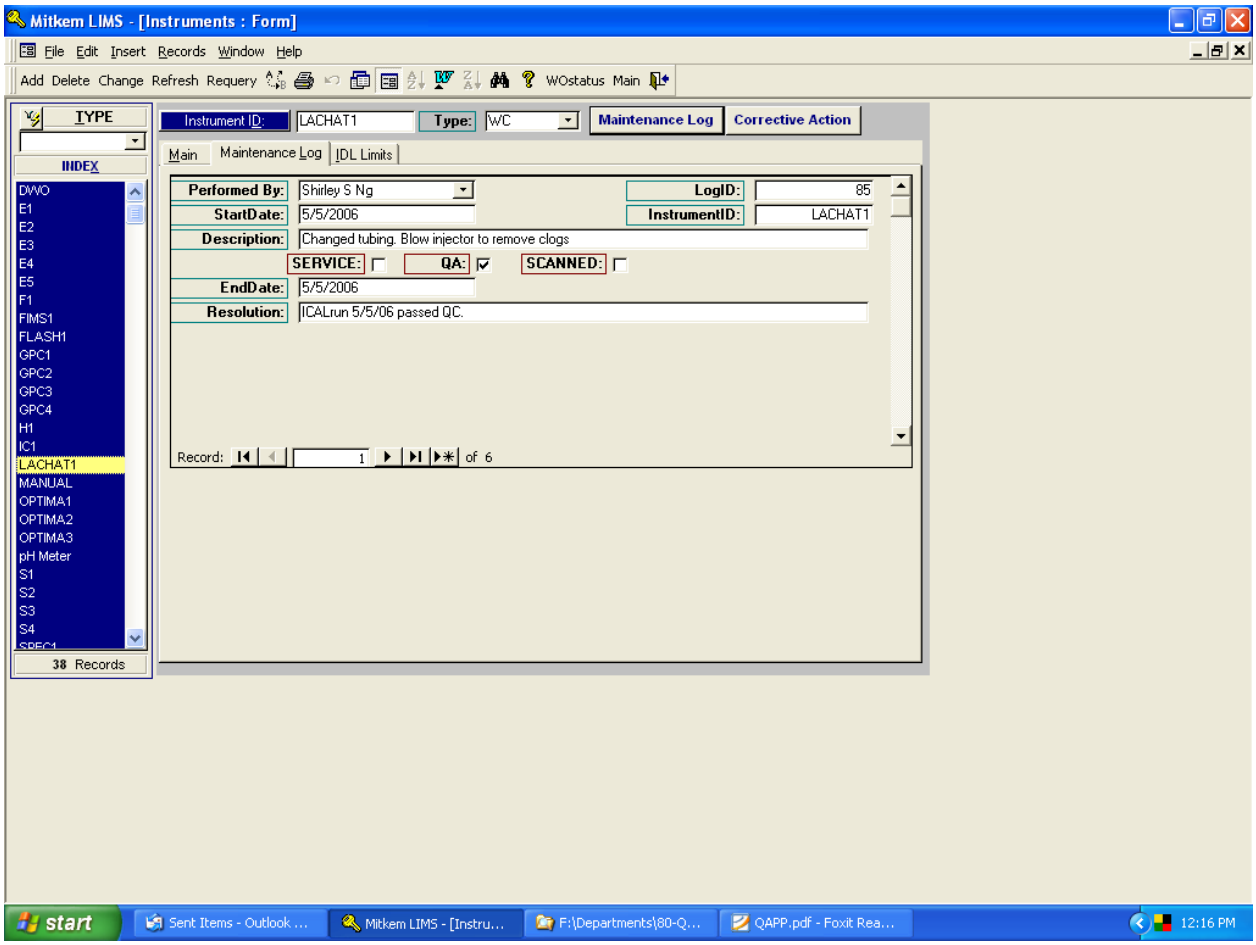
Spectrum Analytical, Inc. RI Division's laboratory personnel are familiar with the routine and non-routine maintenance requirements of the instruments they operate. This familiarity is based on education, hands-on experience and manufacturer's training courses. As needed, major equipment may under-go extensive maintenance or service by a contracted technician.

Instrument maintenance logs are kept for each instrument in the LIMS (figure 14-1). All employees have password protected access to the LIMS. The person performing the maintenance is required to provide the following information in the online log:

- Equipment identifier
- The inspection, maintenance, calibration or corrective action(s) performed.
- The trigger(s) for the maintenance action(s)
- The identity of the person(s) performing the maintenance
- The date on which the work was performed
- The need for a service call
- The condition of the equipment upon completion of the work (may include resolution of problems, date and type of ICAL run or other method of determining that the system is in good working order), and
- The presence of any scanned paperwork associated to the maintenance

Spectrum Analytical, Inc. RI Division maintains an inventory of replacement parts required for preventive maintenance and spare parts that often need replacement, such as filaments for GC/MS systems and the more mundane electrical fuses and GC column ferrules. To control cost, the appropriate supervisor shall decide the types and numbers of spare parts kept on hand for each equipment item.

Figure 14-1



Example of Instrument Maintenance Log

15.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, COMPLETENESS, METHODS DETECTION LIMIT AND LINEAR DYNAMIC RANGE

These mathematical equations represent the means of calculating analytical figures of merit on a routine basis at Spectrum Analytical, Inc. RI Division. However, they may be supplanted with other calculations if requested by the client. Precision, accuracy and completeness are also discussed in Section 6.

15.1 Precision:

Precision is frequently determined by the comparison of replicates, where replicates result from an original sample that has been split for identical analyses. Standard deviations, *s*, of a sample are commonly used in estimating precision.

Sample standard deviation, *s*:

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2}$$

where a quantity, x_i (e.g. a concentration), is measured *n* times with a mean, $\bar{x}$.

The relative standard deviation, *RSD* (or sample coefficient of variation, *CV*), which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates (although it may be applied in the case of n = 2).

$$\%RSD = 100\ (s\ /\ \bar{x}\ )$$

or

$$CV = 100\ (s\ /\ \bar{x}\ )$$

In which: *RSD* = relative standard deviation, or
CV = coefficient of variation
s = standard deviation
$\bar{x}$ = mean

For duplicates (samples that result when an original sample have been split into two for identical analyses), the relative percent difference (*RPD*) between the two samples may be used to estimate precision.

$$RPD = \frac{2(D_1 - D_2)}{(D_1 + D_2)} \times 100\%$$

In which: D_1 = first sample value
D_2 = second sample value (duplicate)

15.2 Accuracy:

The determination of accuracy of a measurement requires knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of bias as follows:

$$Bias = X - T$$

$$\%Bias = 100\frac{(X - T)}{T}$$

In which: X = average observed value of measurement
T = "true" value

Accuracy also may be calculated in terms of the recoveries of analytes in spiked samples:

$$\%\,\mathrm{Recovery}(\%R) = 100 \times \frac{(SSR - SR)}{SA}$$

where: SSR = spikes sample result
SR = sample result
SA = spike added

15.3 Completeness:

Determine whether a database is complete or incomplete may be quite difficult. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. Less obvious is whether the data are sufficient to achieve the goals of the project. All data are reviewed in terms of goals in order to determine if the data set is sufficient.

Where possible, the percent completeness for each set of samples is calculated as follows:

$$\%\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100$$

15.4 Method Detection Limit:

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is not zero. It is computed as follows from data obtained by repeatedly determining an analyte in a given sample matrix:

1. Analyze at least seven samples of a homogeneous matrix spike that contains the analyte(s) of interest at concentrations of three to five times the expected MDL. The entire sample preparation and analysis protocol must be applied in each analysis; simply preparing one sample and repeating a measurement three or more times on the sample in not acceptable.

2. Upload the acceptable data into LIMS.

3. The LIMS will compute the standard deviation of the results for each analyte using the following equation:

$$MDL = t_{(n-1,\ \alpha=0.99)}\ (s)$$

Where *t* is the one-sided student's *t* value appropriate for the number of samples analyzed, *n*; α is the statistical confidence level; and *s* is the standard deviation.

The one-sided *t*-values are presented below:

Number of samples	*t*-value
7	3.14
8	2.996
9	2.90
10	2.82

4. The MDL is then checked against 40CFR136 requirements by the QA Department. If the MDL is acceptable then it is uploaded into the LIMS by either the QA Department or LIMS Administrator.

5. Immediately following the determination of the MDL, MDL check samples are analyzed at a concentration approximately equal to 2-3 x the new MDL for SW846 tests. The analyte of interest must be detected at this concentration, or the raising the MDL may be required. Once the MDL check is acceptable, the detection limit (DL) has been established.

6. An elevated MDL can be uploaded if necessary into the LIMS as long as documentation is available to show that the applicable method can produce an MDL at least that low. This can commonly occur for ICP

analysis in which extremely low MDLs can cause method compliance issues. When appropriate, the MDL study may be prepared and analyzed over several days to increase the variability of the preparation and/or analytical steps.

7. More detail on MDLs can be found in SOP 80.0005 Method Detection Limit Determination.

15.5 Linear Dynamic Range:

The linear dynamic range is the concentration range over which the instrument response is linear. It is determined by analyzing a series of standard solutions that extends beyond the non-linear calibration region at both the low and high extremes, and selecting that range of standards which demonstrates a linear relationship between instrument response and concentration.

For ICP analysis, the linear dynamic range is determined by analyzing each metal at 3 different concentrations. The concentration which produces results within a 10% error is determined to be the linear dynamic range. This procedure must be performed per individual method requirements.

ILM5.4 requires the analysis of the linear dynamic range be determined quarterly, with a 5 % error.

16.0 CORRECTIVE ACTION

An essential element of the QA Program, Corrective Action provides systematic, active measures taken in the resolution of problems and the restoration of analytical systems to their proper functioning.

Corrective actions for laboratory problems are described in Spectrum Analytical, Inc. RI Division's laboratory standard operating procedures (SOP). Personal experience often is most valuable in alerting the bench scientist to questionable results or the malfunctioning of equipment. Specific QC procedures are designed to help the analyst determine the need for corrective actions (see Section 11, Data Reduction, Validation and Reporting). Corrective actions taken by scientists in the laboratory help avoid the collection of poor quality data. The lab's corrective action program divides these issues into routine and non-routine corrective actions as described below.

Routine Corrective Action – A routine corrective action is taken when the out-of-control event encountered is one that is detected at the appropriate level in the QA process. Routine corrective actions are defined in the analytical SOP with specific steps to be taken as corrective action (i.e., low surrogate recovery, continuing calibration verifications, project specific protocols that do not meet acceptance criteria, etc.) Routine corrective actions must be documented as described in the analytical SOP, but do not require further documentation in the corrective action logbook. Examples of routine corrective action situations: surrogate/surrogates out, LCS out, CCV out, ICV out, IS area/areas out, typographical errors, random blank contamination, or false positive hit/spectral ID match corrected during data review.

Non-Routine Corrective Action – A non-routine corrective action is taken when the out-of-control event encountered is not typical for the method. For example, QC failures that passes through the final review to the client, procedural errors – not following the SOP, or a situation not being detected by normal QA procedures that could adversely impact the accuracy, precision, etc. of a result. Non-routine corrective actions must be documented in the Corrective Action Request (CAR) system, located within the LIMS. The analyst, using his/her own judgement, may deem any corrective action situation non-routine and formally document it in a CAR. When in doubt about a corrective action, the analysts are instructed to err on the side of formal CAR documentation. Examples of non-routine corrective action situations include: bad standard, expired standard mix being used, incorrect equation, "client-detected" problems, not following SOP protocols, using bad or contaminated lot of chemical/reagent/solvent, deciding to release data not conforming to SOP requirements, compound retention time outside of range, or improper library spectrum that leads to re-occurring mis-identification of compounds.

The essential steps in Spectrum Analytical, Inc. RI Division's corrective action system are:

1. Identify and define the problem.

2. Assign responsibility for investigating the problem. Usually this individual is the department supervisor.

3. Investigate and determine the root cause of the problem.

4. Determine a corrective action to eliminate the problem and prevent recurrence. Any changes that result from the corrective action investigation must be documented.

5. Assign and accept responsibility for implementing the corrective action.

6. Establish effectiveness of the corrective action and implement it.

7. Verify that the corrective action has eliminated the problem.

8. Both the laboratory and the QA Department need to monitor the corrective action to ensure it is effective.

9. Any corrective actions that cast doubt on the laboratory's compliance with its own policies and procedures may require an internal audit by the QA Department.

This scheme is generally accomplished through the use of Corrective Action Report Forms available to each of the laboratory areas within the LIMS system. Use of this report notifies the QA Department of a potential problem as described in SOP No. 80.0007. The QA Director initiates the corrective action by relating the problem to the appropriate laboratory managers and/or project managers who then investigate or assign responsibility for investigating the problem and determine its cause. Once determined, the QA Director will approve appropriate corrective action. Its implementation is later verified through an internal laboratory audit. Once the QA Director feels the system has returned to control, s/he will finalize the CAR using a password protected QA step.

Information contained on corrective action reports is kept confidential within Spectrum Analytical, Inc. RI Division and is generally limited to the individuals involved. Severe problems and difficulties may warrant special reports to the President of Spectrum Analytical Inc., who will ensure that the appropriate corrective actions are taken.

Nonconformance:

Any breech of standard protocols is a nonconformance item that is documented on the Corrective Action Request Form and management informed immediately. The following are nonconformance items:

1. Sample holding time exceeded.

2. Hoods, Class “1” weights, NIST Thermometers, balances, automatic pipettes, being used but not certified.

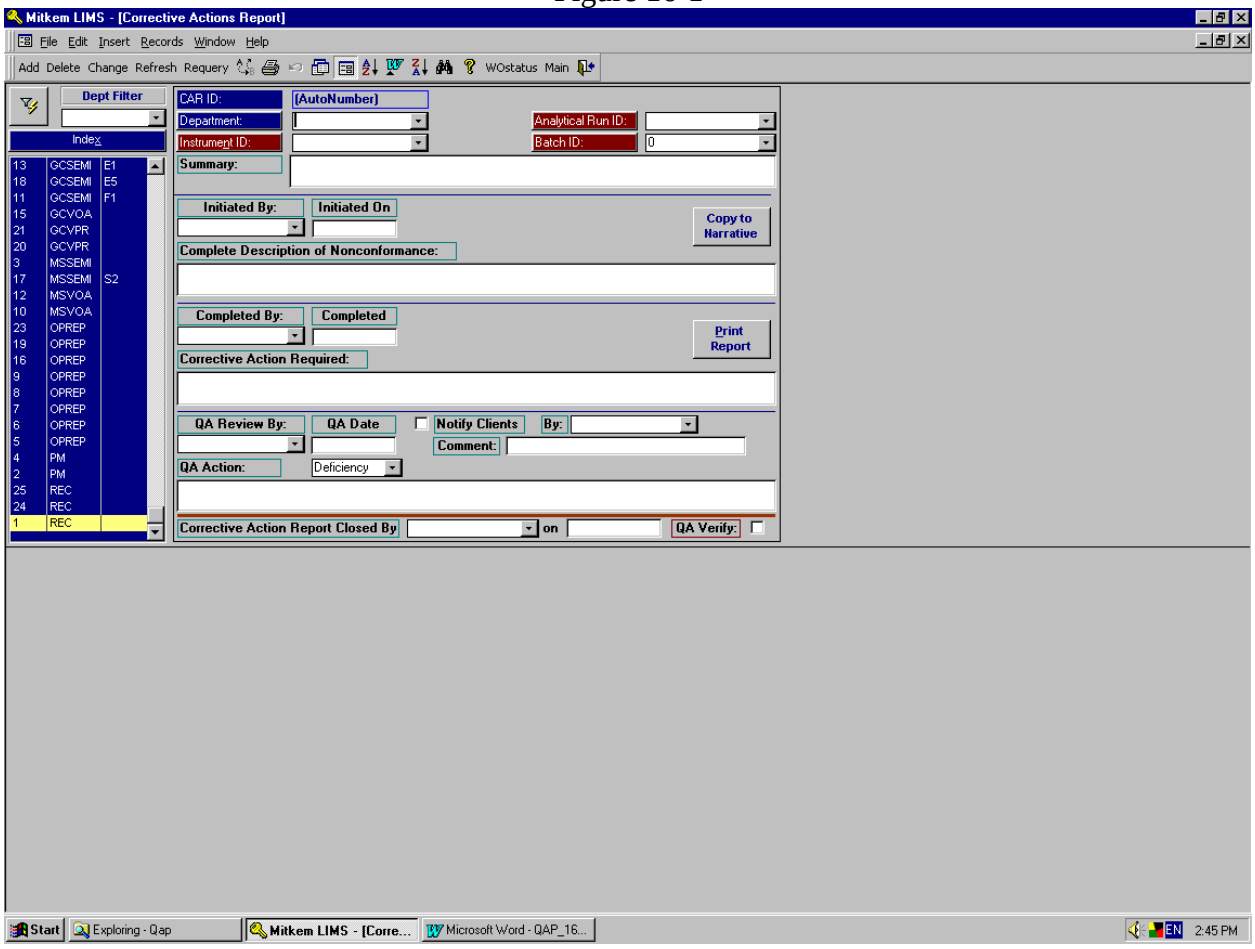
3. Expired standards being used.

4. Manual integration being misrepresented.

16.1 Client Complaints:

Spectrum Analytical, Inc. RI Division ensures client complaints are dealt with quickly and completely. The policies are stated in the laboratory Client Complaint Standard Operating procedure (SOP No. 80.0002).

Figure 16-1



Quality Assurance Corrective Action Request Form

17.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The Spectrum Analytical, Inc. RI Division Quality Assurance Director submits a QA report annually to upper management. The report should be completed and submitted no later than the 15th of July in any calendar year.

The report contains detailed laboratory information and QA activities during the previous twelve months. Items to include are the status of internal and external audits, client complaints, quality control activities, resources and staffing. See the following pages for the report format.

Management will review the QA report and respond to outstanding issues. Management will add a review of the suitability of policies and procedures, and any other relevant issues**.** The response report is due within 30 days of the QA Report receipt.

A copy of the report is kept on file in the QA department.

In case of a severe problem or difficulty, a special report is prepared by the QA Director and submitted immediately to management.

Figure 17-1

SPECTRUM ANALYTICAL, INC. RI DIVISION
Annual Quality Assurance Report to Management

1. Status of Internal Audits.

2. Status of External Audits.

3. Identification of Quality Control issues in the laboratory.

4. Discussion of corrective action issues.

5. Proficiency Testing.

6. Changes in volume and type of work undertaken.

7. Client Feedback.

8. Reports from management and supervisory personnel.

18.0 SAFETY

Spectrum Analytical, Inc. RI Division maintains safety through a program managed by the Safety Officer and the Safety Committee. Responsibilities include many activities needed to comply with the Right-to-Know Laws.

- Training seminars with information on OSHA safety instruction for new employees.
- Introductory training to include location of fire extinguishers, first aid supplies, etc.
- Health and Safety manual review when hired.
- Annual Health and Safety Manual review and revision as needed.
- Monthly Safety Committee meetings.
- Centralized MSDS information.
- Maps with safety equipment and all exits noted.
- Posted safety rules.

If a chemical spill occurs, proper actions are described in Spectrum Analytical, Inc. RI Division's Contingency Plan. Additionally, the local fire department (North Kingstown) and hospital (Kent County) also have a copy in case a need arises. Each new hire is required to read the Contingency Plan and sign off on this. An annual meeting is held as a refresher for all employees. A copy of the Contingency Plan is located on the company Intranet and is available to all personnel.

Emergency equipment, such as spill control kits, fire extinguishers and fire blankets are located throughout the laboratory areas. The Contingency Plan has instructions for evacuation, notification of emergency authorities and regulatory personnel in the event of a chemical accident.

19.0 WASTE MANAGEMENT

19.1 Pollution Prevention

The waste management option of choice is to prevent pollution by minimizing the amount or types of chemical wastes that are generated. Spectrum Analytical, Inc. RI Division's ability to minimize waste generation is limited by the chemical analysis techniques that are required by the EPA or other authors of test methods. As new test methods are utilized in the laboratory, the type and volume of chemical waste generated by the new test is considered. Analysts and Supervisors are encouraged to look for ways to reduce the amount of chemical waste, or the type of chemical waste generated during the testing process; HOWEVER, no method is allowed to be modified without discussion among the Laboratory and/or Technical Director, QA Director and other management personnel to determine the affect of the change on the resulting data.

19.2. Waste Management

Spectrum Analytical, Inc. RI Division has identified and routinely disposes of chemical wastes in several hazardous waste streams. In general these are acids, caustics, solvent wastes and various laboratory waste solids. No laboratory chemical waste is disposed in the trash or dumped down the drain. All remaining sample volume following testing, and after contract-required disposal date has past, are disposed in one of these waste streams. These wastes are fully described in Spectrum Analytical Inc., RI Division's Contingency/Waste Management Plan and in the lab's Profile Log. New England Disposal Technologies is Spectrum Analytical, Inc. RI Division's waste hauler. Other hazardous wastes are identified and properly disposed according to these documents.

Continued compliance is monitored monthly by an outside consultant to ensure all RI DEM regulations are met. Key personnel attend an annual RCRA Facility Training, which focuses on the requirements for hazardous waste disposal and its proper documentation.

20.0 DEFINITIONS, ACRONYMS, ABBREVIATIONS:

ACCURACY: The closeness of agreement between an observed value and an accepted reference value.

ALIQUOT: A measured portion of a field sample, standard, or solution taken for sample preparation and/or analysis.

ANALYTICAL SERVICES BRANCH (ASB): The division of United States Environmental Protection Agency's (USEPA) Office of Superfund Remediation and Technology Innovation (OSRTI) responsible for the overall management of the Contract Laboratory Program (CLP).

ASTM: American Society for Testing and Materials, a developer and provider of voluntary consensus standards.

BATCH: A group of samples of the same matrix that are processed as a unit at the same time in the same location using the same method.. Unless defined differently by a specific analytical method (such as Oil & Grease by Method 1664), the maximum batch size is 20 samples.

BIAS: The deviation due to analytical or matrix effects of the measured value from a known spiked amount.

BLANK: A "clean" matrix analysis. Such as: Equipment Blank, Method Blank, and Trip Blank.

BREAKDOWN: A measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

CAS: Chemical Abstracts Service, a registry where chemicals are assigned identification numbers.

CCB: Continuing Calibration Blank

CCV: Continuing Calibration Verification standard.

CLP: Contract Laboratory Program. A contract used by EPA to purchase analytical services. Also refers to the test protocols described in that contract. The CLP analyses can be used for EPA or for other clients. CLP-format data reports are arranged as described in the EPA CLP contract, including specified data report pages and all raw data.

CONTROL SAMPLE: A QC sample introduced into a process to monitor the performance of the system.

DL: Dilution, not used when the initial analysis is performed at dilution, but is used for a secondary dilution.

DoD: Department of Defense.

DUPLICATE: See Matrix Duplicate, Field Duplicate, and Matrix Spike Duplicate.

EQUIPMENT BLANK: A sample of analyte-free water that has been used during sample collection to measure any contamination introduced during sample collection.

ICB: Initial Calibration Blank

ICV: Initial Calibration Verification standard

IDL: Instrument Detection Limit. Statistical value similar to MDL, but with analyses performed on standards that have not been through the sample preparation process.

FIELD DUPLICATES: Independent samples that are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

HT Holding Time. The maximum times that samples may be held prior to analysis and still be considered valid or not compromised (40CFR Part 136). DoD also clarifies the HT to mean the time elapsed from the time of sampling to the time of extraction or analysis , or from extraction to analysis…

LAB CONTROL SAMPLE (LCS): A blank spiked with compound(s) representative of the target analytes. This is used to document laboratory performance in a "clean" matrix.

LOD: Limit of Detection. The smallest amount of concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%), per DoD.

LOQ: Limit of Quantitation (LOQ). The lowest concentration that produces a quantitative result within specified limits of precision and bias. The LOQ

is typically set at or above the concentration of the lowest initial calibration standard.

MATRIX: The component or substrate (e.g., water, soil, air, and oil) which contains the analyte of interest.

MATRIX DUP (DUP): A sample split by the laboratory that is used to document the precision of a method in a given sample matrix.

MATRIX SPIKE (MS): An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

MATRIX SPIKE DUP (MSD): Laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

MCL: Maximum Contaminant Level (MCL) is the highest concentration of a contaminant that is allowed in drinking water.

METHOD BLANK(MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

METHOD DETECTION LIMIT (MDL): The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. For operational purposes, when it is necessary to determine the MDL in the matrix, the MDL should be determined by multiplying the appropriate one-sided 99% t-statistic by the standard deviation obtained from a minimum of seven analyses of a matrix spike containing the analyte of interest at a concentration estimated to be three to five times the MDL, where the t-statistic is obtained from standard references.

MSA: Method of Standard Additions

ND: Not Detected. Used in conjunction with the reporting limit.

ORGANIC-FREE REAGENT WATER: For volatiles, all references to water in the methods refer to water in which an interferent is not observed at the reporting limit of the compounds of interest. Organic-free reagent water

can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water. For semivolatiles and nonvolatiles, all references to water in the methods refer to water in which an interferent is not observed at the reporting limit of the compounds of interest.

PPB: Parts Per Billion, ug/L, ug/Kg

PPM: Parts Per Million, mg/L, mg/Kg

PQL: Practical Quantitation Limit. Equivalent to Reporting Limit.

PRECISION: The agreement among a set of replicate analyses.

PS: Post Spike. Spike added at the analysis level (as opposed to at the beginning of sample preparation) to determine interferences.

REPORTING LIMIT (RL): The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The RL is generally 5 to 10 times the MDL. However, it may be nominally chosen other than these guidelines to simplify data reporting. For many analytes the RL concentration is selected as the lowest non-zero standard in the calibration curve. Sample RLs are matrix-dependent, and are adjusted by the amount of sample analyzed, dilution, and percent moisture. Also see LOQ.

RE: Reextraction or Reanalysis

RPD: Relative Percent Difference, used to determine precision.

RRF: Relative Response Factor. Used for quantification with the internal standard procedure.

RT: Retention Time for a chromatographic peak, as calculated from the time of injection.

SAMPLE: A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLE DELIVERY GROUP (SDG): A unit within a sample Case that is used to identify a group of samples for delivery.

SERIAL DILUTION (SD): A five-fold dilution of a sample. When corrected by the dilution factor, the diluted sample must agree with the original undiluted

sample within specified limits. Serial dilution may reflect the influence of interferents.

SAMPLE MANAGEMENT OFFICE (SMO) - A Contractor-operated facility operated under the SMO contract, awarded and administered by USEPA.

SOP: Standard Operating Procedure.

STANDARD ADDITION: The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate method. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

SURROGATE: An organic compound that is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

TRIP BLANK: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

From EPA SW-846, Revision 4, 40CFR Part 136, DoD QSM and other sources.

SPECTRUM ANALYTICAL, INC. RI DIVISION

MAJOR INSTRUMENTATION and EQUIPMENT LIST

APPENDIX A

Laboratory Information System Equipment

1. **Data Collection**:

1.1. Seventeen- Hewlett Packard (HP) chem station software for collecting GC and GC/MS data (below) and one Perkin Elmer (PE) Total Chrom for collecting data from the GC-TCD/SCD.

- 5 GC-ECD (GCSEMI)
- 1 GC-FID (GCSEMI)
- 6 GC-MS (MSSEMI)
- 5 GC-MS (MSVOA)
- 1 GC-Hall/PID (GCVOA)
- 1 GC-FID/NPD (GCVOA)

1.2. Hardware varies but is x86 compatible

1.3. OS is Windows, Various Versions (9x, NT, 2000,Xp)

2. **Data Storage**:

2.1. Dell Poweredge servers (Windows 2003 server)

2.1.1. Bernoulli (primary file server, non-organic instrument data)
- Dual core Xeon processor
- 4 GB RAM
- 1 TB storage
- Symantec Backup Exec 12.5
- Tape drive Tandberg Data LTO-5 (1500-3000 GB)

2.1.2. Avogadro (organic instrument data)
- Dual P IV Xeon processors
- 2 GB RAM
- 105 GB storage
- Tape drive Tandberg LTO-2 (200-400 GB)

2.1.3. Planck (database server)
- Dual P IV Xeon processors
- 2 GB RAM
- 450 GB storage
- Tape drive Seagate LTO-1 (100-200 GB) not currently used

2.2. Tapes are for daily backup, long term archiving and data restoration

3. **Compound Identification**:

3.1. Fourteen - Target 4.14 chromatographic software

3.2. Hardware is Intel based for Target 4.14

3.3. OS is Windows Xp

4. **Forms Generation**:

4.1. In-house forms generation LIMS modules for SW-846, ILM and ISM metals

4.2. In-house forms generation LIMS modules for SW-846, OLC, OLM/ASP and SOM organics

4.3. Hardware varies but is x86 compatible

4.4. OS is Windows, Various Versions (2000 and Xp)

Spectrum RI
Equipment List **Department: Inorganics : Metals& Wet Chemistry**

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID	Location
ICP/OES	Perkin Elmer	077N3102302	Nov-03	Nov-03	New	Optima3	Metals
ICP/AES	Perkin Elmer	069N8060801	Nov-98	Nov-98	New	Optima2	Metals
ICP/MS	ThermoScientific	SN01407C	Oct-08	Dec-09	New	X1	Metals
Mercury Analyzer	Perkin Elmer	1131	Mar-00	Mar-00	Used	FIMS1	Metals
Mercury Analyzer	Perkin Elmer	101S7071002	Feb-11	Feb-11	new	FIMS2	Metals
GPR Centrifuge	Beckman Instruments	7M149	Apr-02	Apr-02	Used	Centrifuge	WC
Conductivity Meter	WTW Inolab Cond Level 1	3370010	Apr-02	May-02	New	COND-1	WC
Total Organic Carbon Analyzer	Tekmar/Dohrmann	US03035002	Apr-03	Apr-03	Used	TOC1	WC
Flow Injection Analyzer	Lachat Instruments	A83000-1020	Apr-96	Apr-96	New	Lachat1	WC
Ion Chromatograph	Dionex	95030498E980802	May-03	May-03	New	IC1	WC
Spectrophotometer	Spectronic Instruments	3SGD332010	Apr-02	Apr-02	New	SPEC2	WC
Spectrophotometer	Milton Roy Company	3310004028	Mar-06	Mar-06	New	SPEC3	WC
Pensky Marten	Koehler 16200	5539	June-95	June-95	New	FLASH1	WC
Turbidity Meter	VWR® Model 800	Tur800 2326	April-12	Feb-13	Used	Turb1	WC

COD Reactor	Hach Company	990900019429	Nov-03	Nov-03	New	COD1	WC
COD Reactor	Hach Company	950200012193	Apr-02	Apr-02	New	COD2	WC
Deionized Water Generator	Barnstead E-Pure D4641	1090001208384	Jun-95	Jun-95	New	DI2	WC
pH meter	Oakton Instruments	875001	Jun-12	Jun-12	new	WC-03	WC

Spectrum RI
Balance List

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID
TOP-LOADING Balance	OHAUS	1121230069	2000	2000	New	TL10
Analytical Balance	Denver A-250	0070742	2010	2010	Used	AB-3
TOP-LOADING Balance	OHAUS Voyager	F2921120391055	2001	2001	New	TL9
TOP-LOADING Balance	Denver	0079896	2000	2000	New	TL1
TOP-LOADING Balance	OHAUS Precision Std.	C22427176	2002	2007	New	TL6
TOP-LOADING Balance	OHAUS Navigator	1121122373	2002	2002	New	TL11
TOP-LOADING Balance	OHAUS	CD8910	2000	2000	New	TL4
TOP-LOADING Balance	OHAUS Navigator	1122173423	2003	2003	New	TL12
TOP-LOADING Balance	OHAUS Scout Pro	7126212230	2007	2007	New	TL13

Spectrum RI
Equipment List **Department: Organic Prep**

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition new/used	Equipment ID
TurboVap II	Caliper	TV0845N14899	Jan-09	Jan-09	New	TV-4
TurboVap II	Caliper	TV0902N15012	Jan-09	Jan-09	New	TV-3
TurboVap II	Caliper	4364	Mar-08	Mar-08	Used	TV-2
TurboVap II	Caliper	Unable to view	Mar-08	Mar-08	Used	TV-1
Shaker	Glas-Col	412383	Mar-08	Mar-08	New	N/A
Water Bath	Precision Scientific	9508-005	Dec-95	Jan-96	Used	N/A
Nitrogen Concentrator Bath	Organomations	16526	Jun-97	Jun-97	New	NZ1
Deionized Water Generator	Barnstead E-Pure D4641	582941018789	Jun-95	Jun-95	New	DI1
Pressurized Fluid Extractor	Dionex	98070129	Jun-00	Jun-00	New	PFE1
Gel Permeation Chromatograph	J2/AccuPrep	P26D031	Jun-05	Jul-05	New	GPC3
Gel Permeation Chromatograph	J2/AccuPrep	06D-1196-4.1	Jul-07	Aug-06	New	GPC4
Misonex Ultrasonic Disruptor	Sonic Dismembrator Fisher Model 550	Unable to view			New	OPH1
Misonex Ultrasonic Disruptor	Sonic Dismembrator Fisher Model 550	Unable to view			New	OPH2
Misonex Ultrasonic Disruptor	Sonic Dismembrator Fisher Model 500	Unable to view			New	OPH3

Misonex Ultrasonic Disruptor	Sonic Dismembrator Fisher Model 500	Unable to view			New	OPH4
Ultrasonic Cleaner FS30H	Fisher Scientific	RTB030721702	Apr-07	Apr-07	New	N/A
Centrifuge Centra CL-2	International Equipment Company	42606943			Used	N/A

Spectrum RI
Equipment List **Department: GC-Semivolatiles**

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID	Location
GC/ECD	Hewelett Packard	3336A59890	Oct-94	Oct-94	New	E2	GC-SVOA
GC/ECD	Hewelett Packard	US00032017				E4	GC-SVOA
GC/ECD	Hewelett Packard	US00037060				E5	GC-SVOA
GC/ECD	Hewelett Packard	US00029100	13-Feb	13-Feb	used	E6	GC-SVOA
GC/FID	Hewelett Packard	US00001898				F1	MS-SVOA

Spectrum RI
Equipment List **Department: Receiving**

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID	Location
Dry Weight Oven	Thello	600011006			used	DWO	REC
Walk in Cooler		Not Applicable			new	R1	REC
Gyrotary Shaker table	New Brunswick Sci. Co.	unable to read			used	n/a	REC
pH meter	Oakton Instruments	1446253	Dec-08	Dec-08	new	WC-02	REC
Kiln model TNF24-3	Paragon Touch n Fire	324341				n/a	WC
Stoppering tray dryer	FTS Systems Dura-Stop M	TD-12-90-133				n/a	WC
Freeze Dryer	FTS Systems Dura-Dry MP	unable to see				n/a	WC
Dessicator	Sanplatec Corp	none	June-06	June-06	New	DryKeeper	REC

Spectrum RI
Equipment List **Department: SVOA**

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID	Location
GC/MS	Hewelett Packard	US00011367 / US72821130	Nov-99	Nov-99	Used	S3	MS-SVOA
GC/MS	Hewelett Packard	CN10315002/ VS30945365	May-03	May-03	New	S4	MS-SVOA
GC/MS/FID	Hewelett Packard	CN107223014 / US73317299	Jan-08	Jan-08	New	S5	MS-SVOA
GC/MS	Hewelett Packard	CN10261100	Nov-10	Nov-10	Used	S6	MS-SVOA

Spectrum RI
Equipment List **Department: VOA**

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID	Location
GC/MS	Hewelett Packard	3336A55963				V1	VOA
Auto sampler	OI	13193				V1	VOA
Concentrator	OI	J651460769				V1	VOA
GC/MS	Hewelett Packard	3336A58222				V2	VOA
Auto sampler	OI	13091				V2	VOA
Concentrator	OI	H340460074				V2	VOA
GC/FID/PID	Hewelett Packard	2843A21041				V4	VOA
Auto sampler	Tekmar/Dohrmann	90312004				V4	VOA
Concentrator	Tekmar/Dohrmann	88341012				V4	VOA

Spectrum RI
Equipment List **Department : VOA**

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID	Location
GC/MS	Hewelett Packard	US00007055				V5	VOA
Auto sampler	OI	13462				V5	VOA
Concentrator	OI	J651460769				V5	VOA
GC/MS	Hewelett Packard	US00031343				V6	VOA
Auto sampler	OI	B03745A407				V6	VOA
Concentrator	OI	J651460769				V6	VOA
GC	Hewelett Packard	3140A37463				V7	VOA
Auto sampler	Tekmar/Dohrmann	US01170015				V7	VOA
GC/MS	Hewelett Packard	CN10411124	Oct-10	Nov-10	NEW	V10	VOA
Auto sampler	Tekmar/Dohrmann	US01157003	Oct-10	Nov-10	USED	V10	VOA
Concentrator	Tekmar/Dohrmann	US02021003	Oct-10	Nov-10	NEW	V10	VOA

Weight Sets

Laboratory weights for daily calibration use:

1. WT1-Organic Prep Weight Set
2. WT2-Organic Prep 100g
3. WT3-Organic Prep 300g
4. WT4-Organic Prep 1kg
5. WT5-Inorganics Weight Set
6. WT6-VOA Weight Set
7. WT7-Unit 3 Weight Set

NIST Class 1 Weight sets:

1. W-01 Denver Instrument set: Serial number 98-121303 Class 1
2. W-03 Troemner set: Serial number 7283 Class 1

Spectrum Analytical, Inc. Rhode Island Division

CONFIDENTIALITY, ETHICS, and DATA INTEGRITY AGREEMENT

APPENDIX B

CONFIDENTIALITY, ETHICS, AND DATA INTEGRITY

The confidentiality, ethics, and data integrity agreement attached must be signed and dated by all new personnel associated with the data generated by Spectrum Analytical, Inc. Rhode Island Division. All said personnel will complete a training course and understand the information stated in the agreement. The course must include the ethical and legal responsibilities including the potential punishments and penalties for improper, unethical, or illegal actions. In addition, personnel are instructed on the importance of data confidentiality in both hard copy and digital forms. All personnel must fully understand this information before signing the agreement. A separate form is used for subcontractors and external auditors that request data for review.

Data Integrity training will be done on an annual basis. All employees are required to attend a training session or read a refresher document and sign off in hardcopy or through the digital SOP Database. All hard copy documents are stored in the employee's personnel file located in the QA Department.

All upper management personnel are required to sign a Non-disclosure Agreement which covers protecting confidentiality and proprietary rights. This Agreement is kept on file at the Spectrum Analytical, Inc., main offices in Agawam, Massachusetts.

SPECTRUM ANALYTICAL, INC.
FEATURING HANIBAL TECHNOLOGY
Rhode Island Division

CONFIDENTIALITY, ETHICS AND DATA INTEGRITY AGREEMENT

I. I, _____ *(Name)*, state that I understand the standards of confidentiality, ethics and data integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Spectrum Analytical, Inc. Rhode Island Division.

II. I agree that in the performance of my duties at Spectrum Analytical, Inc. Rhode Island Division. :

A. I shall not improperly use manual integrations to meet calibration or method QC criteria, such as peak shaving or peak enhancement.

B. I shall not intentionally misrepresent the date or time of analysis by resetting computer or instrument date/time.

C. I shall not falsify analytical results.

D. I shall not report analytical results without proper analysis documentation to support the results; dry-labbing.

E. I shall not selectively exclude data to meet QC criteria, such as calibration points, without technical or statistical justification.

F. I shall not misrepresent laboratory performance by presenting calibration data or QC limits within data reports that are not linked to the data set reported.

G. I shall not represent matrix interference as basis for exceeding acceptance criteria in interference-free matrices, such as method blanks and Laboratory Control Standards (LCS).

H. I shall not manipulate computer software for improper background subtraction or chromatographic baseline manipulations.

I. I shall not alter analytical conditions such as EM voltage, GC temperature program, etc. from standards analysis to sample analysis.

J. I shall not misrepresent QC samples such as adding surrogates after sample extraction, omitting sample preparation steps, or over-spiking/under-spiking.

K. I shall not report analytical results from the analysis of one sample for those of another.

L. I shall not intentionally represent another individual's work as my own.

III. I agree to report immediately any accidental or intentional reporting of non-authentic data by myself. Such report must be made to any member of Spectrum Analytical, Inc. Rhode Island Division Management or the QA Director (Hanibal Tayeh, Yihai Ding, Edward Lawler, Cinde Gomes, Sharyn Lawler) both orally and in writing.

IV. I agree to report immediately any accidental or intentional reporting of non-authentic data by other employees. Such report must be made to any member of Spectrum Analytical, Inc. Rhode Island Division Management or the QA Director (Hanibal Tayeh, Yihai Ding, Edward Lawler, Cinde Gomes, Sharyn Lawler) both orally and in writing.

V. Questions pertaining to confidentiality, ethics, and integrity may be posed to any of the above individuals.

VI. I agree not to divulge any pertinent confidential information including but not limited to data and any other information about a project to outside sources without the prior consent from the client.

I understand that failure to comply with the above confidentiality, ethics and data integrity agreement can result in my immediate dismissal from Spectrum Analytical, Inc. Rhode Island Division.
.

_____ _____

(Signature) *(Date)*

_____

(Print Name)

Training Session Record

Please read, sign and follow the instruction (s) below.

Subject: Confidentiality, Ethics and Integrity Training to include proper laboratory practices with an understanding of examples and consequences for falsifying data or sharing confidential information. Falsifying data can lead to written warning, termination, business closure, and/or civil or criminal prosecution. It is my responsibility to report to my supervisor (anonymously if I prefer) any acts that could lead to the falsifying of data.

I agree that I understand the procedure referenced above and have attended a training session for its proper implementation.

Staff Member Name	Date	Signature	Staff Member Name	Date	Signature

SUBCONTRACTORS

CONFIDENTIALITY, ETHICS AND DATA INTEGRITY AGREEMENT

I. I, _____ *(Name)*, authorized representative of

_____(Subcontractor) state that I understand the standards of integrity required of me and the Subcontractor with regard to the duties performed and the data reported in connection with the analysis/analyses contracted by Spectrum Analytical, Inc. Rhode Island Division.

II. Subcontractor agrees that in the performance of analysis for Spectrum Analytical, Inc. Rhode Island Division:

A. Subcontractor shall not intentionally report data values or results that are not the actual values measured or observed;

C. Subcontractor shall not modify data values unless the modification can be technically justified through a measurable analytical process;

D. Subcontractor shall not intentionally report the dates and times of data analyses that are not the true and actual dates and times of analyses; and

D. Subcontractor shall not intentionally represent another's work as its own.

III. Subcontractor agrees to report immediately any accidental or intentional reporting of non-authentic data to Spectrum Analytical, Inc. Rhode Island Division.

IV. Subcontractor agrees not to divulge any pertinent information including but not limited to data and information about any Spectrum Analytical, Inc. Rhode Island Division projects to outside sources without the prior consent from Spectrum or its clients.

I understand that failure to comply with the above ethics and data integrity agreement can result in immediate termination of the subcontract relationship with Spectrum Analytical, Inc. Rhode Island Division.

_____ _____

(Signature) *(Date)*

_____

(Name)

_____

(Title)

Confidentiality Agreement for External Audits

During the course of the laboratory audit/assessment certain information may become available to the auditor/assessor that is confidential.

All sample-related and project-related information at Spectrum Analytical, Inc. Rhode Island Division is confidential between Spectrum Analytical, Inc. Rhode Island Division and its client.

Any information obtained during the course of this audit/assessment may be used for audit/assessment purposes only.

No information obtained during the course of this audit/assessment may be disclosed by the auditor/assessor to any outside party, regardless of affiliation with the auditor/assessor.

Auditor/Assessor (signature): _____

(Print name): _____

(Date):_____

Company/organization name: _____

QAF.0014

Spectrum Analytical, Inc. RI Division
Resumes of Key Personnel

APPENDIX C

YIHAI DING
Laboratory Director

Mr. Ding has experience in a wide variety of analytical chemistry techniques, including GC, GC/MS, HPLC and FTIR. His expertise includes the operation, calibration and maintenance of sophisticated analytical instrumentation, and the efficient operation of state of the art environmental service laboratories.

Mr. Ding's responsibilities as Laboratory Director at Spectrum Analytical, Inc. Featuring Hanibal Technology Rhode Island Division, involves the daily coordination of all laboratory sections to insure the production of high quality data meeting customer's technical and schedule requirements. His duties in this role include overseeing department supervisors and analysts in the daily calibration, maintenance and troubleshooting of analytical instruments, monitoring schedules and holding times, analysis of samples, review of sample and QC data. He also is involved with the implementation of Standard Operating Procedures, documentation of instrument and method QC criteria and development of new methods and implementation of new analytical technology.

Mr. Ding's prior experience includes research into the mechanisms and kinetics of various biochemical processes. A large portion of this research has required the analysis of reactants and products using state-of-the-art chemical instrumentation. Mr. Ding has also taught chemistry and biochemistry laboratory courses at the university level.

EDUCATION

MIDDLE TENNESSEE STATE UNIVERSITY
Murfreesbro, Tennessee
- Chemistry, MS

JILIN UNIVERSITY
Changchun, China
- Biochemistry, BS

RELATED EXPERIENCE

2005-present

Spectrum Analytical, Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)
- Laboratory Director

2005 **STL LABORATORIES**
Savannah, Georgia
- Supervisor of Semi-Volatile GC and GC/MS
- GC/MS Analyst
- GC/ECD Analyst

1998-2005 **MITKEM CORPORATION**
Warwick, Rhode Island
- GCMS Supervisor for both Volatile Organics and Semi-Volatile Organics Laboratories
- GC/MS Analyst

1994-1998 **MIDDLE TENNESSEE STATE UNIVERSITY**
Murfreesboro, Tennessee
- Researcher
- Laboratory Instructor, chemistry and biochemistry

1993-1994 **NATIONAL ENZYME ENGINEERING LAB**
Changchun, China
- Researcher

SHARYN B. LAWLER

Quality Assurance Director

Ms. Lawler has over twenty years of experience in the environmental laboratory industry. She has experience in implementation, operation and management of QA systems operating under USEPA, US Army Corps of Engineers and NELAC programs.

Ms. Lawler's responsibilities as Quality Assurance Director include development and implementation of the Quality Assurance Plan and Standard Operating Procedures. Her duties include interacting with federal and state regulatory officials in the acquisition and maintenance of laboratory certifications. She is also responsible for managing Spectrum Analytical, Inc. Rhode Island Division's document control program. Ms. Lawler performs both internal and external audits as well as overseeing the corrective action system, training program and evaluating QC check samples.

Previously Ms. Lawler was a senior data reviewer, where she was responsible for final QA/QC review of organic, metals and wet chemistry data. She insured final data met all method and in-house QC criteria prior to release to the customer, and that any issues were documented and described for inclusion in case narratives. A significant portion of this work involved review of full CLP-format data deliverables packages, both for standard as well as non-routine analyses. Prior to Spectrum Analytical Inc., Ms. Lawler worked for two CLP laboratories where she held positions including senior data review specialist, CLP Organics Task Manager and analyst in several laboratory sections.

EDUCATION:

UNIVERSITY OF MASSACHUSETTS
Amherst, Massachusetts
Independent Conc., Coastal Plant Ecology, BS

RELATED EXPERIENCE:

1997-Present

Spectrum Analytical Inc., Featuring Hanibal Technology, RI Division (formerly Mitkem)
- QA Director
- Senior Data Reviewer

1988-1997 **NATIONAL ENVIRONMENTAL TESTING**
Bedford, Massachusetts
- Senior Data Reviewer
- CLP Organic Task Manager

1983-1988 **CAMBRIDGE ANALYTICAL ASSOCIATES**
Boston, Massachusetts
- CLP Organic Task Manager
- Semivolatiles Analyst
- Preparation Laboratory Analyst

EDWARD A. LAWLER

Business Development Coordinator /Sr. Project Manager

Mr. Lawler has over thirty years of experience in environmental laboratory operations. He has extensive experience in managing laboratory workflow and in establishing and maintaining customer relationships. He also has considerable experience in a wide range of environmental chemical analyses, with a concentration in trace level volatile organics analysis.

As Business Development Coordinator, Mr. Lawler is responsible for securing contracts and BOA agreements with clients as well as pursuing new contracts and bids. He also works closely with lab staff to ensure they are aware of specific data deliverable requirements for new projects.

As Senior Project Manager, Mr. Lawler manages certain significant analytical testing programs, acting as principal technical liaison with the client. His extensive experience in laboratory data review allows him to ensure QA/QC criteria have been achieved, as well as preparing project narratives detailing these findings to the client.

Mr. Lawler's past responsibilities as Deputy Director for Quality Services included the prioritization of all analytical chemistry testing at Spectrum Analytical, Inc. Rhode Island Division. This included daily meetings with laboratory supervisors and managers to insure all technical and schedule requirements were met.

Mr. Lawler's previous experience includes various staff, management and senior management positions at several environmental testing laboratories. Direct project experience includes EPA CLP, Army MRD, Navy NEESA and NFESC, DOD HAZWRAP and New York DEC ASP programs. Mr. Lawler has also provided expert testimony at several Superfund trials including pre-trial consulting and trial witness services.

EDUCATION: **UNIVERSITY OF MASSACHUSETTS**
Amherst, Massachusetts
Environmental Sciences, BS 1977

RELATED EXPERIENCE:

1997- Present	**Spectrum Analytical Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)** - Business Development Coordinator - Senior Project Manager - Deputy Director for Quality Services - Operations Manager
1989-1997	**NATIONAL ENVIRONMENTAL TESTING, CAMBRIDGE DIVISION** Bedford, Massachusetts - Division Manager - Proposal/Contract Manager - Director of Project Management
1983-1989	**CAMBRIDGE ANALYTICAL ASSOCIATES, INC.** Boston, Massachusetts - Project Manager - Volatile Organic Laboratory Manager
1978-1983	**ENERGY RESOURCES COMPANY, INC. - ERCO** Cambridge, Massachusetts - Volatile Organics (GC) Manager - Analytical Chemist-Volatile Organics Lab - Analytical Chemist-Organic Preparation Lab
1978	**LAPUCK LABORATORIES, INC.** Watertown, Massachusetts - Analytical Chemist-Wet Chemistry & Metals - Microbiologist

SCOTT P. HUNTLEY

IT Manager

Mr. Huntley has over twenty years experience in the environmental testing field. He has considerable experience in computer sciences and had been involved, throughout his career, in the setup and implementation of several Laboratory Information Management Systems (LIMS) and automated data reduction systems. Mr. Huntley's responsibilities include the set-up and validation of automated data transfer, reduction, storage, evaluation and reporting programs within Spectrum Analytical, Inc. RI Division's LIMS. He also is responsible for set-up of the electronic data delivery capabilities as well as the control charting capabilities of this system.

Previously Mr. Huntley has held several supervisory positions in environmental laboratories focusing on CLP and other DOD analytical programs. He has a wide range of experience in routine and state of the art analytical programs and methods. Mr. Huntley is experienced in the use of automated data transfer and reduction systems and laboratory automation techniques.

EDUCATION:

RHODE ISLAND COLLEGE
Providence, Rhode Island
Chemistry, BS
Computer Science, BS

RELATED EXPERIENCE:

1999-Present	**Spectrum Analytical, Inc., Featuring Hanibal Technology, RI Division (formerly Mitkem)** MIS Senior Systems Analyst
1996-1999	**MITKEM CORPORATION** Warwick, RI - Senior Chemist - Organic Lab Manager
1991-1996	**EA LABORATORIES** Sparks, MD

- Supervisor of Organic Chemists

1989-1991 **CEIMIC CORPORATION**
Narragansett, RI
- Night shift supervisor

1986-1989 **RI ANALYTICAL LABORATORIES**
Providence, RI
- GC Chemist

Catherine L. Mosher

Organics (SVOA/VOA) Department Manager

Ms. Mosher has experience in a wide variety of analytical chemistry techniques, including GC/FID and GC/MS. Her expertise includes the operation, calibration and maintenance of sophisticated, computer controlled instrumentation. Her expertise also includes analyses and QA review of forensics extended alkylated PAH and Biomarker analyses.

Ms. Mosher is employed as the Organics Department Manager in Spectrum Analytical Inc. Rhode Island Division, and oversees both the Volatile and Semivolatile departments. Ms. Mosher's responsibilities involve the coordination of organics analyses using GC/MS and GC instrumentation following both US EPA CLP and SW846 protocols. Her duties in this role include supervising analysts in the daily calibration, maintenance and troubleshooting of analytical instruments, monitoring schedules and holding times, analysis of samples, review of sample and QC data. She is involved with the implementation of Standard Operating Procedures, documentation of instrument and method QC criteria and development of new methods and implementation of new analytical technology. Ms. Mosher also insures the production of organic data is coordinated with other laboratory sections.

EDUCATION

Community College of Rhode Island
Warwick, Rhode Island
Certificate of Chemical Technology - 1991

RELATED EXPERIENCE

02/2007-Present

Spectrum Analytical Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)
- Manager, SVOA Department
- Senior Scientist, SVOA Laboratory

05/2005 – 12/2006

Alpha Woods Hole Laboratories
Raynham, MA
- Development of Volatile Air Laboratory

- Supervisor for Organics analyses including GC/MS VOA and SVOA, ECD's and FIDs
- Forensic Team Leader

03/1997 – 05/2005 **Woods Hole Group Laboratories**
Raynham, MA
- Forensic Team Leader
- GC/MS Group Leader

04/1996 – 03/1997 **Inchcape Testing**
New Bedford and Raynham, MA
- Semivolatile analyst
- Volatile analyst

09/1991 – 04/1996 **Energy and Environmental Engineering Inc.**
Somerville, MA
- Semivolatile GC/MS Supervisor
- GC-Pesticide/PCB analyst

01/1989 – 09/1991 **New England Testing Laboratory**
North Providence, RI
- Senior Chemical Technician - including Organic, Inorganic, Metals, and Microbiology analyses

10/1987 – 09/1988 **Rhode Island Analytical Laboratory**
Warwick, RI
- Chemical Technician

HUIYAN HEATHER ZHAO-ANDERSON
Inorganics Department Manager

Ms. Zhao-Anderson is employed as the Manager in Spectrum Analytical Inc. Rhode Island Division's Inorganics Department. Ms. Zhao-Anderson's responsibilities involve the coordination of metals and wet chemistry analyses using ICP/MS, ICP/AES and a variety of other instrumentation following both US EPA CLP and SW846 protocols. Her duties in this role include supervising analysts in the daily calibration, maintenance and troubleshooting of analytical instruments, monitoring schedules and holding times, analysis of samples, review of sample and QC data. She is involved with the implementation of Standard Operating Procedures, documentation of instrument and method QC criteria and development of new methods and implementation of new analytical technology. Ms. Zhao-Anderson also insures the production of inorganics organic data is coordinated with other laboratory sections. Prior to managing the inorganic department, Ms Zhao-Anderson was the department manager of our volatile organics laboratory for several years.

EDUCATION

Yale University
New Haven, CT
School of Forestry and
Environmental Study, MS 2005

Peking University
Beijing, China
Environmental Science and Economics
BS 2002

RELATED EXPERIENCE

09/2005 -Present

Spectrum Analytical Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)
- Manager, Inorganic Department
- Manager, VOA Department
- GC/MS Chemist, VOA Laboratory

DAWNE SMART

Data Reviewer, Project Manager, Data Reporting Supervisor

Ms. Smart's responsibilities as project manager involve the management of Spectrum Analytical Inc. Rhode Island Division's EPA Contract Laboratory Program (CLP) analytical services contract for ISM. This includes the daily oversight of incoming samples, maintenance of chain of custody documentation and communication records and resolution of any discrepancies or other issues involving CLP ISM sample assignments. Her responsibilities also include ongoing communication with EPA, sampler and CSC personnel, as well as monitoring data delivery schedules, writing project narratives and finalizing case communication.

Ms. Smart also is currently supervising the Data Reporting staff. She oversees the staff that generates data packages for all inorganic and organic fractions for different levels of report packages that will then go to data review personnel. Additionally, she and her staff are responsible for final report generation when all fractions of a project are completed, including bookmarking, pagination, final package posting to the website and hard copy report mailing if applicable.

Ms Smart also reviews sample and QC data, and completed CLP data packages for both organic and inorganic programs. Ms. Smart has extensive experience in Data Review as well as Quality Assurance. A significant portion of her previous employment included management of the Data Review department as well as the on-site QA Specialist for a major specialized laboratory.

EDUCATION

COMMUNITY COLLEGE of RHODE ISLAND
Warwick, Rhode Island
Certificate of Chemical Technology - 1991
Associate in Applied Science - 1997

RELATED EXPERIENCE

2007-Present

Spectrum Analytical Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)
- Data Reporting Supervisor
- ISM Contract manager

	-Manager, Metals Department -Supervisor, Inorganic Department
1999 – 2007	**ALPHA WOODS HOLE LABORATORIES** Raynham, Massachusetts -QA Specialist -Manager, Data Review Department
1996 – 1999	**ANALYTICAL BALANCE COMPANY** Middleboro, Massachusetts - Department Head, Metals Analysis
1995 – 1996	**FOXBORO COMPANY** West Bridgewater, Massachusetts - Chemist
1988 – 1995	**NEW ENGLAND TESTING LABORATORY** North Providence, RI - Senior Laboratory Technician - Laboratory Technician
1987 – 1988	**RHODE ISLAND ANALYTICAL LABORATORIES** Warwick, RI - Metals Preparation Technician - Laboratory Assistant -

AGNES R. HUNTLEY

Project Manager

Ms. Huntley has gained extensive and diversified experience in environmental laboratories using U.S. EPA CLP and SW846 methodologies, as well as participating in US Navy and Army analytical services programs.

Ms. Huntley's responsibilities involve the management of Spectrum Analytical Inc. Rhode Island Division's EPA Contract Laboratory Program (CLP) analytical services contracts. This includes the daily oversight of incoming samples, maintenance of chain of custody documentation and communication records and resolution of any discrepancies or other issues involving CLP sample assignments. Her responsibilities also include ongoing communication with EPA, sampler and CSC personnel, as well as monitoring data delivery schedules, writing project narratives and finalizing case communication. Ms. Huntley has managed four contracts with the EPA, which included one Organics Low Concentration (OLC), two Organics Low/Medium Concentration (OLM) and one Inorganics Low/Medium Concentration (ILM) analytical services contracts. At present Ms. Huntley manages the Organics Multi-Media, Multi-Concentration (SOM01.2) Analytical Services Contract.

Previously, Ms. Huntley held the position of QA/QC Manager where her responsibilities included the development and implementation of Standard Operating Procedures, documentation of instrument and method performance using Method Detection Limit studies, and routine review of final laboratory data reports, review of analyst training and performance data and management of the corrective action system. Her duties also included interaction with federal and state regulatory officials in the acquisition and maintenance of laboratory certifications.

Prior experience includes management of the daily operations of the Organic Preparation Laboratory. Duties in this position included monitoring sample backlog, holding times, process work flow, and delivery due dates. Ms. Huntley also developed and implemented new test methods, trained laboratory staff, performed instrument maintenance and reviewed sample and QC data. Prior to joining Spectrum Analytical Inc. Ms. Huntley worked as an analytical chemist at NET Cambridge Division performing analyses under a wide variety of programs including Army COE, Navy NEESA, DOE HAZWRAP and EPA CLP.

EDUCATION

SIMMONS COLLEGE
Boston, Massachusetts
- Chemistry, BS
- Mathematics, BS

RELATED EXPERIENCE

1997-Present	**Spectrum Analytical, Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)** - Project Manager, SOM Contract manager - Supervisor, Sample Receiving Department
1997-2008	**MITKEM CORPORATION** Warwick, Rhode Island - CLP Project Manager - QA/QC Manager - Manager, Sample Preparation Laboratory
1995-1997	**NATIONAL ENVIRONMENTAL TESTING** Bedford, Massachusetts - Chemist, Organic Preparation
1992-1995	**SIMMONS COLLEGE CHEMISTRY DEPT.** Boston, Massachusetts - Teaching Assistant, Chemistry Department

QAP Revision Page:

Rev 1 (02/01/2013): Included Facility floor plan, Updated Org Chart, updated equipment list, DW metals reporting requirements per 310 CMR 42

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Appendix B
Standard Operating Procedures
Field and Laboratory

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APPENDIX B-1
EA
Standard Operating Procedures - Field

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Standard Operating Procedure No. 001
for
Sample Labels

Prepared by

EA Engineering, Science, and Technology, Inc.
225 Schilling Circle, Suite 400
Hunt Valley, Maryland 21031

Revision 0
August 2007

CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for the use of sample labels. Every sample will have a sample label uniquely identifying the sampling point and analysis parameters. An example label is provided below. Other formats with similar levels of detail are acceptable.

PROJECT NAME _____ PROJECT NUM. _____
SAMPLE LOCATION/SITE ID _____
DATE: ____/____/____ TIME: _____:_____
ANALYTES: METALS VOC EXPLOSIVES ORGANICS OTHER
FILTERED: [NO] [YES]
PRESERVATIVE: [NONE] [HNO_3] [OTHER _____]
SAMPLER: _____

2. MATERIALS

The following materials may be required: sample label and indelible laboratory marker.

3. PROCEDURE

The following sections describe how to use the sample labeling system.

3.1 LABEL INFORMATION

As each sample is collected/selected, fill out a sample label. Enter the following information on each label:

- Project name
- Project number
- Location/site identification—Enter the media type (i.e., well number, surface water, soil, etc.) sampling number, and other pertinent information concerning where the sample was taken
- Date of sample collection

- Time of sample collection
- Analyses to be performed (NOTE: Due to number of analytes, details of analysis should be arranged with laboratory *prior to start of work)*
- Whether filtered or unfiltered (water samples only)
- Preservatives (water samples only)
- Number of containers for the sample (e.g., 1 of 2, 2 of 2).

3.2 ROUTINE CHECK

Double-check the label information to make sure it is correct. Detach the label, remove the backing, and apply the label to the sample container. Cover the label with clear tape, ensuring that the tape completely encircles the container.

3.3 RECORD INFORMATION

Record the sample number and designated sampling point in the field logbook, along with the following sample information:

- Time of sample collection (each logbook page should be dated)
- Location of the sample
- Organic vapor meter or photoionization meter readings for the sample (when appropriate)
- Any unusual or pertinent observations (oily sheen on groundwater sample, incidental odors, soil color, grain size, plasticity, etc.)
- Number of containers required for each sample
- Whether the sample is a quality assurance sample (split, duplicate, or blank).

3.3.1 Logbook Entry

A typical logbook entry might look like this:

- 7:35 a.m. Sample No. MW-3. PID = 35 ppm
- Petroleum odor present. Sample designated MW-3-001.

NOTE: Duplicate samples will be given a unique sample designation rather than the actual sample number with an added prefix or suffix. This will prevent any indication to the laboratory that this is a duplicate sample. This fictitious sample number will be listed in the logbook along with the actual location of the sample.

3.4 SHIPMENT

Place the sample upright in the designated sample cooler. Make sure there is plenty of ice in the cooler at all times.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

5.1 INCIDENTAL ODORS

Note that although incidental odors should be noted in the logbook, it is unwise from a safety and health standpoint to routinely "sniff test" samples for contaminants.

5.2 DUPLICATE SAMPLE

No indication of which samples are duplicates is to be provided to the laboratory.

6. REFERENCES

U.S. Environmental Protection Agency. 1980. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans. QAMS-005/80.

Standard Operating Procedure No. 002
for
Chain-of-Custody Form

Prepared by

EA Engineering, Science, and Technology, Inc.
225 Schilling Circle, Suite 400
Hunt Valley, Maryland 21031

Revision 0
August 2007

CONTENTS

Page

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for use of the chain-of-custody form. An example is provided as Figure SOP002-1. Other formats with similar levels of detail are acceptable.

2. MATERIALS

The following materials may be required: chain-of-custody form and indelible ink pen.

3. PROCEDURE

- Give the site name and project name/number.
- Enter the sample identification code.
- Indicate the sampling dates for all samples.
- List the sampling times (military format) for all samples.
- Indicate “grab” or “composite” sample with an “X.”
- Specify the sample location.
- Enter the total number of containers per cooler.
- List the analyses/container volume.
- Obtain the signature of sample team leader.
- State the carrier service and airbill number, analytical laboratory, and custody seal numbers.
- Sign, date, and time the “relinquished by” section.
- Upon completion of the form, retain the shipper copy, and affix the other copies to the inside of the sample cooler, in a zip-seal bag to protect from moisture, to be sent to the designated laboratory.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

U.S. Environmental Protection Agency (U.S. EPA). 1980. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80.

———. 1990. Sampler's Guide to the Contract Laboratory Program. EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C. December.

———. 1991. User's Guide to the Contract Laboratory Program. EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response. January.

Company Name:	Project Manager or Contact: Phone:	Parameters/Method Numbers for Analysis	Chain of Custody Record
			EA Laboratories 19 Loveton Circle Sparks, MD 21152 Telephone: (410) 771-4950 Fax: (410) 771-4077
Project No. Dept.: Task:	Project Name:		
Sample Storage Location:	ATO Number:		**Report Deliverables:** **1 2 3 4 D E** **EDD: Yes/No** **DUE TO CLIENT:** __________
Page of	**Report #:**		

Date	Time	Water	Soil	Sample Identification 19 Characters	**No. of Containers**														**EA Labs Accession Number**	Remarks
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Samples by: (Signature)	Date/Time	Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Date/Time	Received by Laboratory: (Signature)	Date/Time	Airbill Number:	Sample Shipped by: (Circle) Fed Ex. Puro. UPS Hand Carried Other:
Cooler Temp. _____C pH: ___ Yes ___ No Comments:				**Custody Seals Intact ___ Yes ___ No**	
NOTE: Please indicate method number for analyses requested. This will help clarify any questions with laboratory techniques.					

Standard Operating Procedure No. 003
for
Subsurface/Utility Clearance

Prepared by

EA Engineering, Science, and Technology, Inc.
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Revision 0
August 2007

CONTENTS

1. SCOPE AND APPLICATION

1.1 PURPOSE

The purpose of this Standard Operating Procedure is to prevent injury to workers and damage to subsurface structures (including tanks, pipe lines, water lines, gas lines, electrical service, etc.) during ground disturbance activities (including drilling, augering, sampling, use of direct-push technologies, excavation, trenching, concrete coring or removal, fence post installation, grading, or other similar operations).

1.2 LIMITATIONS

The procedures set forth in this document are the suggested procedures but may not be applicable to particular sites based on the site-specific considerations. The Project Manager is responsible for making a site-specific evaluation of each site to determine whether the Subsurface/Clearance Procedures should be utilized or require modification. If safety or other site-specific considerations require a modified or different procedure, the Project Manager should review the modified procedure with the Business Unit Director, Profit Center Manager, or Senior Technical Reviewer.

1.3 SCOPE

This procedure provides minimum guidance for subsurface clearance activities, which must be followed prior to and during ground disturbance activities at EA project sites. Even after completing the subsurface clearance activities required in this procedure, all ground disturbance activities should proceed with due caution.

Deviations from this procedure may be provided on an exception basis for specific situations, such as underground storage tank systems removals, verified aboveground/overhead services/lines, undeveloped land/idle facilities, shallow groundwater conditions, soil stability, or well construction quality assurance/quality control concerns, etc.

EA or its subcontractors are responsible for, and shall ensure that, all ground disturbance activities are completed safely, without incident, and in accordance with applicable federal, state, and local regulations.

This procedure shall not override any site-specific or consultant/contractor procedures that are more stringent or provide a greater degree of safety or protection of health or the environment.

2. PROCEDURES

The EA Project Manager or his designee must complete the Subsurface Clearance Procedure Checklist (Appendix A) in conjunction with the following procedures. The checklist must be completed before initiating any ground disturbance activities. The completed checklist must be submitted to the appropriate team individuals, subcontractors, and/or the client and included in the project files.

2.1 SAFETY

A Health and Safety Plan must be available onsite and followed by all contractors and subcontractors.

All work areas shall be defined and secured with safety cones, safety tape, construction fence, other barriers, or signs as appropriate.

Site work permits must be obtained as required by site procedures. Based on site conditions or classification, the use of intrinsically-safe equipment may be required.

To ensure the safety of all onsite personnel and subsurface structure integrity, consideration should be given to de-energizing and locking out selected site utilities or temporarily shutting down a portion of or the entire facility.

2.2 PREPARATION TASKS

Objective—To gather all relevant information about potential subsurface structures prior to the actual site visit.

2.2.1 Obtain Permits and Site Access

The consultant/contractor is responsible for following all applicable laws, guidance, and approved codes of practice; obtaining all necessary permits and utility clearances; and securing site access permission.

2.2.2 Historic Site Information

Obtain most recent as-built drawings and/or site plans (including underground storage tank, product, and vent lines) as available.

NOTE: As-built drawings may not accurately depict the locations and depths of improvements and subsurface structures and should, therefore, not be **solely** relied upon.

EA should obtain any other site information such as easements, right-of-ways, historical plot plans, fire insurance plans, tank (dip) charts, previous site investigations, soil surveys, boring logs, and aerial photographs, etc. as relevant to the planned ground disturbance activities.

Where applicable, EA should also contact contract personnel who may have historic site knowledge.

2.2.3 Mark-Outs

Objective—To identify location of subsurface structures on surface.

EA must ensure that a thorough mark-out at the site is completed to locate electrical, gas, telephone, water, sewer, low voltage electric lines, product delivery pipelines, fiber optic, and all other subsurface utilities/services.

- Where available, public utility companies must be contacted to identify underground utilities. (This can be accomplished through the One-Call system in most instances.)

- In addition, where available and warranted by site conditions, a private utility/pipeline mark-out company should be contracted to perform an electronic subsurface survey to identify the presence of suspected hazardous or critical underground utilities and subsurface structures. In some cases, this is necessary to confirm public utility mark-outs in the vicinity of planned ground disturbance activities.

EA will review all available site plan subsurface information with the private mark-out company to assist in locating utilities and other subsurface structures.

NOTE: Mark-outs may not accurately depict the exact locations of improvements and subsurface structures and should, therefore, not be **solely** relied upon.

Where possible, EA personnel are encouraged to be onsite at the time of subsurface mark-outs. This is to ensure accuracy and understanding of subsurface structures identified and provides an opportunity to exchange information with mark-out company personnel regarding planned work activities.

Subsurface structures should be marked throughout the entire work area(s) with adequate materials (e.g., site conditions may require paint and tape/flags). Ground disturbance activities must be started within 30 days of mark-out, unless local ordinances specify a shorter time period. If activities are not started within required time period or markings have faded, mark-outs must be redone.

EA personnel will record time and date of mark-out request and list all companies contacted by the service and confirmation number. This should be available for review onsite and checked off after visual confirmation of markings.

2.2.4 Initial Site Visit

Objective—To compare the site plan to actual conditions based on information gathered in Procedures 2 and 3 above, obtain additional site information needed, and prepare a vicinity map.

EA will document all findings and update the site plan with this information. On third party sites, close coordination with the site owner's representatives for mark-outs, review of as-builts, and other information reviews should be conducted prior to work. Project Managers are encouraged to provide updated as-built information to the client.

In some regions, it may be more effective and efficient to conduct the site visit at the same time the contractor and drill rig are mobilized to the site. The inspection should include the following activities and may include others as determined by the consultant/contractor and the Project Manager.

2.2.5 Utilities

EA shall perform a detailed site walk-through for the purpose of identifying all aboveground indicators of subsurface utilities/services that may be leading to or from buildings within the planned work area. The inspection shall include, but not be limited to, the following:

- Utility mark-outs
- Aboveground utilities
- Area lights/signs
- Phones
- Drains
- Junction boxes
- Natural gas meters or connections
- Other utilities including: fire hydrants, on/below grade electrical transformers, splice cages, sewer lines, pipeline markers, cable markers, valve box covers, clean-outs/traps, sprinkler systems, steam lines (including insulated tanks that may indicate steam lines), and cathodic protection on lines/tanks
- Observe paving scars (i.e., fresh asphalt/concrete patches, scored asphalt/concrete).

NOTE: In many cases, the onsite location of low-voltage electrical lines and individual property water and sewer line branches may be approximated by using the following technique:

- Locate the entry/connection location at the facility building

- Attempt to identify utility connections for the mains (water sewer, etc.) by locating clean-outs, valve manways, etc. The location path of the utility is likely with the area between the main connection and facility building connection. Subsurface electrical line locations from the facility building to signs, lamps, etc. can be estimated with the same process.

2.2.6 Other Subsurface Systems

Some other subsurface systems to be cognizant of during subsurface activities include product delivery systems (i.e., at gas stations) and existing remediation systems.

2.2.7 Selection of Ground Disturbance Locations

EA will utilize the information collected to this point in combination with regulatory requirements and project objectives to select ground disturbance locations. Ground disturbance locations should also consider the location of overhead obstructions (e.g., power lines). Work at active gasoline retail locations must consider several special considerations that should be outlined in the site-specific safety and health plan.

2.2.8 Review of Selected Locations with the Client

EA will review the selected ground disturbance locations with the client. EA will not proceed with the subsurface activities until the plan has been discussed with the client. During execution of the project, subsurface activities are required outside of the area previously approved by the client. EA will submit these changes to the client for approval prior to execution.

2.2.9 Ground Disturbance Activity Sequence

EA will plan ground disturbance activities starting at the point farthest from the location of suspected underground improvements. This is done to determine the natural subsurface conditions and to allow EA site personnel to recognize fill conditions.

Experience has shown that the following warning signs may indicate the presence of a subsurface structure:

- Warning tape (typically indicative of underground services).

- Pea gravel/sand/non-indigenous material (typically indicative of tanks or lines).

- Red concrete (typically indicative of electrical duct banks).

- The abrupt absence of soil recovery in a hand auger. This could indicate pea gravel or sand that has spilled out of the auger. This may not be indicative in areas where native soil conditions typically result in poor hand auger recoveries.

- Any unexpected departure from the native soil or backfill conditions as established by prior onsite digging.

If any of these conditions is encountered by EA site personnel, digging should stop and the client should be contacted.

3. SUBSURFACE CLEARANCE METHODS

The method used to delineate the subsurface should be compatible with the inherent associated risk given the type of facility/property, soil stratigraphy, and the location of the ground disturbance activity, such that required delineation is obtained. It should be noted that in areas where there is paving, sufficient paving should be removed to allow clear visibility of the subsurface conditions during clearance activities. The following is a list of potential clearance methods that may be used on a job site:

- Vacuum digging
- Probing
- Hand digging
- Hand augering
- Post-hole digging.

EA personnel will evaluate the potential for electrical shock or fire/explosion for each subsurface disturbance project and will evaluate as necessary the use of non-conductive or non-sparking tools (i.e., fiberglass hand shovels, and thick electrically insulating rubber grips on hand augers or probes). The potential need for the use of non-conductive materials, electrical safety insulated gloves, and footwear will also be evaluated on a case-by-case basis.

3.1 SUBSURFACE CLEARANCE PROCEDURES FOR DRILLING, DIRECT-PUSH TECHNOLGY, AUGERING, FENCE POST INSTALLATION, OR OTHER BOREHOLE INSTALLATION ACTIVITIES

The area to be delineated will exceed the diameter of the largest tool to be advanced and sufficiently allow for visual inspection of any obstructions encountered.

3.2 SUBSURFACE CLEARANCE PROCEDURES FOR TRENCHING/ EXCAVATION ACTIVITIES

Appropriate subsurface clearance methods should be conducted along the length and width of the excavation at a frequency sufficient to ensure adequate precautions have been applied to the entire work area. The frequency and density of investigations will be based on site knowledge, potential hazards, and risks of the work area to surrounding locations (e.g., proximity to a residential area or school).

Whenever subsurface structures are exposed, EA will cease work and mark the area (e.g., flags, stakes, cross bracing) to ensure the integrity of these exposed structures is maintained during subsequent trenching/excavation/backfilling.

Uniform color codes for marking of underground facilities are provided in Appendix B.

Appendix A

Subsurface Clearance Procedure Checklist

Subsurface Clearance Procedure Checklist

Site Identification: _____

Project Consultant/Contractor: _____

Section 1: Safety, Preparation Tasks, and Mark-Outs

Activity	Yes	No	N/A	Comments including Justification if Response Is No or Not Applicable
Health and Safety Plan is available and all contractors and subcontractors are familiar with it.				
All applicable local, state, and federal permits have been obtained.				
Site access/permission has been secured.				
Most recent as-built drawings and/or site plans (including underground storage tank, product, and vent lines) obtained.				
Reviewed site information to identify subsurface structures relevant to planned site activities (easements, rights-of-way, historical plot plans, fire insurance plans, tank dip charts, previous site investigations, soil surveys, boring logs, aerial photographs, etc.).				
Utility mark-outs have been performed by public utility company(s). Mark-outs clear/visible.				
Subsurface structure mark-outs performed by private mark-out company. Mark-outs clear/visible.				
Additional Activities: Were dig locations reviewed with site representative?				

Section 2: Initial Site Visit and Selecting Ground Disturbance Locations

Activity	Yes	No	N/A	Comments including Justification if Response Is No or Not Applicable
Location of all aboveground indicators of subsurface utilities/services that may be leading to or from buildings within the planned work area has been identified.				
Location of utility mark-outs by all utility companies previously contacted has been identified within required time period.				
Location of all subsurface structure mark-outs by private mark-out company has been identified within required time period.				
Location of area lights/signs and associated subsurface lines identified.				
Location of all phones and associated subsurface lines identified.				
Location of all drains and associated interconnecting lines identified.				
Location of all electrical junction boxes and associated interconnecting lines identified				
Location of all natural gas meters or connections and all interconnecting lines identified				

Completed by: _____
Name

Signature: _____ _____
Company Date

Appendix B

Uniform Color Codes for Excavation

APWA UNIFORM COLOR CODE

WHITE - Proposed Excavation

PINK - Temporary Survey Markings

RED - Electric Power Lines, Cables, Conduit and Lighting Cables

YELLOW - Gas, Oil, Steam, Petroleum or Gaseous Materials

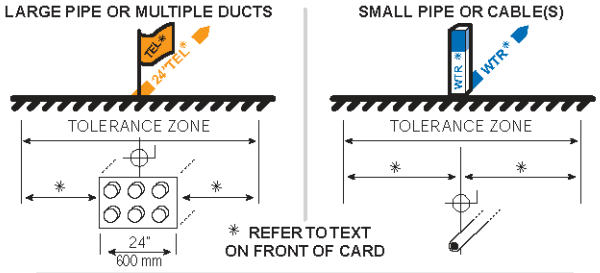
ORANGE - Communication, Alarm or Signal Lines, Cables or Conduit

BLUE - Potable Water

PURPLE - Reclaimed Water, Irrigation and Slurry Lines

GREEN - Sewers and Drain Lines

TYPICAL MARKING



Customize with your center's phone and address information

GUIDELINES FOR UNIFORM TEMPORARY MARKING OF UNDERGROUND FACILITIES

This marking guide provides for universal use and understanding of the temporary marking of subsurface facilities to prevent accidents and damage or service interruption by contractors, excavators, utility companies, municipalities or any others working on or near underground facilities.

ONE-CALL SYSTEMS
The One-Call damage prevention system shall be contacted prior to excavation.

PROPOSED EXCAVATION
Use white marks to show the location, route or boundary of proposed excavation. Surface marks on roadways do not exceed 1.5" by 18" (40 mm by 450 mm). The facility color and facility owner identity may be added to white flags or stakes.

USE OF TEMPORARY MARKING
Use color-coded surface marks (i.e., paint or chalk) to indicate the location or route of active and out-of-service buried lines. To increase visibility, color coded vertical markers (i.e., stakes or flags) should supplement surface marks. Marks and markers indicate the name, initials or logo of the company that owns or operates the line, and width of the facility if it is greater than 2" (50 mm). Marks placed by other than line owner/operator or its agent indicate the identity of the designating firm. Multiple lines in joint trench are marked in tandem. If the surface over the buried line is to be removed, supplementary offset markings are used. Offset markings are on a uniform alignment and clearly indicate the actual facility is a specific distance away.

TOLERANCE ZONE
Any excavation within the tolerance zone is performed with non-powered hand tools or non-invasive method until the marked facility is exposed. The width of the tolerance zone may be specified in law or code. If not, a tolerance zone including the width of the facility plus 18" (450 mm) measured horizontally from each side of the facility is recommended.

ADOPT UNIFORM COLOR CODE
The American Public Works Association encourages public agencies, utilities, contractors, other associations, manufacturers and all others involved in excavation to adopt the APWA Uniform Color Code, using ANSI standard Z535.1 Safety Colors for temporary marking and facility identification.

Rev. 4/99

Standard Operating Procedure No. 004
for
Sample Packing and Shipping

Prepared by

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Revision 0
August 2007

CONTENTS

Page

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for the packing and shipping of samples to the laboratory for analysis.

2. MATERIALS

The following materials may be required:

Clear tape	Plastic garbage bags
Custody seals	Sample documentation
Ice	Waterproof coolers (hard plastic or metal)
Metal cans with friction-seal lids (e.g., paint cans)	Zip-seal plastic bags
Packing material[1]	

3. PROCEDURE

Check cap tightness and verify that clear tape covers label and encircles container. Wrap sample container in bubble wrap or closed cell foam sheets. Enclose each sample in a clear zip-seal plastic bag.

Place several layers of bubble wrap, or at least 1 in. of vermiculite on the bottom of the cooler. Line cooler with open garbage bag, place all the samples upright inside a garbage bag, and tie the bag.

Double bag and seal loose ice to prevent melting ice from soaking the packing material. Place the ice outside the garbage bags containing the samples.

Pack shipping containers with packing material (closed-cell foam, vermiculite, or bubble wrap). Place this packing material around the sample bottles or metal cans to avoid breakage during shipment.

Enclose all sample documentation (i.e., Field Parameter Forms, chain-of-custodies) in a waterproof plastic bag and tape the bag to the underside of the cooler lid. If more than one cooler is being used, each cooler will have its own documentation.

Seal the coolers with signed and dated custody seals so that if the cooler were opened, the custody seal would be broken. Place clear tape over the custody seal to prevent damage to the seal.

1. Permissible packing materials are: (a) (non-absorbent) bubble wrap or closed cell foam packing sheets, or (b) (absorbent) vermiculite. Organic materials such as paper, wood shavings (excelsior), and cornstarch packing "peanuts" will not be used.

Refer to SOP Nos. 001, 002, 016, and 039.

Tape the cooler shut with packing tape over the hinges and place tape over the cooler drain. Ship all samples via overnight delivery on the same day they are collected if possible.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

Any samples suspected to be of medium/high contaminant concentration or containing dioxin must be enclosed in a metal can with a clipped or sealable lid (e.g., similar to a paint can). Label the outer metal container with the sample number of the sample inside.

6. REFERENCES

U.S. Environmental Protection Agency (U.S. EPA). 1980. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80.

———. 1990. Sampler's Guide to the Contract Laboratory Program. EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C. December.

———. 1991. User's Guide to the Contract Laboratory Program. EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response. January.

Standard Operating Procedure No. 005
for
Field Decontamination

Prepared by

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August 2007

CONTENTS

1. SCOPE AND APPLICATION

All personnel or equipment involved in intrusive sampling, or which enter a hazardous waste site during intrusive sampling, must be thoroughly decontaminated prior to leaving the site to minimize the spread of contamination and prevent adverse health effects. This Standard Operating Procedure describes the normal decontamination of sampling equipment and site personnel.

2. MATERIALS

The following materials may be required:

0.01N HCl	Non-phosphate laboratory detergent (liquinox)
0.10N nitric acid	Plastic garbage bags
Aluminum foil or clean plastic sheeting	Plastic sheeting, buckets, etc. to collect wash water and rinsates
Approved water	Pressure sprayer, rinse bottles, brushes
High performance liquid chromatography (HPLC)-grade water[1]	Reagent grade alcohol[2]

3. PROCEDURE

3.1 SAMPLE BOTTLES

At the completion of each sampling activity, the exterior surfaces of the sample bottles must be decontaminated as follows:

- Ensure the bottle lids are on tight.
- Wipe the outside of the bottle with a paper towel to remove gross contamination.

3.2 PERSONNEL DECONTAMINATION

Review the project Health and Safety Plan for the appropriate decontamination procedures.

1. For the purposes of this Standard Operating Procedure, HPLC-grade water is considered equivalent to "deionized ultra filtered water," "reagent-grade distilled water," and "deionized organic-free water." The end product being water which is pure with no spurious ions or organics to contaminate the sample. The method of generation is left to the individual contractor.
2. For the purposes of this Standard Operating Procedure, the term "reagent grade alcohol" refers to either pesticide grade isopropanol or reagent grade methanol.

3.3 EQUIPMENT DECONTAMINATION

3.3.1 Water Samplers

3.3.1.1 Bailers

After each use, polytetrafluoroethelyne (PTFE) double check valve bailers used for groundwater sampling will be decontaminated as follows:

- Discard all ropes used in sampling in properly marked sealable container, or as directed by the Health and Safety Plan. NOTE: No tubing is to be used in conjunction with a bailer in collecting samples.
- Scrub the bailer to remove gross (visible) contamination, using appropriate brush(es), approved water, and non-phosphate detergent.
- Rinse off detergent three times with approved water.
- Rinse bailer with reagent grade alcohol.
- Rinse bailer three times with HPLC-grade water.
- Rinse bailer with 0.10N nitric acid solution.
- Rinse bailer three times with HPLC-grade water.
- Allow bailer to air dry.[3]
- Wrap bailer in aluminum foil or clean plastic sheeting, or store in a clean, dedicated polyvinyl chloride or PTFE storage container.
- Dispose of used decontamination solutions with drummed purge water.
- Rinse bailer with HPLC-grade water immediately prior to re-use.

3.3.1.2 Pumps

Submersible pumps will be decontaminated as follows:

3. If the bailer has just been used for purging and is being decontaminated prior to sampling, do not air dry. Double rinse with HPLC-grade water and proceed to collect samples.

- Scrub the exterior of the pump to remove gross (visible) contamination, using appropriate brush(es), approved water, and non-phosphate detergent. (Steam cleaning may be substituted for detergent scrub.)

- Calculate the volume of pump plus any tubing which is not disposable and not dedicated to a single well. Pump three volumes of non-phosphate laboratory detergent solution to purge and clean the interior of the pump.

- Rinse by pumping no less than nine volumes of approved water to rinse.

- Rinse pump exterior with reagent grade alcohol.

- Rinse pump exterior with HPLC-grade water.

- Allow pump to air dry.

- Wrap pump in aluminum foil or clean plastic sheeting, or store in a clean, dedicated polyvinyl chloride or PTFE storage container.

- Prior to reusing pump rinse exterior again with HPLC-grade water. (Double rinse in Bullet 5 above may be substituted for this step).

3.3.1.3 Dip Samplers

All dip samplers, whether bucket, long-handled, or short-handled, will be decontaminated in the same manner as provided in Section 3.3.1.1.

3.3.1.4 Labware

Labware, such as beakers, which are used to hold samples for field measurements, water chemistry, etc. will be decontaminated according to the procedures in Section 3.3.1.1.

3.3.1.5 Water Level Indicators

Electric water level indicators, weighted measuring tapes, or piezometers used in the determination of water levels, well depths, and/or non-aqueous phase liquid levels will be decontaminated in accordance with Section 3.3.1.1. Clean laboratory wipes may be substituted for brushes. Tapes, probes, and piezometers should be wiped dry with clean laboratory wipes, and coiled on spools or clean plastic sheeting rather than allowed to air dry.

3.3.2 Solid Materials Samplers

Solid materials samplers include soil sampling probes, augers, trowels, shovels, sludge samplers, and sediment samplers, which will be decontaminated as follows:

- Scrub the sampler to remove gross (visible) contamination, using appropriate brush(es), approved water, and non-phosphate laboratory detergent.
- Rinse off detergent with approved water.
- Rinse sampler with reagent grade alcohol.
- Rinse sampler with HPLC-grade water.
- For non-metallic samplers only, rinse sampler with 0.10N nitric acid solution.
- For non-metallic samplers only, rinse sampler with HPLC-grade water.
- Allow sampler to air dry.
- Wrap sampler in aluminum foil clean plastic sheeting, or store in a new zipseal bag (size permitting) or clean, dedicated polyvinyl chloride or PTFE storage container.
- Dispose used decontamination solutions properly according to the site-specific Health and Safety Plan.
- Rinse sampler with HPLC-grade water immediately prior to re-use.

3.3.3 Other Sampling and Measurement Probes

Soil gas sampling probes will be decontaminated as solids sampling devices.

Temperature, pH, conductivity, redox, and dissolved oxygen probes will be decontaminated according to manufacturer's specifications. If no such specifications exist, remove gross contaminant and triple rinse probe with HPLC-grade water. A summary of the decontamination procedures to be used must be included in the instrument-specific standard operating procedure.

Measuring tapes that become contaminated through contact with soil during field use will be decontaminated as follows:

- Wipe tape with a clean cloth or laboratory wipe that has been soaked with non-phosphate laboratory detergent solution to remove gross contamination. Rinse cloth in the solution and continue wiping until tape is clean.
- Wipe tape with a second clean, wet cloth (or laboratory wipe) to remove soap residues.
- Dry tape with a third cloth (or laboratory wipe) and rewind into case, or re-coil tape.

3.3.4 Drilling Rigs and Other Heavy Equipment

All drilling rigs and associated equipment such as augers, drill casing, rods, samplers, tools, recirculation tank, and water tank (inside and out) will be decontaminated prior to site entry after over-the-road mobilization and immediately upon departure from a site after drilling a hole. Supplementary cleaning will be performed prior to site entry when there is a likelihood that contamination has accumulated on tires and as spatter or dust enroute from one site to the next.

- Place contaminated equipment in an enclosure designed to contain all decontamination residues (water, sludge, etc.).
- Steam clean equipment until all dirt, mud, grease, asphaltic, bituminous, or other encrusting coating materials (with the exception of manufacturer-applied paint) have been removed.
- Water used will be taken from an approved source.
- Containerize in 55-gal drums; sample; characterize; and, based on sample results, dispose of all decontamination residues properly.

Other heavy equipment includes use of backhoes, excavators, skid steers, etc. If heavy equipment is utilized during field activities, i.e., a backhoe for test pitting, the bucket should not come in contact with soil to be sampled. If the bucket contacts the soil to be sampled, then it should be decontaminated between sample locations, following the same procedures as listed above for a drill rig.

3.3.5 High Performance Liquid Chromatography-Grade Water Storage

Dedicated glass storage containers will be used solely for dispensing HPLC-grade water. New HPLC-grade water containers will be decontaminated as follows:

- Clean with tap water from approved source and non-phosphate laboratory detergent while scrubbing the exterior and interior of the container with a stiff-bristled brush.
- Rinse thoroughly with approved water.
- Rinse with 0.01N nitric acid.
- Rinse with approved water.
- Rinse thoroughly with HPLC-grade water.
- Fill clean container with HPLC-grade water. Cap with one layer of PTFE-lined paper and one layer of aluminum foil. Secure cap with rubber band and date the container.

Used HPLC-grade water containers will be decontaminated as follows:

- Clean the exterior with tap water from an approved source, non-phosphate laboratory detergent, and a stiff-bristled brush.
- Rinse the exterior thoroughly with HPLC-grade water.
- Rinse the interior twice with pesticide-grade isopropanol.
- Rinse interior thoroughly with HPLC-grade water.
- Fill clean container with HPLC-grade water. Cap with one layer of PTFE-lined paper and one layer of aluminum foil. Secure cap with rubber band and date the container.

3.3.6 Ice Chests and Reusable Shipping Containers

- Scrub exterior/interior with approved brush and liquinox detergent.
- Rinse off detergent three times with approved water.
- Let air dry and properly store until re-use.

NOTE: If container/ice chest is severely contaminated, clean as thoroughly as possible, render unusable, and properly dispose.

4. MAINTENANCE

HPLC-grade water will be stored only in decontaminated glass containers with aluminum foil lids as stipulated above. The water may not be stored for more than nor used more than 3 days after manufacture.

HPLC-grade water will be manufactured onsite. An approved tap water source will be used as the influent to the system. Procedures for system setup, operation, and maintenance will conform to manufacturer's specifications.

5. PRECAUTIONS

Dispose of all wash water, rinse water, rinsates, and other sampling wastes (tubing, plastic sheeting, etc.) in properly marked, sealable containers, or as directed by the Health and Safety Plan.

Once a piece of equipment has been decontaminated, be careful to keep it in such condition until needed.

Do not eat, smoke, or drink onsite.

6. REFERENCES

Site-specific Health and Safety Plan.

Standard Operating Procedure No. 010
for
Water Level and
Well Depth Measurements

Prepared by

EA Engineering, Science, and Technology, Inc.
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Revision 0
August 2007

CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for measuring water level and well depth. This procedure is applicable to the sampling of monitoring wells and must be performed prior to any activities which may disturb the water level, such as purging or aquifer testing.

2. MATERIALS

The following materials may be required:

Electric water level indicator (dipmeter) with cable measured at 0.01-ft increments OR weighted steel tape and chalk OR transducer and datalogger
Oil/water interface probe
Plastic sheeting
Photoionization detector or intrinsically safe flame ionization detector

3. PROCEDURE

3.1 PRELIMINARY STEPS

Locate the well and verify its position on the site map. Record whether positive identification was obtained, including the well number and any identifying marks or codes contained on the well casing or protective casing. Gain access to the top of the well casing.

Locate the permanent reference mark at the top of the casing. This reference point will be scribed, notched, or otherwise noted on the top of the casing. If no such marks are present, measure to the top of the highest point of the well casing and so note this fact in the field logbook. Determine from the records and record in the notebook the elevation of this point.

Record any observations and remarks regarding the completion characteristics and well condition, such as evidence of cracked casing or surface seals, security of the well (locked cap), and evidence of tampering.

Keep all equipment and supplies protected from gross contamination; use clean plastic sheeting. Keep the water level indicator probe in its protective case when not in use.

3.2 OPERATION

Sample the air in the well head for gross organic vapors by lifting the well cap only high enough for an organic vapor meter (photoionization detector or flame ionization detector) probe to be entered into the well casing. This will indicate the presence of gross volatile contaminants as well as indicating potential sampler exposure.

Remove cap. Allow well to vent for 60-90 seconds. Resample headspace. Record both readings. If the second reading is lower than the first, use the second reading to determining whether respiratory protection will be required during subsequent water level and well depth determinations, and sampling.

Note that all headspace sampling must be performed at arm's length and from the upwind side of the well if possible.

Refer to SOP Nos. 011, 023, or 024 as appropriate.

If non-aqueous phase liquid (NAPL) contamination is suspected[1], use an interface probe to determine the existence and thickness of NAPLs.

Open the probe housing, turn the probe on, and test the alarm. Slowly lower the probe into the well until the alarm sounds. A continuous alarm indicates a NAPL while an intermittent alarm indicates water. If a NAPL is detected, record the initial level (first alarm). Mark the spot by grasping the cable with the thumb and forefingers at the top of the casing. If a mark is present on the casing, use the mark as the reference point. If no mark is present, use the highest point on the casing as the reference point. Withdraw the cable sufficiently to record the depth.

Continue to slowly lower the probe until it passes into the water phase. Slowly retract the probe until the NAPL alarm sounds and record that level in the manner as described above.

Record the thickness of the light NAPL[2] (Section 3.3).

Continue to slowly lower the interface probe through the water column to check for the presence of dense NAPL.

Measure and record the thickness of the dense NAPL layer (if any) as described above.

Slowly raise the interface probe, recording the depth to each interface as the probe is withdrawn. If there is a discrepancy in depths, clean the probe sensors and re-check the depths.

NOTE: Air/liquid interface depth is more reliable if probe is lowered into liquid. NAPL/water depths are more accurate if probe is moved from water into NAPL.

Always lower and raise interface probe slowly to prevent undue mixing of media.

1. Interface probes will be used in all wells for first round sampling, regardless of site history. If no NAPLs are detected during the first round of sampling, this step may be omitted during subsequent sampling events **unless** conditions such as site history or headspace vapors would indicate otherwise.
2. If NAPL is viscous, such as coal tar or weather bunker oil, several confirmation measurements should be made after decontamination of the probe to verify that the NAPL is not sticking to the probe and causing erroneous readings. One way to accomplish this would be to partially fill a 5-gal bucket with water and dip the probe to ensure that decontamination has effectively removed the NAPL.

Always perform NAPL check in wells installed in areas with suspected NAPL contamination. Always perform NAPL check if headspace test reveals presence of volatiles. Always perform NAPL check the first time a well is sampled. **If** a well has been sampled previously **and** no NAPLs were present **and** none of the preceding conditions are met, the NAPL check may be omitted.

If no NAPL is present, use an electronic water level detector as follows:

- Remove the water level indicator probe from the case, turn on the sounder, and test check the battery and sensitivity scale by pushing the red button. Adjust the sensitivity scale until you can hear the buzzer.

- Slowly lower the probe and cable into the well, allowing the cable reel to unwind. Continue lowering until the meter buzzes. Very slowly, raise and lower the probe until the point is reached where the meter **just** buzzes. Marking the spot by grasping the cable with the thumb and forefingers at the top of the casing. If a mark is present on the casing, use the mark as the reference point. If no mark is present, use the highest point on the casing as the reference point. Withdraw the cable and record the depth.

Alternatively use a steel tape with an attached weight if aquifer gradients are lower than 0.05 ft/ft. Due to the possibility of adding unknown contaminants from chalk colorants, only white chalk is permitted.

Rub chalk onto the first 1 ft of the steel tape and slowly lower the chalked end into the well until the weighted end is below the water surface. (A small splash can be heard when the weighted end hits the water surface.)

Using the method described above read and record the length from the steel tape.

Remove the steel tape. The chalk will be wet or absent where the tape was below the water surface. Locate, read, and record this length. Subtract wetted length from total length and record the difference. This is the depth to water table.

Transducers and dataloggers will be used where water level fluctuations over time are to be measured, such as tidal fluctuation studies (SOP No. 045) and aquifer (hydraulic) tests (SOP No. 033). Note that transducers are inappropriate for measuring well depth.

Slowly lower the transducer into the well until it is below the lowest possible piezometric level (typically 2-3 ft below the water table).

Tape the umbilical to the protective casing to prevent the transducer from falling further.

Attach the umbilical leads to the datalogger.

Turn datalogger on.

To measure the well depth, lower electric water level indicator probe or tape until slack is noted. Very slowly raise and lower the cable until the exact bottom of the well is "felt." Measure (cable) or read the length (tape) and record the depth.

Note that if the electric water level indicator is used to determine depth of well, the offset distance between the tip of the probe and the electrode must be added to the reading to determine actual depth.

Withdraw the probe or tape. Decontaminate the probe(s) and cable(s).

3.3 DATA RECORDING AND MANIPULATION

Record the following computations:

- Date and time
- Weather
- Method of measurement
- Casing elevation
- NAPL surface elevation = casing elevation – depth to NAPL
- NAPL thickness = depth to bottom of NAPL – depth to top of NAPL
- Water level elevation = casing elevation – depth to water
- Well bottom elevation = casing elevation – depth to bottom (or read directly from tape).

Refer to SOP Nos. 005 and 016.

4. CALIBRATION

No calibration is needed.

5. PRECAUTIONS

Depending upon the device used, correction factors may be required for some measurements. Check instrument batteries prior to each use. Exercise care not to break the seals at the top of the electric water level indicator probe.

6. REFERENCES

McAlary, T.A. and J.F. Barker. 1987. Volatilization Losses of Organics during Groundwater Sampling from Low Permeability Materials in Groundwater Monitoring Review. Fall.

Thornhill, J.T. 1989. Accuracy of Depth to Groundwater Measurements in U.S. Environmental Protection Agency Superfund Groundwater Issue EPA/540/4-89/002.

Standard Operating Procedure No. 011
for
Photoionization Detector

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Revision 0
August 2007

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for field operations with the photoionization detector (MiniRae). The photoionization detector uses an ultraviolet emitting lamp designed to detect, measure, and display the total concentration of airborne ionizable gases and vapors. This information is used to determine control measures such as protection and action levels.

Use of brand names in this SOP is not intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor will provide applicable and comparable SOPs for the maintenance and calibration of same.

2. MATERIALS

The following materials may be required:

Battery pack	Tedlar bag
Calibration gas (100 ppm isobutylene)	Tygon tubing
Microtip/MiniRae	Regulator

3. STARTUP/CALIBRATION PROCEDURE

Turn the instrument on by pressing the back of the power switch located on the handle of the instrument.

The message “Warming up now, please wait” will be displayed for up to 3 minutes. After normal display appears, the instrument is ready for calibration.

Fill a Tedlar bag with the desired calibration gas (usually 100 ppm Isobutylene).

Press SETUP button and select the desired Cal Memory using the arrow keys (normally set to 200 ppm). Press EXIT button to leave setup function.

Press CAL button and expose instrument to Zero Gas. (Usually clean outdoor air will be suitable. If any doubt exists as to the cleanliness of the background air a commercial source of zero gas should be used.)

The instrument then asks for the Span Gas concentration. Enter the known span gas concentration and then connect the Tedlar bag containing the Span Gas.

NOTE: The span gas concentration is dependent upon both the concentration of the span gas used and the rating of the UV lamp in the instrument at time of calibration. If using 100 ppm isobutylene and the standard 10.6 eV lamp, the span gas concentration will be 56 ppm.

Press enter and the instrument sets its sensitivity. Once the display reverts to normal, the instrument is calibrated and ready for use. Remove the Span Gas from the inlet probe. The instrument should be calibrated at least once a day.

4. BATTERY CHARGING

Ensure instrument is off. Set the voltage selector switch on the bottom of the battery charger to the appropriate AC line voltage. Press the release button on the bottom of the instrument and remove the battery pack by sliding it backwards. Plug charger into the battery pack and then into an AC outlet and allow the battery to charge for at least 8 hours. After charging, remove the charger, first from the outlet then from the battery pack, and slide the battery pack back onto the instrument.

5. PRECAUTIONS

Instrument does not carry an Intrinsic Safety Rating and must not be used in a hazardous location where flammable concentrations of gases or vapors are constantly present.

All calibration, maintenance, and servicing of this device, including battery charging, must be performed in a safe area away from hazardous locations.

Do not open or mutilate battery cells. Do not defeat proper polarity orientation between the battery pack and battery charger. Substitution of components may affect safety rating.

6. REFERENCES

Instrument User's Manual.

Standard Operating Procedure No. 013
for
Collection of Monitoring Well Samples

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for the collection of groundwater samples from monitoring wells.

2. MATERIALS

The following materials may be required:

0.45 µM filters	Polyvinyl chloride bailer (for purging only)
Bladder pump (dedicated to one well only)	Sample bottles and labels
Conductivity meter	Stainless steel bailer (for purging and sampling)
Dissolved oxygen meter	Submersible pump and hose (for purging only)
Generator	Thermometer (optional)[1]
Logbook or book of field parameter forms	Transparent bailer with a double check valve
Peristaltic pump with tubing for filtering samples	Turbidity meter
pH meter with oxidation-reduction potential probe	Tygon tubing
Photoionization detector organic vapor analyzer.	Variable speed, low flow submersible pump (e.g., Grundfos MP1 groundwater sampling pump) (for purging and sampling)
Plastic sheeting	Water level indicator
Polypropylene rope	
Polytetrafluoroethelyne (PTFE) bailer with PTFE-coated stainless steel cable, double check valve top, and controlled flow bottom discharge attachment[2] for volatile organic compound (VOC) sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles) (for purging and sampling)	

3. PROCEDURE

3.1 GENERAL

Groundwater sampling will follow these general steps:

- Arrive onsite
- Set up apparatus (generators, pumps, etc.)
- Glove
- Organic vapor check, water level, and well depth measurements

1. Temperature compensation and measurement capabilities are generally available as integral functions of pH meters and conductivity meters. If this is the case, a separate thermometer is not required.
2. Although use of a controlled flow bottom discharge valve is historically preferred, use of such a device can cause aeration of the sample.

- Sample non-aqueous phase liquids (NAPLs) (as required)

- Begin purge procedure

  — If using bailer to purge and sample, see Section 3.6
  — If using pump to purge and bailer to sample, see Section 3.7
  — If using bladder or low-flow pump to purge and sample, see Section 3.8

- Decontaminate/reglove

- Take samples

  — If with bailer, see Section 3.6
  — If with bladder or low flow pumps, see Section 3.8

- Decontaminate/dispose of wastes, move equipment to next site.

3.2 GENERAL RULES FOR GROUNDWATER FIELD PARAMETER LOGBOOK

Only one site or installation per logbook, and only one sampling location per page or form (if using pre-printed forms). The same book may be used for more than one sampling event. First five pages will be reserved for index, general notes, etc. Sign and date each entry. Last five pages will be reserved for recording calibration data for the pH, temperature, turbidity, oxidation-reduction potential, dissolved oxygen, and conductivity meters. Use the page number or a separately recorded "Cal Reference Number" to refer to each calibration. As appropriate, insert the cardboard flap under the form being filled out, so that writing does not go through to the pages below. As appropriate, fill in the forms from front to back of the logbook, tearing out the white copy for each sample when the sample has been collected. This copy goes in the cooler with the sample, directly to the laboratory. The original copy must be torn out before you write on the back of the duplicate form. As appropriate, duplicate copies, index pages, and calibration sheets remain intact.

3.3 GROUNDWATER SAMPLING GENERAL RULES

Groundwater samples will be collected from the least contaminated wells first, progressing to the most contaminated[3]. Upon arrival at the well site, immediately set up and organize the purging, sampling, and filtration equipment. If needed, due to muddy or contaminated ground, remoteness from sampling vehicle, and\or for placement of hose(s) and\or power cord if a pump is used, place clean plastic sheeting at, or around the well, to serve as a clean staging area for purging and sampling equipment, as conditions warrant. Care must be exercised not to step on plastic sheeting. If the well is remote from the sampling vehicle, set up the filtration equipment

3. First round samples are to be collected from upgradient wells first, moving to downgradient wells under the assumption that upgradient wells will be less contaminated than downgradient wells. Results of first round analysis may mandate a change in sampling sequence.

and place rope, wrapped bailer, and pre-labeled sample containers on the plastic sheet, from the well. When a pump is to be used, situate the portable generator on level ground approximately 15 ft away from and downwind from the well. All generator maintenance (oil and fueling) is to be performed offsite. If the hose(s) and/or power cord of the pump are not on a reel, place the pump with its hose and power cord on the plastic sheeting downhill from the well.

Check well headspace for organic vapor which may pose a health and safety hazard and indicate the presence of NAPL. Measure depth(s) to and thickness(es) of NAPL(s) as appropriate. Measure the depth to water and depth of well. From the water depth, well diameter, sand pack length, etc., calculate the equivalent volume (1 EV) of water in the well.

1 EV = volume in casing + volume in saturated sand pack. Therefore, if the water table lies below the top of the sandpack, use the following equation:

$$1\ EV = (\pi R_w^2 h_w) + (0.30\pi(R_s^2 - R_w^2)h_w) * (0.0043)$$

If the water table lies above the top of the sandpack use this equation:

$$1\ EV = [(\pi R_w^2 h_w) + (0.30\pi(R_s^2 - R_w^2)h_s)] * (0.0043)$$

where

R_s = Radius of sandpack in inches
R_w = Radius of well casing in inches
h_s = Height of sandpack in inches
h_w = Water depth in inches

0.0043 gal/in.^{3}
Assumed filter pack porosity = 30 percent.

Samples will always be collected in order of decreasing volatility (i.e., the samples to be analyzed for the volatile constituents should be collected first). Deliver the VOC sample to the vial by allowing the water to trickle down the inside wall of the vial at a rate no greater than approximately 100 ml/min. Other samples may be delivered at a faster rate. Sampling rates will at no time exceed 1 L/min. Procedures for each class of samples are contained in the site-specific Quality Assurance Project Plan.

When collecting samples for volatile analysis, care should be taken to prevent analyte loss by volatilization. The following procedures should be adhered to when collecting these samples:

- Avoid excessive aeration and agitation of sample.
- Fill vial so that a reverse meniscus is present by adjusting the flow rate from the sampling device.

- Place septum on vial so that the PTFE side is in contact with the sample. After the cap is on the bottle, check for air bubbles in the sample. If air bubbles are present, properly dispose of that sample and recollect the sample in the same vial.

- Make sure vial is labeled and immediately transfer the vial to the cooler with ice.

Filtered and unfiltered samples will be taken for inorganics (metals) analyses. The samples will be filtered through an in-line 0.45-µM filter (preferred method), or by gravity through a 0.45-µM membrane placed in a filter funnel. Use forceps to place the membrane into the funnel and pour sample through funnel until appropriate volumes have been filtered.

If necessary, due to slow filtering, a peristaltic pump may be used to filter the sample through an in-line filter. Connect the pump to the generator, attach tygon tubing to the bottom discharge valve on the bailer. Start pump and collect sample from the end of the in-line filter directly into the proper container, preserved, and placed in the cooler. Filtered samples will be preserved in the field with acid to a pH of less than 2. Make sure sample bottle is labeled and the cap is on tightly. Then place in cooler with ice immediately.

— OR —

If a low flow pump is used collect the samples, filtered samples will be taken by installing a 0.45-µM filter in-line and pumping the water through the filter. Collect sample from the end of the in-line filter directly into the proper container, preserved, and placed in the cooler. Filtered samples will be preserved in the field with acid to a pH of less than 2. Make sure sample bottle is labeled and the cap is on tightly. Then place in cooler with ice immediately.

Unfiltered samples will be collected by slowly pouring the sample water into the appropriate sample container, being careful not to agitate or cause bubbles to form. Do not overfill bottles. Make sure sample bottle is labeled and the cap is on tightly, then place the sample in cooler with ice immediately.

All samples will be delivered to the laboratory as soon as possible. If possible, samples will be shipped on the same day as they are collected. If samples must be retained due to weekend sampling (Friday through Sunday), the laboratory will be notified as to the time sensitive nature of the samples.

3.4 SAMPLING OF NON-AQUEOUS PHASE LIQUIDS

If NAPLs are detected in the well, a sample from all layers must be collected prior to any purging activities. NAPLs may be indicated by the presence of volatiles in the well headspace, and confirmed by the oil/water interface probe.

Collecting light non-aqueous phase liquid (LNAPL) will be accomplished using a transparent bailer with a double check valve. This bailer will be slowly lowered until the bottom of the bailer is 1-2 in. below the LNAPL-water interface, then slowly withdrawn. Verify that the interface was sampled by visual inspection of the bailer contents through the side of the bailer. Measure the thickness of the LNAPL in the bailer and note in the Field Logbook. Sample for laboratory analysis. An additional field verification may be performed by decanting the remainder of the contents of the bailer into a glass jar, adding a hydrophobic dye such as Sudan IV, or Redoil, shaking the sample and looking for coloration of NAPL. Alternate field tests are: examine the sample under ultraviolet light (many fluoresce), or allow the sample to stand overnight, and examine for interface and/or volatiles in the headspace the following day. Refer to following sections on purging and sample collection for setup and general operation.

Collecting dense non-aqueous phase liquids (DNAPLs) will be accomplished using a transparent bailer with a double check valve. The bailer must be lowered very slowly to the bottom of the well and raised slowly out of the well in a controlled fashion. Sample for analysis as above. The same field check described above may be employed for DNAPL. Refer to following sections on purging and sample collection for set up and general operation.

If NAPLs are present in the well, **and** a low-flow pump is to be used for purging and sampling, the well will be allowed to re-equilibrate prior to purging and sampling. This will be accomplished by allowing the well to stand undisturbed for at least 8 hours prior to purging and sample collection.

3.5 WELL PURGING GENERAL RULES

Water within the casing of a well will stagnate, degas, lose volatiles, possibly precipitate metals due to changes in redox potential, and may react with the screen and/or casing material. It is, therefore, necessary to purge a sufficient volume of this stagnant water from the well and/or casing to ensure that a representative sample of formation water can be obtained. Traditionally, the volume of water to be purged was arbitrarily set at 3-5 equivalent volumes. Recent advances in sampling technologies have caused a re-thinking of such arbitrary purge volumes. It is for this reason that monitoring of select chemical and physical properties of the sample medium will be used instead of strict volumes to determine when a representative sample may be taken from a well.

Acceptable purge/sampling devices include: bailers, high-discharge submersible pumps (purge only), and variable speed, low-flow pumps which include both submersible pumps (purge and sample) and dedicated bladder pumps (purge and sampling). It is recommended to purge and sample at similar rates with one type device per well. An acceptable exception to this general rule is to use a high-discharge submersible pump to purge a deep, fast-recharging well, and a bailer to sample the same well.

Peristaltic, gas-lift, and centrifugal pumps can cause volatilization, produce high pressure differentials, and can result in variability in the analysis of some analytes of interest. These types of pumps will not be used to purge or sample wells.

To prevent groundwater from cascading down the sides of the screen into an open hole, thereby aerating the sample, purge rates will closely match recharge rates. If the static water level is within the casing, the initial purge rates may be set high enough to lower the water level to the top of the screen, then reduced to maintain that level.

Purging will be accomplished with either a submersible pump, a low-flow (submersible or bladder) pump, or bailer. The choice of bailer or pump will be based on depth to water table, volume to be purged, and permeability of the aquifer. If the well recharges rapidly and/or has greater than 20 gal (estimated EV) to be purged, water may be removed with a submersible pump or a low-flow pump. If the well recharges slowly and/or has less than 20 gal to be purged, water will be removed with a bailer or a low-flow pump.

Purging will be accomplished with as minimal disturbance to the surrounding formation as possible.

Purge water will be containerized onsite until analysis of samples is completed. Based on sample results, accumulated purge water will be properly disposed.

If the water level is within the screened interval and the well recharge rate is less than 0.1 L/min, purge the well using a low-flow pump as follows:

1. Draw the water down to within 1 ft of the top of the pump.

2. Allow the well to recover.

3. Check and record field parameters.

4. Repeat Steps 1 through 3 then collect samples for metals analysis only[4].

5. Note the event in the Field Logbook, and report the problem to the Project Manager. If this extremely low recharge problem consistently occurs in a given well, the well may be considered for re-development and/or replacement.

6. If adjacent wells have elevated VOC levels, additional soil gas surveys will be considered in the vicinity of the low recharge well to help determine the need for replacement.

3.6 PURGING AND SAMPLING WITH BAILERS

Bailers may be used for both purging and sampling wells if: (a) the well recharge rate is less than 4 L/min, (b) depth to the water table is less than 50 ft, and (c) less than 20 gal are to be purged (5 EV < 20 gal)[5].

4. Analyte losses due to volatilization in a drained well are too high for valid VOC sampling (McAlary and Barker 1987).

When purging with a bailer, either a polyvinyl chloride, PTFE, or stainless steel bailer may be used. The bailer will be attached to either a spool of PTFE-coated stainless steel cable or polypropylene rope. If using cable, attach it to the bailer using stainless steel cable clamps. Thoroughly decontaminate the cable after each use, prior to rewinding cable onto spool. Cable clamps and raw cable ends may serve to trap contamination. Exercise particular caution in decontaminating these areas. If using rope, attach the rope to the bailer using a bowline knot, dispense the needed length (a few feet more than the well depth) and cut the remainder away; then, at the end opposite the bailer, make a slip knot and place it around the well casing or protective posts to prevent losing the bailer and rope down the well. The polypropylene rope will be not reused; it will be properly disposed of. Either type of bailer will be repeatedly lowered gently into the well until it fills with water, removed, and the water will be discharged into an appropriate container until purging is complete. Care must be taken not to unduly agitate the water, as this tends to aerate the sample, increase turbidity, makes stabilization of required parameters difficult to achieve, and generally prolongs purging.

After purging 2 EV, obtain a sample of groundwater and measure the following stabilization parameters: temperature, conductivity, pH, turbidity, redox potential (Eh), and dissolved oxygen level at each successive half-well volume. When three of these stabilization parameters are in agreement within approximately 10 percent in three consecutive half-well volume samples, sufficient water has been purged from the well. The results of these tests should be recorded in the sampling logbook. Should these parameters not reach agreement, no more than five well volumes will be purged.

Immediately upon completion of purging, collect samples for laboratory analysis using a PTFE bailer on a PTFE-coated stainless steel cable. The bailer will be equipped with double check valve top and controlled flow bottom discharge attachments for VOC sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles).

Slowly, so as not to agitate the water, lower the bailer into the well, using a spool of PTFE-coated cable. Allow bailer to fill, withdraw smoothly. Refill bailer as needed.

If the controlled flow bottom discharge attachment is used for VOC sampling, attach it to the bottom of the bailer. Using the stopcock valve on the bailer to control the flow, fill sample vials as described above in Section 3.3.

Remove check valve top and pour unfiltered sample into inorganics sample bottles.

Collect filtered samples as described in Section 3.3. Decontaminate bailer and cable.

5. These numbers are based on the following assumptions: (1) In purging, it is preferable to remove water at approximately the recharge rate; (2) 4 L/min is estimated as the approximate maximum rate at which water can be removed with a bailer from depths of 20-50 ft; and (3) 20 gal is estimated to be at the limit of the sampler's endurance, at which point fatigue and sloppiness of technique begin.

3.7 PURGING WITH PUMP, SAMPLING WITH BAILER

If the recharge rate of the well is greater than 30 L/min, or the water level is deeper than 50 ft, or more than 20 gal of purge water will be generated (5 EV > 20 gal), then purging and sampling may be accomplished using a submersible pump/bailer combination.

When purging with a pump, gradually lower the intake until it is submerged within the screened interval. Lower an electronic water level probe to the top of the screen (as determined from completion records) to the monitor water level, start pump, and slowly lower the pump as the water level continues to fall. Care should be exercised to lower the water column to the top of the screened interval (water level probe will stop beeping) but not below the top of the screen if possible. This will ensure that the stagnant layer has been removed, but should minimize the detrimental effects of over pumping the well. Secure hose(s) and/or power cord to casing and place discharge hose into the proper container, downhill and as far away from the well as possible. Determine and record the discharge rate.

Discharge rate = volume of container/time to fill container

The discharge rate will be established at approximately equal to or just greater than the well's recharge rate (determined from well development). If well development records are incomplete, recharge rate can be determined by monitoring the rise/fall of the water level within the casing as one purges the well. If the water level is static at a given pumping rate, but fluctuates up or down as pumping rate is decreased or increased, the pumping rate at which the water level is static is the recharge rate.

After purging 2 EV, obtain a sample of groundwater and measure the following stabilization parameters: temperature, conductivity, pH, turbidity, redox potential (Eh), and dissolved oxygen level at each successive half-well volume. When three of these stabilization parameters are in agreement within approximately 10 percent in three consecutive half-well volume samples, sufficient water has been purged from the well. The results of these tests should be recorded in the sampling logbook. Should these parameters not reach agreement, no more than five well volumes will be purged.

Immediately upon completion of purging, collect samples for laboratory analysis using a PTFE bailer on a PTFE-coated stainless steel cable. The bailer will be equipped with double check valve top and controlled flow bottom discharge attachments for VOC sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles). Filtration of metals samples will be accomplished using either an in-line filter attached to the bottom of the bailer, or a funnel and appropriate filter (Section 3.3).

Slowly, so as not to agitate the water, lower the bailer into the well, using a spool of PTFE-coated cable. Allow bailer to fill, withdraw smoothly, fill sample containers as described in Section 3.6. Decontaminate bailer and cable in and decontaminate pump.

3.8 PURGING AND SAMPLING WITH LOW-FLOW PUMP

To obtain representative samples, subsurface disturbances should be kept to a minimum, thereby preventing sample alteration due to sampling actions. The reasoning behind the use of low-flow pumps to purge and sample monitoring wells is that these pumps minimize physical disturbance (turbulence) at the sampling point and chemical changes (aeration) in the medium. For these reasons, the low-flow pump is the preferred method for both purging and sampling in most cases. For the purposes of this SOP, "low-flow pumps" are defined as either dedicated bladder pumps or variable speed submersible pumps. Practical operational flow rates for these sampling devices range from 0.1 L/min to 30 L/min.

Low-flow pumps may be used for purging and sampling any well having recharge greater than 0.1 L/min, which is the practical lower limit of pump performance. Below that pumping rate, pump inefficiencies and/or overheating may alter the physical and chemical properties of the sample. If the pump is continuously operated at sampling rates higher than the well recharge rate, the water level will be lowered in the well, possibly allowing aeration of the sample which is unacceptable sampling procedure. Low-flow pumps are suitable for sampling wells with recharge rates lower than 0.1 L/min if precautions are taken to avoid aeration of the sample.

Low flow submersible pumps will be used as follows:

- Lower the pump into the well, slowly so as not to agitate the water, until the pump is at the mid-point of the screened interval or the mid-point of the water column if the static water table lies below the top of the screen[6]

- Attach the pump's umbilical cord (which will consist of power cord and sampling tubing) to the protective casing, or lock the cord spool so that the pump cannot move vertically in the well during sampling.

- Lower the water level probe into the well behind the pump until it just touches water. This will allow the sampler to monitor the water level while purging and sampling, and prevent the inadvertent drying of the well.

6. This assumes a 10-ft screened interval. If the screened interval is greater than 10 ft, multiple samples should be taken as follows:
   - If the screen is 10-12 ft, sample the canter of the water column, as outlined above.
   - If the screen is longer than 12 ft, and the water column is 10 ft or less, sample the center of the water column.
   - If the screen is longer than 12 ft, and the water column fills the screen, or extends above the screen, sample at 1/3 and 2/3 the height of the water column, or about every 6 ft.

- Begin purging at the pump's lowest setting, then gradually increase rate[7] until the pumping rate matches the aquifer recharge rate. **If the water level is above the top of the screen**, the pumping rate may be allowed to slightly exceed recharge rate, lowering the water level to no less than 1 ft above the screen, then reduced until it matches recharge rate and purging continued. **If the water level is below the top of the screen**, always keep the purge rate lower than well's recharge rate.

- Monitor stabilization parameters listed in Section 3.6 beginning immediately, using an in-line monitoring system. Record parameters regularly, at a rate of one set of parameters per each 1-3 liters of water removed from the well. When these parameters stabilize to within 10 percent over three consecutive readings, reduce[8] flow rate to 0.1 L/min (if needed) and begin collecting VOC samples directly from the discharge line.

- If the well recharges at a rate less than 0.1 L/min, purge until the water level is even with the top of the screen, allow the well to recover, and sample immediately.

- Remove and decontaminate water level probe and pump.

4. MAINTENANCE

Refer to manufacturer's requirements for maintenance of pumps and generators.

5. PRECAUTIONS

Refer to the site-specific Health and Safety Plan for appropriate personal protective equipment.

6. REFERENCES

Garske, E.E. and M.R. Schock. 1986. An Inexpensive Flow-Through Cell and Measurement System for Monitoring Selected Chemical Parameters in Groundwater.

Gass, T.E., J.F. Barker, R. Dickhout, and J.S. Fyfe. 1991. Test Results of the Grundfos Groundwater Sampling Pump, in Proceedings of the Fifth National Symposium on Aquifer Restoration and Groundwater Monitoring.

7. Some sources indicate that the pumping rate should not exceed 1 L/min, with 0.5 L/min being preferable. The optimal purge rate is highly aquifer dependent, and may range from less than 0.5 L/min to greater than 10 L/min. The purge rate for a given well will, therefore, be a field decision, based on well development, purge, and sampling records rather than SOP mandate.
8. Sampling should occur at the same rate as purging as long as aeration of sample does not occur.

McAlary, T. A. and J.F. Barker. 1987. Volatilization Losses of Organics During Groundwater Sampling From Low Permeability Materials, in Groundwater Monitoring Review. Fall.

Puls, R.W. and R.M. Powell. 1992. Acquisition of Representative Groundwater Quality Samples for Metals, in Groundwater Monitoring Review. Summer.

Puls, R.W., J.H. Eychaner, and R.M. Powell. 1990. Colloidal-Facilitated Transport of Organic Contaminants in Groundwater: Part I. Sampling Considerations, in EPA Environmental Research Brief. EPA/600/M-90/023. December.

Puls, R.W., R.M. Powell, D.A. Clark, and C.J. Paul. 1991. Facilitated Transport of Inorganic Contaminants in Groundwater: Part II Colloidal Transport, in EPA Environmental Research Brief. EPA/600/M-91/040. July.

Puls, R.W., R.M. Powell, B. Bledsoe, D.A. Clark, and C.J. Paul. 1992. Metals in Groundwater: Sampling Artifacts and Reproducibility, in Hazardous Waste & Hazardous Materials. Volume 9, No. 2.

Standard Operating Procedure No. 015
for
Document Control System

Prepared by

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August 2007

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for identifying and storing a complete set of documents relating to project tasks. Each document will receive a unique identification number comprised of elements describing the document.

2. MATERIALS

Not applicable.

3. PROCEDURE

Each project-related document will be given to the Document Control Officer. The Document Control Officer will record information for each document on a Document Control Sheet which will be retained as a backup record. The information from each Document Control Sheet will be maintained in a computer database.

The individual Document Control Number will be entered on the Document Log Sheet and will be written on the document.

The storage location for each document will be recorded on the Document Control Log Sheet and the documents will be stored in the recorded location.

The database file will be backed up on a regular basis to prevent accidental loss of the data.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

None.

Standard Operating Procedure No. 016
for
Surface Water, Groundwater, and Soil/Sediment Field Logbooks

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Revision 0
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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for recording surface water, groundwater, soil/sediment sampling information, instrument calibration data, and data from hydrologic testing in the field logbooks. Acceptable field logbooks are: bound, unprinted books such as a surveyor's field book, or a federal supply service No. 7530-00-222-3525 record book (or equivalent); or they may be company-proprietary, pre-printed forms bound into a field logbook. Example forms are provided herein. Alternate, equivalent forms are acceptable.

2. MATERIALS

The following material may be required: applicable field logbook and indelible ink pen.

3. PROCEDURE

Information pertinent to soil/sediment, groundwater, or surface water sampling will be recorded in the appropriate logbook. Each page/form of the logbook will be consecutively numbered. Entries will be made in indelible ink. Corrections will consist of line-out deletions that are initialed and dated. If using carbon paper or self-duplicating forms, before entering data in logbook, insert a sheet protector between form sets to isolate first blank form from remaining forms.

3.1 SOIL/SEDIMENT LOGBOOK (Requires Figures SOP016-1 and SOP016-3)

3.1.1 Field Parameter Form (Items on Figures SOP016-1 and SOP016-2)

1. HIGH CONCENTRATION EXPECTED?: Answer "Yes" or "No."

2. HIGH HAZARD?: Answer "Yes" or "No."

3. SITE: Record the complete name of the site.

4. AREA: Record the area designation of the sample site.

5. INST CODE: Record the 2-letter installation code appropriate for the installation or site. Correct abbreviations can be found on Pages 3-6 of the IRDMS User's Guide for chemical data entry.

6. FILE NAME: Record "CSO" for a soil sample or "CSE" for a sediment sample.

7. SITE TYPE: Record the abbreviation appropriate for where the sample was taken. Correct abbreviations can be found on Pages 18-21 of the IRDMS User's Guide for chemical data entry. This entry must match the Site Type on the map file form.

8. SITE ID: Record a code up to 10 characters or numbers which is unique to the site.

9. FIELD SAMPLE NUMBER: Record a code specific for the sample.

10. DATE: Enter the date the sample was taken.

11. TIME: Enter the time (12-hour or 24-hour clock acceptable as long as internally consistent) the sample was taken.

12. AM PM: Circle "AM" or "PM" to designate morning or afternoon (12-hour clock).

13. SAMPLE PROG: Record "GQA" (Groundwater Quality Assessment) or other appropriate sample program.

14. DEPTH (TOP): Record the total depth sampled.

15. DEPTH INTERVAL: Record the intervals at which the plug will be sampled.

16. UNITS: Record the units of depth (feet, meters)

17. SAMPLE MEASUREMENTS: Check the appropriate sampling method.

18. CHK: Check off each container released to a laboratory.

19. ANALYSIS: Record the type of analysis to be performed on each sample container.

20. SAMPLE CONTAINER: Record the sample container type and size.

21. NO.: Record the number of containers.

22. REMARKS: Record any remarks about the sample

23. TOTAL NUMBER OF CONTAINERS FOR SAMPLE: Record the total number of containers.

24. SITE DESCRIPTION: Describe the location where the sample was collected.

25. SAMPLE FORM: Record the form of the sample (i.e., clay, loam, etc.) using The Unified Soil Classification System.

26. COLOR: Record the color of the sample as determined from standard Munsell Color Charts.

27. ODOR: Record the odor of the sample or "none." See SOP No. 001 Section 5.

28. PID (HNu): Record the measured PID (HNu) values.

29. UNUSUAL FEATURES: Record anything unusual about the site or sample.

30. WEATHER/TEMPERATURE: Record the weather and temperature.

31. SAMPLER: Record your name.

3.1.2 Map File Form (Figure SOP016-3)

1. The map file logbook form will be located on the reverse of the field parameter logbook form, or on an adjoining page of the field logbook (if level book is used).

2. SITE ID: Record the Site ID from the field parameter form.

3. POINTER: Record the field sample number for the sample being pointed to.

4. DESCRIPTION/MEASUREMENTS: Describe the location where the sample was taken, along with distances to landmarks.

5. SKETCH/DIMENSIONS: Diagram the surroundings and record the distances to landmarks.

6. MAP REFERENCE: Record which U.S. Geological Survey Quad Map references the site.

7. COORDINATE DEFINITION: Write the compass directions the X- and Y-Coordinates of the map run.

8. COORDINATE SYSTEM: Write "UTM" (Universal Transverse Mercator).

9. SOURCE: Record the 1-digit code representing the Map Reference.

10. ACCURACY: Give units (e.g., write "1-M" for 1 meter).

11. X-COORDINATE: Record the X-Coordinate of the sample site location.

12. Y-COORDINATE: Record the Y-Coordinate of the sample site location.

13. UNITS: Record the units map sections are measured in.

14. ELEVATION REFERENCE: Record whether topography was determined from a map or a topographical survey.

15. ELEVATION SOURCE: Record the 1-digit code representing the elevation reference.

16. ACCURACY: Record the accuracy of the map or survey providing the topographical information.

17. ELEVATION: Record the elevation of the sampling site.

18. UNITS: Write the units in which the elevation is recorded.

19. SAMPLER: Write your name.

3.2 SURFACE WATER LOGBOOK (Requires Figures SOP016-2 and SOP016-3)

3.2.1 Field Parameter Form (Items Unique to Figure SOP016-3)

1. CAL REF: Record the calibration reference for the pH meter.

2. pH: Record the pH of the sample.

3. TEMP: Record the temperature of the sample in degrees Celsius.

4. COND: Record the conductivity of the water.

5. For all other sections, see Section 3.2.1.

3.3 GROUNDWATER SAMPLING LOGBOOK (Requires Figures SOP016-2, SOP016-3, and SOP016-4)

3.3.1 Field Parameter Form (Items on Figure SOP016-4)

1. WELL NO. OR ID: Record the abbreviation appropriate for where the sample was taken. Correct abbreviations can be found on Pages 18-21 of the IRDMS User's Guide for chemical data entry.

2. SAMPLE NO.: Record the reference number of the sample.

3. WELL/SITE DESCRIPTION: Describe the location where the sample was taken, along with distances to landmarks.

4. X-COORD and Y-COORD: Record the survey coordinates for the sampling site.

5. ELEV: Record the elevation where the sample was taken.

6. UNITS: Record the units the elevation was recorded in.

7. DATE: Record the date in the form MM/DD/YY.

8. TIME: Record the time, including a designation of AM or PM.

9. AIR TEMP.: Record the air temperature, including a designation of C or F (Celsius or Fahrenheit).

10. WELL DEPTH: Record the depth of the well in feet and inches.

11. CASING HT.: Record the height of the casing in feet and inches.

12. WATER DEPTH: Record the depth (underground) of the water in feet and inches.

13. WELL DIAMETER: Record the diameter of the well in inches.

14. WATER COLUMN HEIGHT: Record the height of the water column in feet and inches.

15. SANDPACK DIAM.: Record the diameter of the sandpack. Generally, this will be the same as the bore diameter.

16. EQUIVALENT VOLUME OF STANDING WATER: Use one of the following equations, to determine one equivalent volume (EV):

    1 EV = Volume in casing + volume in saturated sand pack. Or to restate:

    $1\ EV = (BR_w^2 h_w + 0.30B(R_s^2 - R_w^2)h_s) * (0.0043)$

    where
    R_s = Radius of sandpack in inches
    R_w = Radius of well casing in inches
    h_s = Height of sandpack in inches
    h_w = Water depth in inches

    $0.0043 = gal/in.^3$
    and filter pack porosity is assumed as 30 percent

    — OR —

    Volume in casing = $(0.0043\ gal/in.^3)(B)(12\ in./ft)(R_c^2)(W_h)$

    where
    R_c = Radius of casing in inches
    W_h = Water column height in feet

    Vol. in sandpack = $(0.0043\ gal/in.^3)(B)(12\ in./ft)(R_b^2 - R_c^2)(W_h)(0.30)$

    (if W_h is less than the length of the sandpack),

— PLUS —

Vol. in sandpack = $(0.0043 \text{ gal/in.}^3)(B)(12 \text{ in./ft})(R_b^2 - R_c^2)(S_h)(0.30)$

(if W_h is greater than the length of the sandpack).

where
R_b = Radius of the borehole
S_h = Length of the sandpack.

Show this calculation in the comments section.

17. VOLUME OF BAILER OR PUMP RATE: Record bailer volume or pump rate.

18. TOTAL NUMBER OF BAILERS OR PUMP TIME: Record the number of bailers required to remove 3 equivalent volumes (EV) of water from the well or the total purge time and volume as applicable.

19. WELL WENT DRY? Write "YES" OR "NO."

20. NUMBER OF BAILERS OR PUMP TIME: Record the number of bailers or pump time which made the well go dry.

21. VOLUME REMOVED: Record the volume of water (gal) removed before the well went dry.

22. RECOVERY TIME: Record the time required for the well to refill.

23. PURGE AGAIN?: Answer "YES" or "NO."

24. TOTAL VOL. REMOVED: Record the total volume of water (in gal) removed from the well.

25. CAL REF.: Record the calibration reference for the pH meter.

26. TIME: Record time started (INITIAL T[0]), 2 times DURING the sampling and the time sampling ended (FINAL).

27. pH: Record the pH at start of sampling (INITIAL), twice DURING the sampling and at the end of sampling (FINAL).

28. TEMP: Record the water temperature (Celsius) at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

29. COND: Record the conductivity of the water at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

30. D.O.: Record the dissolved oxygen level in the water at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

31. TURBIDITY: Record the readings from the turbidity meter (nephelometer) and units at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

32. ORD: Record the oxidation/reduction (RedOx) potential of the water sample at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

33. HEAD SPACE: Record any positive readings from organic vapor meter reading taken in well headspace prior to sampling.

34. NAPL: Record the presence and thickness of any non-aqueous phase liquids (light or dense)

35. COMMENTS: Record any pertinent information not already covered in the form.

36. SIGNATURE: Sign the form.

3.4 FIELD CALIBRATION FORMS (Maintained as a separate logbook, or incorporated into sampling logbooks)

3.4.1 Items on Figure SOP016-5

1. Record time and date of calibration. Note whether 12- or 24-hour clock was used.

2. Record calibration standard reference number.

3. Record meter I.D. number

4. Record initial instrument reading, recalibration reading (if necessary), and final calibration reading on appropriate line.

5. Record value of reference standard (as required).

6. COMMENTS: Record any pertinent information not already covered on form.

7. SIGNATURE: Sign form.

3.5 GROUNDWATER HYDROLOGY TESTS LOGBOOK (Must include Figures SOP016-6 and SOP016-7 and/or SOP016-8, OR SOP016-9 or SOP016-10)

3.5.1 Field Permeability Test Data Sheet (Items on Figures SOP016-6)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

4. PROJECT NO.: Record the contractor assigned project number or the contract number.

5. LOCATION: Specific location

6. CLIENT: Agency or company with the contract under which the work is being performed.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

9. TEST TYPE: Short description of the type of test to be performed.

10. RISING/FALLING HEAD WITH SLUG: Check if the test involved the insertion/removal of and inert object.

11. RISING/FALLING HEAD WITHOUT SLUG: Check if the test involved the addition/removal of a quantity of water.

12. START DATE: Date on which the test was begun.

13. CLOCK TIME: Time each datum (depth to groundwater level) is collected. Note whether 12- or 24-hour clock was used.

14. ELAPSED TIME: Time since the last datum was collected.

15. DEPTH TO GWL (ft): Depth to the top of the groundwater table (Groundwater Level) as measured by manual methods.

16. REC. (ft): Water level as reported by transducer/datalogger (this is the depth of water above the transducer.

17. TIME: Time the discharge rate check was begun (addition or removal of water method). Note whether 12- or 24-hour clock was used.

18. FLOW METER (Addition or removal of water method): The amount of water added or removed as registered by the flowmeter, in gal of liters.

19. DISCHARGE RATE: Flowmeter reading divided by time interval (gal/min or liters/min).

20. SIGNATURE: The person completing this form must sign the form at the end of the test.

21. DATE: Date the form was signed.

3.5.2 Groundwater Levels – Single Well (Items on Figure SOP016-7)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

Well Data

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: Remedial investigation wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

15. DATE: Date of each water level reading

16. TIME: Time of each water level reading. Note whether 12- or 24-hour clock was used.

17. ELAPSED TIME: Time since test was begun.

18. DEPTH TO WATER: Measured depth to the groundwater table.

19. WATER ELEVATION: Elevation of the top of the groundwater table (use datum listed above).

20. MEAS. METH.: Method used to measure the water level in the well (see abbreviation key at the bottom of the data sheet).

21. TAPE NO.: The unique identification number of the traceable standard tape used to calibrate the measuring device.

22. WELL STATUS: Condition of the well at the time of measuring (see abbreviation key at the bottom of the data sheet).

23. REMARKS: Any additional pertinent comments not specifically required above.

24. INITIALS: Initials of person completing this data entry.

25. ABBREVIATION KEYS: Self explanatory.

26. SIGNATURE: The person completing this form must sign the form at the end of the test.

27. DATE: Date the form was signed.

3.5.3 Groundwater Levels – Single Well (Items on Figure SOP016-8)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

WELL DATA

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: All Rhode Island wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

15. DATALOGGER: This section is record of pertinent datalogger information.

16. MANUFACTURER: Record the manufacturer/brand name as stated on the datalogger.

17. MODEL: Enter the model number of the datalogger.

18. S/N: Enter the serial number of this datalogger.

19. TAG PROGRAMMED IN LOGGER: What is the identifier used in the datalogger's program to indicate that this unit was used to record a given data set?

20. TRANSDUCER: This section is a listing of pertinent information about the transducer used.

21. MANUFACTURER: Record the manufacturer/brand name as stated on the transducer.

22. MODEL: Enter the model number of the transducer.

23. S/N: Enter the serial number of this transducer.

24. INPUT/UNITS: What are the units this transducer uses?

25. RANGE: Record the pressure or depth range over which this transducer is certified.

CALIBRATION

26. PRESSURE RATING: This is taken from the manufacturer's specifications for a given transducer. (Usually in psi, or kpa).

27. "SUBMERGENCE = ___ (V) / (MV)": Record the voltage returned by the transducer at a given depth of submergence. Indicate whether the reading is in volts (v), or millivolts (mv).

28. VOLUME WATER ADDED/REMOVED: (Applicable if inert object insertion/removal method was not employed.) Record the volume of water added to or removed from the well.

29. DISCHARGE RATE: If z (above) is filled, enter the rate at which this water was added or removed.

30. INITIAL WATER LEVEL (ft): Enter the water level in the well at the beginning of the test.

31. PRESSURE TRANSDUCER SUBMERGENCE: Record the depth to which the transducer is submerged at the beginning of the test and the depth to the transducer at the end if the test. All depths will be recorded to the nearest 0.01 ft.

32. TIME: Record the time the test is begun and ended. Note whether 12- or 24-hour clock was used.

33. OBSERVED CHANGES IN ADJACENT WELLS: Note any changes in water levels in nearby wells.

34. RESULTS RECORDED ON DISKETTE #: Tracking number of the diskette on which these data are archived.

35. DISKETTE FILE NAME: Name of the file(s).

36. SIGNATURE: The person completing this form must sign the form at the end of the test

37. DATE: Date the form was signed.

3.6 GROUNDWATER LEVELS – MULTIPLE WELLS (Items on Figure SOP016-9)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

5. LOCATION: Specific location.

6. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

7. CLIENT: Agency with the contract under which the work is being performed.

8. REMARKS: Any pertinent observations not specifically required above.

9. WELL: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

10. DATE: Date this measurement was made.

11. TIME: Time this measurement was made. Note whether 12- or 24-hour clock was used.

12. DEPTH TO WATER: Depth from MP to top of groundwater table.

13. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

14. MP ELEV.: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

15. MEAS. METH.: Method used to measure the water level in the well (see abbreviation key at the bottom of the data sheet).

16. REMARKS/MP: Describe the location and nature of the measuring point.

17. INITIALS: Initials of the person completing this form.

18. ABBREVIATION KEYS: Self explanatory.

19. SIGNATURE: The person completing this form must sign the form at the end of the test.

20. DATE: Date the form was signed.

3.7 GROUNDWATER LEVELS – DATALOGGERS (Items on Figure SOP016-10)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

WELL DATA

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: All Rhode Island wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019, Section 3.4).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

DATALOGGER (This section is a record of pertinent datalogger information)

15. MANUFACTURER: Record the manufacturer/brand name as stated on the datalogger.

16. MODEL: Enter the model number of the datalogger.

17. S/N: Enter the serial number of this datalogger.

18. TAG PROGRAMMED IN LOGGER: What is the identifier used in the datalogger's program to indicate that this unit was used to record a given data set?

TRANSDUCER (This section is a listing of pertinent information about the transducer used)

19. MANUFACTURER: Record the manufacturer/brand name as stated on the transducer.

20. MODEL: Enter the model number of the transducer.

21. S/N: Enter the serial number of this transducer.

22. INPUT/UNITS: What are the units this transducer uses?

23. RANGE: Record the pressure or depth range over which this transducer is certified.

CALIBRATION

24. PRESSURE RATING: This is taken from the manufacturer's specifications for a given transducer (usually in psi, or kpa).

25. "SUBMERGENCE = ___ (V) / (MV)": Record the voltage returned by the transducer at a given depth of submergence. Indicate whether the reading is in volts (v), or millivolts (mv).

26. DATE: Date of each water level reading

27. TIME: Time of each water level reading. Note whether 12- or 24-hour clock was used.

28. LOGGING TIME INTERVAL: Time since test was begun.

29. WL FEET BELOW MP: Measured depth to the groundwater table from measuring point.

30. SUBMERGENCE: Depth of water above the transducer.

31. MEAS.METHOD: What device/method was used to measure the water level.

32. TAPE NO.: Record the tape identification number.

33. TRANSDUCER MOVED?: Was the transducer moved since the last water level reading?

34. REMARKS: Any pertinent remarks not otherwise specified.

35. INITIALS:

DATA TRANSFER TO DISKETTE:

36. DATE: Date data were archived onto diskette.

37. TIME: Time stamp the computer assigns the data file.

38. FILE NAME: Name assigned the data file.

39. SOFTWARE USED FOR TRANSFER: Any special software, or computer operating system used to write the files to diskette. NOTE: If a "shareware" archiver which compresses files was used, and the archived file is not self-extracting, a copy of the unarchive program should be copied onto the diskette also.

40. OUTPUT FORMAT: What is the format of the output file? (DOS, UNIX, Binary, Compressed?)

41. INITIALS: Initials of the person who copied the data to diskette.

42. ABBREVIATION KEY: Self-explanatory.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

U.S. Environmental Protection Agency. 1984. User's Guide to the Contract Laboratory Program. July.

FIGURE SOP016-1
FIELD PARAMETER LOGBOOK
SOIL AND SEDIMENT SAMPLES

HIGH CONCENTRATION EXPECTED? HIGH HAZARD?

INSTALLATION/SITE _____ AREA _____

INST CODE FILE NAME _____

SITE TYPE SITE ID
FIELD SAMPLE NUMBER

DATE (MM/DD/YY) / / TIME AM PM SAMPLE PROG.

DEPTH (TOP) DEPTH INTERVAL UNIT _____

SAMPLING METHOD:

SPLIT SPOON AUGER SHELBY TUBE SCOOP OTHER

CHK	ANALYSIS	SAMPLE CONTAINER	NO.	REMARKS

TOTAL NUMBER OF CONTAINERS FOR SAMPLE

DESCRIPTION OF SITE AND SAMPLE CONDITIONS
SITE DESCRIPTION:_____
SAMPLE FORM _____ COLOR _____ ODOR _____
PID (HNu) _____ UNUSUAL FEATURES _____
_____
WEATHER/TEMPERATURE
SAMPLER

HIGH CONCENTRATION EXPECTED? HIGH HAZARD?

FIGURE SOP016-2
FIELD PARAMETER LOGBOOK
GROUNDWATER AND SURFACE WATER SAMPLES

INSTALLATION/SITE AREA
INST CODE FILE NAME SITE TYPE
SITE ID FIELD SAMPLE NUMBER
DATE (MM/DD/YY) / / TIME AM PM SAMPLE PROG.
DEPTH (TOP) DEPTH INTERVAL UNITS

SAMPLING MEASUREMENTS

CAL REF.	pH	TEMPERATURE C	CONDUCTIVITY	OTHER

CHK	ANALYSIS	SAMPLE CONTAINER	NO.	REMARKS

TOTAL NUMBER OF CONTAINERS FOR SAMPLE

DESCRIPTION OF SITE AND SAMPLE CONDITIONS
SITE DESCRIPTION
SAMPLING METHOD
SAMPLE FORM COLOR ODOR
PID (HNu)
UNUSUAL FEATURES
WEATHER/TEMPERATURE _____ SAMPLER _____

FIGURE SOP016-3
MAP FILE LOGBOOK

SITE ID POINTER _____
DESCRIPTION/MEASUREMENTS
SKETCH/DIMENSIONS:

MAP REFERENCE
COORDINATE DEFINITION (X is Y is)
COORDINATE SYSTEM SOURCE ACCURACY
X-COORDINATE Y-COORDINATE UNITS
ELEVATION REFERENCE
ELEVATION SOURCE ACCURACY ELEVATION
UNITS

SAMPLER

FIGURE SOP016-4
MAP FILE AND PURGING LOGBOOK
GROUNDWATER SAMPLES

WELL COORD. OR ID SAMPLE NO._____
WELL/SITE
DESCRIPTION_____
_____
X-COORD. Y-COORD. _____ ELEV. UNITS
DATE ____/____/____ TIME AIR TEMP.

WELL DEPTH _____ ft _____ in. CASING HT. _____ ft _____ in.
WATER DEPTH _____ ft _____ in. WELL DIAMETER _____ in.
WATER COLUMN HEIGHT _____ ft _____ in. SANDPACK DIAM. _____ in.
EQUIVALENT VOLUME OF STANDING WATER _____ (gal) (L)
VOLUME OF BAILER _____ (gal) (L) or PUMP RATE _____ (gpm) (lpm)
TOTAL NO. OF BAILERS (5 EV)_____ or PUMP TIME _____ MIN.
WELL WENT DRY? [Yes] [No] NUM. OF BAILERS _____ or PUMP TIME_____MIN
VOL. REMOVED _____ (gal) (L) RECOVERY TIME _____ MIN
PURGE AGAIN? [Yes] [No] TOTAL VOL. REMOVED _____ (gal) (L)

Date and Time	Quantity Removed	Time Required	pH	Cond	Temp	ORD	Turb	DO	Character of water (color/ clarity/odor/partic.)
(before)									
(during)									
(during)									
(during									
(after)									

COMMENTS:_____
_____
_____
_____

_____
SIGNATURE

FIGURE SOP016-5
FIELD CALIBRATION: pH, CONDUCTIVITY, TEMPERATURE, TURBIDITY, OXIDATION-REDUCTION POTENTIAL, AND DISSOLVED OXYGEN METERS

INITIAL CALIBRATION	FINAL CALIBRATION
DATE:	DATE:
TIME:	TIME:

pH METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

pH STANDARD	INITIAL READING	RECALIB. READING	FINAL READING
7.0			
10.0			
4.0			

CONDUCTIVITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

COND. STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

TEMPERATURE METER CALIBRATION

METER ID _____

TEMP. STANDARD	INITIAL READING	RECALIB. READING	FINAL READING
ICE WATER			
BOILING WATER			
OTHER			

FIGURE SOP016-5 *(continued)*

TURBIDITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

ORD METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

DISSOLVED OXYGEN METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

COMMENTS:_____

_____

_____

_____

_____

SIGNATURE

FIGURE SOP016-6
FIELD PERMEABILITY TEST DATA SHEET

Contractor: **Seq. #** /

Project Name:	Project #:
Location:	Client:
Party Chief:	Contractor:
Observation Well:	
Test Type: Rising/Falling Head w/Slug	Rising/Falling Head w/out Slug
Start Date:	Discharge Rate

Clock Time	Elapsed Time (min)	Depth to GWL (ft)	Rec (ft)	Clock Time	Elapsed Time	Depth to GWL (ft)	Rec (ft)	Time	Flow Meter	Discharge Rate

Signature: _____ **Date:** _____

FIGURE SOP016-7
GROUNDWATER LEVELS – SINGLE WELL

Contractor: **Seq. #** /

Project No.:
Project Name:
Field Party Chief:

WELL DATA:

Stickup: (ft)
MP Elevation:
Well No.: Site: Area:
Site: Area:
Area:

up (+)/down (-) from: Datum = MSL or:
Datum = MSL or:

Measuring Point Description:

Datalogger:
Manufacturer: Model: S/N:
Tag No. Programmed in Logger:

Transducer: Manufacturer: Model: S/N:
Input/Units: Range:

Calibration:
Pressure Rating:
0 ft submergence = (v) / (mv) ft submergence = (v) / (mv)

Volume Water Added/Removed:
Discharge Rate:
Initial Water Level (ft):

Pressure Transducer Submergence
Initial (ft): Final(ft): Time:Start: End:
Observed Changes in Adjacent Wells:

Results Recorded on Diskette #:
Diskette File Name:

Signature: _____ Date: _____

FIGURE SOP016-8
GROUNDWATER LEVELS – MULTIPLE WELLS

Contractor: **Seq. #** /

Project No.:
Project Name:
Field Party Chief:

WELL DATA:

Stickup: (ft)
MP Elevation:

Measuring Point Description:
Remarks:
Well No.:
Site: Area: up (+)/down (-) from:
up (+)/down (-) from:
up (+)/down (-) from:
Datum = MSL or:

Date	Time	Elapsed Time	Depth to Water	Water Elevation	Meas. Meth.	Tape No.	Well Status	Remarks	Initials

Measurement Method:
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Well Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently pumped
NP = Nearby well pumping
NRP = Nearby well recently
X = Obstructed

Signature: _____ Date: _____

FIGURE SOP016-8 *(continued)*

Contractor: **Seq. #** /

Project No.:
Project Name: Location:
Field Party Chief: Client:

Well	Date	Time	Depth to Water	Stickup	MP Elev.	Meas. Meth.	Tape No.	Remarks/MP	Initials

Measurement Method:
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Well Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently pumped
NP = Nearby well pumping
NRP = Nearby well recently
X = Obstructed

FIGURE SOP016-9
GROUNDWATER LEVELS DATALOGGERS

Contractor
Project No.: **Well No.:**
Project Name: **Site:**
Field Party Chief: **Area:**

WELL DATA: Stickup: (**ft**) **up (+)/down (-) from:**
MP Elevation: **Datum = MSL or:**

Measuring Point Description:
Remarks:

Datalogger:
Manufacturer: Model: S/N:
Tag No. Programmed in Logger:
Transducer: Manufacturer: Model: S/N:
Input/Units: Range:
Calibration: Pressure Rating:
0 ft submergence = (v) / (mv) ft submergence = (v)

Logging	Date	Time	Logging Time Interval	WL, ft Below MP	Submergence (logger reading)	Meas. Method	Tape No.	Well Status	Transducer Moved	Remarks	Initials
Start											
Stop											
Start											
Stop											

Data Transfer to Disk

Date	Time	File Name	Software Used for Transfer	Output Format	Initials

Measurement Method:
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Well Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently
NP = Nearby well pumping
NRP = Nearby well recently pumped
X = Obstructed

Signature **Date**

Standard Operating Procedure No. 019
for
Monitoring Well Installation

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CONTENTS

1. SCOPE AND APPLICATION

The installation of monitoring wells is contingent upon the existing conditions at the project site. The purpose of this Standard Operating Procedure is to delineate the quality control measures required to ensure the accurate installation of monitoring wells. The applicable Work Plan should be consulted for specific installation instructions. The term "monitoring wells," as used herein, is defined to denote any environmental sampling well. An example well log form is provided in Appendix A. Alternate, equivalent forms are acceptable.

2. MATERIALS

2.1 DRILLING EQUIPMENT

The following drilling equipment may be required:

- Appropriately sized drill adequately equipped with augers, bits, drill stem, etc.
- Steam cleaner and water obtained from approved source for decontaminating drilling equipment.
- Photoionization Detector: Microtip HL-200 (or equivalent)
- Water level indicator
- Weighted steel tape measure
- Lower explosive limit – oxygen monitor
- Steel drums for intrusion derived wastes (drill cuttings, contaminated personal protective equipment, decontamination solutions, etc.)
- Source of approved water
- Heavy plastic sheeting
- Sorbent pads and/or log.

2.2 WELL INSTALLATION MATERIALS[1]

The following well installation materials may be required:

- Well screen:[2]
  - — Polyvinyl chloride (PVC): JOHNSON (or equivalent); PVC 0.010 slot; Schedule 40; flush-threaded (leak-proof) joints; PVC complies with American Society for Testing and Materials (ASTM) D2665, ASTM D1784, and ASTM F480; free of ink markings; cleaned and prepackaged by manufacturer.
  - — Stainless steel: JOHNSON (or equivalent); stainless steel 0.010 slot; 304 stainless steel[3]; ASTM F480 flush threads; cleaned, wrapped, and heat sealed by manufacturer.
- Riser pipe:
  - — PVC: JOHNSON (or equivalent); STD; PVC; Schedule 40; flush-threaded (leak-proof) joints; PVC complies with ASTM D2665, ASTM D1784, and ASTM F480; free of ink markings; cleaned and prepackaged by manufacturer.
  - — Stainless steel: JOHNSON (or equivalent); Schedule 5; 304 stainless steel; ASTM Type A312 material; 4-in. diameter; cleaned, wrapped, and heat sealed by manufacturer.
- Plugs/caps: JOHNSON (or equivalent); standard PVC or stainless steel.
- Filter pack: MORIE, 100 well gravel (or equivalent). NOTE: Final gradation may vary as a function of the gradation of the formation.[2]
- Fine Ottawa sand.
- Bentonite seal: BAROID, bentonite pellets (3/8-in. diameter)
- Cement: Type II Portland Cement (table below).

1. Technical information on all installed materials (screens, riser pipe, filter pack, bentonite, cement, etc.) and representative samples of the proposed filter pack, bentonite powder, and bentonite pellets will be supplied to the Project Manager.
2. Well screen slot size and filter pack gradation will be determined from sieve analysis of aquifer materials. Screen and casing material type will be determined based on field tests of groundwater chemistry and contaminants.
3. Unless the sum of Cl-, F-, and Br- is >1,000 ppm, in which case Type 316 should be used.

Cement Type	Special Characteristics	Recommended Usage
I	No special properties	General use as grout mix or cement plug (if sulfates <250 ppm), surface pad.
IA	Air-entraining Type I (Note that air entrainment properties can be achieved by chemical admixtures)	Air entrainment gives cement greater freeze-thaw resistance. Recommended for surface pads.
II	Moderate sulfate resistance, low heat of hydration	General use as grout mix or cement plug where groundwater sulfate >250 ppm and <1,500 ppm, surface pad.
IIA	Air-entraining Type II	See Type IA.
III	High early strength, high heat of hydration	Elevated temperature can damage well casing and fracture grout/cement plugs. NOT RECOMMENDED.
IIIA	Air-entraining Type III	NOT RECOMMENDED.
IV	Low heat of hydration	General use as grout mix or cement plug preferred type for well abandonment to ensure intact grout/cement plug.
V	High Sulfate resistance	Use when groundwater sulfate levels >1,500 ppm.

- Bentonite powder: BAROID, Aquagel Gold Seal.
- Steel protective casing: BRAINARD-KILMAN (or equivalent) zinc-plated steel, lockable, painted.[4]
- Geotextile: MIRAFI (or equivalent); GTF 130; non-woven; 4 oz.
- Coarse (blanket) gravel: Crushed stone aggregate.
- Containers for purged water, as required.
- Submersible pump or bailer of appropriate capacity, and surge block sized to fit well.
- Hach DREL 2000 portable laboratory (or equivalent).
- Conductivity, pH, oxidation-reduction potential (ORP), turbidity, dissolved oxygen, and temperature meters.
- Electric well sounder and measuring tape.
- Portland Type II cement (see previous table).
- Steel Posts (pickets), painted (see footnote).

4. All painted components (protector casing, steel pickets) will be painted high-visibility orange and allowed to dry completely prior to being brought onsite.

2.3 DOCUMENTATION

The following document may be provided:

- Copy of appropriate Work Plan
- Copy of approved Health and Safety Plan
- Copies of well and excavation permits
- Boring log forms
- Well completion diagram form
- Well development form.

2.4 GEOLOGIST'S PERSONAL EQUIPMENT

The following equipment may be required for the geologist:

- 10X handlens
- Unified Soil classification System chart
- Munsell color chart
- Sieve set (Keck model SS-81 or equivalent)
- Personal protective equipment as required by the Health and Safety Plan.

3. PROCEDURE

3.1 MATERIALS APPROVAL

Water sources for drilling, grouting, sealing, filter placement, well installation, and equipment decontamination must be approved by the Project Manager prior to arrival of the drilling equipment. Information required for the water source includes: water source, manufacturer/ owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analyses over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives (bentonite) will be the only drilling fluid additive allowed, and its use must be approved by the Project Manager prior to the arrival of the drilling equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product description, and intended use for the product.

Granular Filter Pack material must be approved by the Project Manager prior to drilling. A 1-pint representative sample must be supplied to the Project Manager. Information required includes: lithology, grain size distribution, brand name, source, processing method, and slot size of intended screen.

Portland Type II cement will be used for grout (see previous table).

3.2 DRILLING

The objective of the selected drilling technique is to ensure that the drilling method provides representative data while minimizing subsurface contamination, cross-contamination of aquifers, and drilling costs. The preferred drilling method is with a hollow-stem auger. Other drilling methods[5] are approved as conditions warrant, and will not require variances be issued by the U.S. Environmental Protection Agency. The method used at a specific site will be proposed in the work plan and evaluated by the Project Manager. Any drilling method not listed herein will require approval on a case by case basis by the U.S. Environmental Protection Agency.

A Site Geologist will be present during all well drilling and installation activities and will fully characterize all tasks performed in support of these activities into the monitoring well logbook. The Site Geologist will be responsible at only one operating rig for the logging of samples, monitoring of drilling operations, recording of water losses/gains and groundwater data, preparing the boring logs and well diagrams, and recording the well installation procedures of the rig. The Site Geologist will have onsite sufficient equipment in operable condition to perform efficiently his/her duties as outlined in the contractual documents. Items in the possession of each Site Geologist will include the approved Health and Safety Plan, this Standard Operating Procedure, a hand lens (10X), a standard color chart, grain-size chart, and a weighted (with steel or iron) steel tape long enough to measure the deepest well, heavy enough to reach that depth, and small enough to fit readily within the annulus between the well and drill casing. The Site Geologist will also have onsite, a water level measuring device, preferably electrical.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole drilling equipment. Additives containing either lead or copper will not be allowed. In addition, polychlorinated biphenyls will not be permitted in hydraulic fluids or other fluids used in the drilling rig, pumps, and field equipment/vehicles.

5. If the design depth of the well is <100 ft, open, hollow-stem augers will be used to drill the well unless "running sands" preclude the use of open augers. In that case, an inert "knockout" plug may be used in the bottom of the auger string. This plug will be driven out of the augers and left at the bottom of the hole when the well is installed.

   If the design depth of the well is >100 ft, rotary drilling methods may be used to install wells. The following drill fluids and methods are approved in the order listed: (1) rotary drilling with water from an approved source as drilling fluid (clays from the formations will tend thicken the fluid and coat the walls of the borehole and this is acceptable); (2) rotary drilling with water as a fluid, advancing a temporary casing with the bit to maintain an open hole; and (3) mud rotary using water with additives as drill fluid. Due to the potential for aquifer contamination and plugging, mud rotary drilling is not recommended for monitoring wells. If, however, "running sands" are encountered and the aquifer is expected to have a relatively high flow rate, then mud rotary is considered an approved method. Pure sodium bentonite is the only approved additive. Mud rotary drilling must be halted at the last aquitard above the target aquifer. Casing must be set, all bentonite-bearing fluids flushed from the hole and drill rig, and drilling may be resumed using water only as the drill fluid until the target depth is reached.

Surface runoff or other fluids will not be allowed to enter any boring or well during or after drilling/construction.

Antifreeze used to keep equipment from freezing will not contain rust inhibitors and sealants. Antifreeze is prohibited in areas in contact with drilling fluid. The ground surface at the well site will be protected from possible coolant, fuel, and hydraulic fluid spills and/or leakage by placement of plastic sheeting with raised edges, draining into a lined catch basin large enough to contain spills and/or leakage from motors, radiators, or vehicle tanks. Sorbent pillows will be placed to catch obvious leaks from the drill rig. Sorbent logs may be used instead of, or in conjunction with, a lined catch basin to contain spills.

An accurate measurement of the water level will be made upon encountering water in the borehole and later upon stabilization. Levels will be periodically checked throughout the course of drilling. Any unusual change in the water level in the hole, such as a sudden rise of a few inches may indicate artesian pressure in a confined aquifer, will be the basis for cessation of drilling. The geologist will immediately contact the Project Manager[6]. Particular attention for such water level changes will be given after penetrating any clay or silt bed, regardless of thickness, which has the potential to act as a confining layer.

Anticipated depths of wells are given in well specific work plans. In case the previously defined criteria have not been met before the depth range for a given hole is reached, the geologist will stop the drilling and confer with the Project Manager. The current boring conditions (depth, nature of the stratigraphic unit, and water table depth) will be compared to those of other wells nearby to decide to continue drilling or to terminate and complete the well.

If the well is to be installed in the surficial aquifer, drilling will be terminated before penetrating the basal aquitard. The basal aquitard is defined as the first 2 ft-thick clay below the water table, or below 5 ft in the case of a shallow aquifer.

If the well is to be installed in a lower, confined aquifer:

- Penetrations of aquifers located lower than the water table aquifer will be limited to avoid cross-contamination.

- Placement of new upper confined aquifer wells will be initially limited to those areas where contamination has been confirmed.

- The location of upper confined aquifer wells will be based upon the findings of the water table aquifer investigation. Areas of known contamination will be targeted for installing upper confined aquifer wells for the purposes of delineating vertical contamination.

6. The contract technical oversight will also be contacted for guidance.

- Where possible, upper-confined aquifer wells will be located such that they afford triangulation with other wells within the same aquifer to allow for a determination of groundwater flow direction.

- Some upper-confined aquifer wells will be installed approximately 10-15 ft from water table wells to enable the accurate assessment of vertical hydraulic gradients. If the direction of groundwater flow is known, wells within a group will be located sidegradient of each other.

- The boring will be advanced until the base of the surficial aquifer is reached (Section 3.2).

- An outer, surface casing will be set 2-5 ft into the confining layer to minimize the potential for cross-contamination from the unconfined aquifer during drilling activities.

- The surface casing will be driven into the confining bed and grouted into place. Grout will be tremied into the annulus around the outside of the casing to within 5 ft of the ground surface. A grout plug at least 2 ft thick will be tremied into the bottom of the surface casing. The grout will be permitted to cure for 24 hours. All drilling fluids within the surface calling will then be removed, and the casing will be flushed with clean potable water.

- The drilling equipment will be decontaminated, a smaller bit or auger selected, and the hole will be continued through the grout plug into the confined aquifer.

- If deeper aquifers are to be screened, repeat preceding steps until total depth is reached.

If dense non-aqueous phase liquid (DNAPL) contamination is detected during drilling, the well will be terminated and completed at the base of the aquifer. Drilling will not continue through the confining unit.

Stainless steel screens will be used in DNAPL wells. Screen size selection will be according to criteria set forth in Section 3.4. The formation grain size will be multiplied by the higher factor (6) to determine filter pack grain size. This will ensure that the filter pack is sufficiently coarse to permit DNAPL to pass freely from the formation into the coarser filter pack, then into the open well (Cohen and Mercer 1993).

DNAPL sampling cups are prohibited. The well screen will be capped, and set 0.3 ft (0.5 ft max.) into the top of the confining bed and rest on the bottom of the hole or bentonite backfill (if used). No sand will be placed below the screen. The remainder of the well installation and completion will be accomplished according to Section 3.4.

3.3 LOGGING

All borings for monitoring wells will be logged by a geologist. Logs will be recorded in a field logbook and/or a boring log. If the information is recorded in a logbook, it will be transferred to Boring Log Forms on a daily basis. Field notes are to include, as a minimum:

- Boring number
- Material description (as discussed below)
- Weather conditions
- Evidence of contamination
- Water conditions (including measured water levels)
- Daily drilling footage and quantities (for billing purposes)
- Notations on man-placed materials
- Drilling method and borehole diameter
- Any deviations from established field plans
- Blow counts for standard penetration tests
- Core and split-spoon recoveries.

Material description for soil samples must include:

- Classification
- Unified Soil Classification symbol
- Secondary components and estimated percentages
- Color
- Plasticity
- Consistency
- Density
- Moisture content
- Texture/fabric/bedding and orientation
- Grain angularity
- Depositional environment and formation
- Incidental odors
- Photoionization detector reading(s)
- Staining.

Material description for rock samples must include:

- Classification
- Lithologic characteristics
- Bedding/banding characteristics
- Color
- Hardness
- Degree of cementation
- Texture
- Structure and orientation

- Degree of weathering
- Solution or void conditions
- Primary and secondary permeability
- Sample recovery
- Incidental odors
- Photoionization detector reading(s)
- Staining.

3.4 WELL CONSTRUCTION AND INSTALLATION

After the hole is drilled and logged, backfill hole as required for proper screen placement. The integrity of the aquitard will be restored by placing a bentonite plug of an appropriate thickness, either to the top of the aquitard (normal well installation) or to within 0.3 ft of the top of the aquitard (DNAPL well). Aquifer fill will be clean filter pack.

Normal screen placement for the water table (surficial) aquifer will be within 2 ft of the screen extending above the static water level. The bottom of the screen will rest no more than 6 in. from the bottom of the hole or backfill material, whichever is applicable.

NOTE: The end cap in DNAPL wells will rest on the bottom of the bottom of the hole, or bentonite backfill if applicable (Section 3.2).

Screen placement for a confined aquifer well will normally be at the top of the confined aquifer.

Screen lengths will not normally exceed 10 ft. If it appears advantageous in a given situation (e.g., to screen an entire aquifer which is thicker than 10 ft), approval must be sought on a case-by-case basis from the appropriate regulatory agency. Otherwise, wells will be screened as follows:

Thickness of Aquifer	Action
<10 ft	Screen entire aquifer
>10 ft <30 ft	Screen top 10 ft consider vertically nested well cluster
>30 ft	Install vertically nested well cluster

The installation of monitoring wells in uncased or partially cased holes will begin within 12 hours of completion of drilling, or if the hole is to be logged, within 12 hours of well logging, and within 48 hours for holes fully cased with temporary drill casings. Once installation has begun, work will continue until the well has been grouted and the drill casing has been removed.

Well screens, casings, and fittings will conform to National Sanitation Foundation Standard 14 or ASTM equivalent for potable water usage. These materials will bear the appropriate rating logo. If the logos are not present, a written statement from the manufacturer/supplier stating that the materials contain the appropriate rating must be obtained. Material used will be new and essentially chemically inert to the site environment.

Well screen and casing should be inert with respect to the groundwater; therefore, the selection of screen and casing material will be based on select field tests of aquifer chemistry and potential contaminants. The screen will be capped without sediment trap or DNAPL sampling cup, and lowered into the hole. The well casing will be pre-cut to extend 2-2.5 ft above ground surface. Prior to placement of the last piece of well casing, a notch or other permanent reference point will be cut, filed, or scribed into the top edge of the casing.

Screen slot size will be appropriately sized to retain 90-100 percent of the filter pack material, the size of which will be determined by sieve analysis of formational material (Section 3.4).

The tops of all well casing will be capped with covers composed of materials compatible with the products used in the well installation. Caps may either be vented, or a telescopic fit, constructed to preclude binding to the well casing caused by tightness of fit, unclean surfaces, or weather conditions. In either case, it should be secure enough to preclude the introduction of foreign material into the well, yet allow pressure equalization between the well and the atmosphere.

Filter pack material will be placed, lightly tamped, and leveled. Filter pack will extend from the bottom of the hole to a height of 1-2 ft above the top of the screen. The filter pack will be capped with a minimum of 1 ft of fine (Ottawa) sand to prevent the bentonite seal from infiltrating the filter pack. If the bentonite seal is placed as a slurry, a minimum of 2 ft of fine sand will be required.

If the hole is less than 20-ft deep, the filter pack may be poured into the annulus directly. If the hole is deeper than 20 ft, the filter pack must be tremied into place.

Granular filter packs will be chemically and texturally clean, inert, and siliceous.

Filter pack grain size will be based on formation grain-size analysis. The D30 (70 percent retained) sieve size multiplied by a factor of not less than 3 nor greater than 6 will be used to determine the appropriate grain size.

Calculations regarding filter pack volumes will be entered into the Field Logbook along with any discrepancies between calculated and actual volumes used. If a discrepancy of greater than 10 percent exists between calculated and actual volumes exists, an explanation for the discrepancy will also be entered in the Field Logbook.

Bentonite seals will be no less than 2-ft thick nor more than 5-ft thick as measured immediately after placement. The normal installation will include a 5-ft seal. Thinner seals may be used in special cases. The final depth to the top of the bentonite seal will be measured and recorded.

3.4.1 Grout

Grout used in construction will be composed by weight of:

- 20 parts cement (Portland cement, type II) (see previous table)
- 0.4-1 part (maximum) (2-5 percent) bentonite
- 8-gal (maximum) approved water per 94-lb bag of cement.

Neither additives nor borehole cuttings will be mixed with the grout. Bentonite will be added after the required amount of cement is mixed with the water.

All grout material will be combined in an aboveground container and mechanically blended to produce a thick, lump-free mixture. The mixed grout will be recirculated through the grout pump prior to placement. Grout placement will be performed using a commercially available grout pump and a rigid, side discharge tremie pipe.

The following will be noted in the Field Logbook: (1) calculations of predicted grout volumes; (2) exact amounts of cement, bentonite, and water used in mixing grout; (3) actual volume of grout placed in the hole; and (4) any discrepancies between calculated and actual volumes used. If a discrepancy of greater than 10 percent exists between calculated and actual volumes exists, an explanation for the discrepancy will also be entered in the Field Logbook.

Well protective casings will be installed around all monitoring wells on the following day as the initial grout placement around the well. Any annulus formed between the outside of the protective casing and the borehole will be filled to ground surface with cement.

The construction of each well will be depicted as built in a well construction diagram. The diagram will be attached to the boring log and will graphically denote:

- Screen location, length
- Joint location
- Granular filter pack
- Seal
- Grout
- Cave-in
- Centralizers
- Height of riser
- Protective casing detail.

3.5 MONITORING WELL COMPLETION

Assemble appropriate decontaminated lengths of pipe and screen. Make sure these are clean and free of grease, soil, and residue. Lower each section of pipe and screen into the borehole, one at a time, screwing each section securely into the section below it. No grease, lubricant, polytetrafluoroethelyne tape, or glue may be used in joining the pipe and screen sections.

If a well extends below 50 ft, centralizers will be installed at 50 ft and every 50 ft thereafter except within screened interval and bentonite seal. Centralizer material will be PVC, polytetrafluoroethelyne, or stainless steel. Determination of centralizer material will be based on the same criteria as screen and calling selection.

Cut the riser with a pipe cutter approximately 2-2.5 ft above grade. All pipe cuts MUST be square to ensure that the elevation between the highest and lowest point of the well casing is less than or equal to 0.02 ft Notch, file, or otherwise permanently scribe a permanent reference point on the top of the casing.

Torches and saws may not be used to cut the riser. Care must be taken that all filings or trimmings cut from the reference point fall outside the riser rather than into the well. **Under no circumstances will a permanent marker or paint pencil be used to mark the reference point**.

In some locations, safety requirements may mandate that a well be flush-mounted with no stick-up. If a flush-mounted well is required at a given location, an internal pressure cap must be used instead of a vented cap to ensure that rainwater cannot pool around the wellhead and enter the well through the cap.

When the well is set to the bottom of the hole, temporarily place a cap on top of the pipe to keep the well interior clean.

Place the appropriate filter pack (Section 3.4). Monitor the rise annulus with a weighted tape to assure that bridging is not occurring.

After the pack is in place, wait 3-5 minutes for the material to settle, tamp and level a capped PVC pipe, and check its depth weighted steel tape.

Add a 1-2 ft cap of fine-grained (Ottawa) sand to prevent infiltration of the filter pack by overlying bentonite seal. See Section 3.4 for guidance on appropriate thickness of fine sand layer.

Install the bentonite seal (2- to 5-ft thick) by dropping bentonite pellets into the hole gradually. If the well is deeper than 30 ft, a tremie pipe will be used to place either bentonite pellets or slurry. Tamp and level pellets. If the well is 30 ft, tamp with a capped PVC pipe, if >30 ft, tamping bay be accomplished with the weighted end of the tape. In either case, check the depth to the top of the seal with a weighted tape as above.

If the bentonite pellets are of poor quality, they may have a tendency to hydrate and swell inside the tremie pipe and bridge. This situation may be solved by the following procedure:

1. Use a different brand of pellets. Different brands may have longer hydration times.

2. Freeze the pellets[7]. Note that this will require a longer wait time to allow proper hydration after the pellets thaw.

3. Place the bentonite seal as a slurry using a side-discharge tremie pipe as though installing grout. Note (Section 3.4) this will require that a minimum of 2 ft of fine sand be placed as a cap on top of the filter pack material.

Wait for the pellets to hydrate and swell. Hydration times will be determined by field test or by manufacturer's instructions. Normally this will be 30-60 minutes. Document the hydration time in the field notebook. If the pellets are above the water level in the hole, add several buckets of clean water to the boring. Document the amount of water added to the hole.

Mix an appropriate cement-bentonite slurry (Section 3.4). Be sure the mixture is thoroughly mixed and as thick as is practicable.

Lower a side discharge tremie pipe into the annulus to the level of the pellet seal.

Pump the grout slurry into the annulus while withdrawing the tremie pipe and temporary casing.

Stop the grout fill at 5 ft below the ground surface. Allow to cure for not less than 12 hours. If grout settles more than 6 in., add grout to bring level back up to within 5 ft of ground surface. Place approximately 2 ft of bentonite pellets (minimum 0.5 ft) in annulus. Seat the protective casing in the bentonite seal, allowing no more than 0.2 ft between the top of the well casing and the bottom of the protective casing cap. Fill inner annulus (between well casing and protective casing) with bentonite pellets to the level of the ground surface. Cover bentonite pellets with 1 ft of clean granular material (coarse sand or pea gravel filter pack). Fill the outer annulus (between the protective casing and the borehole) with neat cement. Allow the cement to mound above ground level and finish to slope away from the casing. Lock the cap.

— OR —

Continue the grout fill to the ground surface. Seat the protective casing in the grout, allowing no more than 0.2 ft between the top of the well casing and the bottom of the protective casing cap. Lock the cap.

— AND —

Allow the grout slurry to set overnight.

7. Bentonite pellets may be "flash-frozen" by brief immersion in liquid nitrogen (LN2). This can be accomplished by pouring LN2 over a small quantity (0.25-0.5 bucket) of pellets, allowing the LN2 to boil off, then pouring the pellets into the tremie pipe. **NOTE:** Use of LN2 is an additional jobsite hazard and must be addressed in the contractor's Health and Safety Plan. This contingency must be covered before drilling starts in order to avoid delays in well installation.

Fill the outer annulus (between the casing and the borehole) with neat cement. Allow the cement to mound above ground level and finish to slope away from the casing.

Slope the ground surface away from the casing for a distance of 2 ft, at a rate of no less than 1 in. in 2 ft. Surface this sloping pad with a geotextile mat covered by 3 in. of coarse gravel.

— OR —

Frame and pour a 4-ft square × 6-in. thick (4 ft × 4 ft × 6 in.) concrete pad centered around the protective casing.

— AND —

Set pre-painted protective steel pickets (3 or 4) evenly around and 4 ft out from well. These pickets will be set into 2 ft deep holes, the holes will then be filled with concrete; and if the pickets are not capped, they will also be filled with concrete.

3.6 WELL DEVELOPMENT

Well development is the process by which drilling fluids, solids, and other mobile particulates within the vicinity of the newly installed monitoring well have been removed while restoring the aquifer hydraulic conductivity. Development corrects any damage to or clogging of the aquifer caused by drilling, increases the porosity of the aquifer in the vicinity of the well, and stabilizes the formation and filter pack sands around the well screen.

Well development will be initiated after 48 consecutive hours but no longer than 7 calendar days following grouting and/or placement of surface protection.

Two well development techniques, over pumping and surging, will be employed in tandem. Over pumping is simply pumping the well at a rate higher than recharge. Surging is the operation of a plunger up and down within the well casing similar to a piston in a cylinder.

3.6.1 Materials Required

The following materials will be required for well development:

- Well Development Form
- Boring Log and Well Completion Diagram for the well
- Submersible pump or bailer of appropriate capacity, and surge block
- Conductivity, pH, ORP, turbidity, dissolved oxygen, and temperature meters
- Electric well sounder and measuring tape
- Containers for purged water, if required.

3.6.2 Summary of Procedures and Data Requirements

Pump or bail the well to ensure that water flows into it, and to remove some of the fine materials from the well. Removal of a minimum of one equivalent volume is recommended at this point. The rate of removal should be high enough to stress the well by lowering the water level to approximately half its original level. If well recharge exceeds 15 gpm, the requirement to lower the head will be waived.

Slowly lower a close-fitting surge block into the well until it rests below the static water level, but above the screened interval. (NOTE: This latter is not required in the case of a light non-aqueous phase liquid well.)

Begin a gentle surging motion which will allow any material blocking the screen to break up, go into suspension, and move into the well. Continue surging for 5-10 minutes, remove surge block, and pump or bail the well, rapidly removing at least one equivalent volume.

Repeat previous step at successively lower levels within the well screen until the bottom of the well is reached. Note that development should always begin above, or at the top of, the screen and move progressively downward to prevent the surge block from becoming sand locked in the well casing. As development progresses, successive surging can be more vigorous and of longer duration as long as the amount of sediment in the screen is kept to a minimum.

Development is expected to take at least 2 hours in a small well installed in a clean sand, and may last several days in large wells, or in wells set in silts with low permeabilities.

Development will continue until little or no sediment can be pulled into the well, and target values for parameters listed below are met.

At a minimum, development will remove 3-5 well volumes of water. One development volume (DV) is defined as (1) equivalent volume, plus (1) the amount of fluid lost during drilling, plus (1) the volume of water used in filter pack placement.

1. Monitor water quality parameters before beginning development procedures, and after removing 2, 2.5, and 3 well volumes of water.

2. If these parameters have stabilized over the three readings, the well will be considered developed.

3. If the parameters have not stabilized after these three readings, continue pumping the well to develop, but stop surging. Monitor the stabilization parameters every half DV.

4. When the parameters have stabilized over three consecutive readings at half DV intervals, the well will be considered developed.

All water removed must be disposed of as directed by the Work Plan.

Record all data as required on a Well Development Record Form (Appendix A), which is made a part of the complete Well Record. These data include:

- Depths and dimensions of the well, casing, and screen obtained from the well diagram.
- Water losses and uses during drilling, obtained from the boring log for the well.
- Measurements of the following indicator parameters: turbidity, pH, conductivity, ORP potential, dissolved oxygen, and temperature.
- Target values for the indicator parameters listed above are as follows: pH – stabilize, conductivity – stabilize, ORP – stabilize, dissolved oxygen –- stabilize, temperature – stabilize, turbidity – 5 nephelometric turbidity units or stabilize. A value is considered to have stabilized when three consecutive readings taken at half DV intervals are within 10 percent of each other.
- Notes on characteristics of the development water.
- Data on the equipment and technique used for development.
- Estimated recharge rate and rate/quantity of water removal during development.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

Refer to the site-specific Health and Safety Plan for discussion of hazards and preventive measures during well development activities.

6. REFERENCES

Aller, L. et al. 1989. Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells, National Water Well Association.

American Society for Testing and Materials (ASTM). D2487-92 Standard Classification of Soils for Engineering Purposes (Unified Soil Classification System).

———. D5092-90 Standard Practice for Design and Installation of Groundwater Monitoring Wells in Aquifers.

Cohen, R.M. and J.W. Mercer. 1993. DNAPL Site Evaluation, CRC Press, Inc.

Nielsen, D.M. 1993. Correct Well Design Improves Monitoring, in *Environmental Protection*, Vol.4, No.7. July.

U.S. Army Toxic and Hazardous Materials Agency. 1987. Geotechnical Requirements for Drilling, Monitoring Wells, Data Acquisition and Reports. March.

U.S. Environmental Protection Agency. 1989. Groundwater Handbook.

Appendix A

Field Record of Well Development Form

EA Engineering, P.C.
EA Science and Technology

FIELD RECORD OF WELL DEVELOPMENT

Project Name:	Project No:	Date:
EA Personnel:	Development Method:	
Weather/Temperature/Barometric Pressure:		Time:

Well No.:	Well Condition:
Well Diameter:	Measurement Reference:
Well Volume Calculations	
A. Depth To Water (ft):	D. Well Volume/ft:
B. Total Well Depth (ft):	E. Total Well Volume (gal)[C*D]:
C. Water Column Height (ft):	F. Five Well Volumes (gal):

Parameter	Beginning	1 Volume	2 Volumes	3 Volumes	4 Volumes	5 Volumes
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

Parameter	6 Volumes	7 Volumes	8 Volumes	9 Volumes	10 Volumes	End
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

NOTE: NTU = Nephelometric turbidity unit.
ORP = Oxidation-reduction potential.

COMMENTS AND OBSERVATIONS: _____

EA Engineering, P.C.
EA Science and Technology

FIELD RECORD OF WELL DEVELOPMENT

Project Name:	Project No:	Date:
EA Personnel:	Development Method:	
Weather/Temperature/Barometric Pressure:		Time:

Well No.:	Well Condition:
Well Diameter:	Measurement Reference:

Parameter	Beginning	1 Volume	2 Volumes	3 Volumes	4 Volumes	5 Volumes
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

Parameter	6 Volumes	7 Volumes	8 Volumes	9 Volumes	10 Volumes	End
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

Standard Operating Procedure No. 024
for
Photoionization Detector (Microtip HL-200)

Prepared by

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Revision 0
August 2007

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for field operations with a photoionization detector (PID). PIDs use an ultraviolet emitting lamp designed to detect, measure, and display the total concentration of airborne ionizable gases and vapors. This information is used to determine control measures such as protection and action levels.

Use of brand names in this Standard Operating Procedure is not intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor will provide applicable and comparable standard operating procedure for the maintenance and calibration of same.

2. MATERIALS

The following materials may be required:

Battery back	Regulator
Calibration gas (100ppm Isobutylene)	Tedlar bag
PID (i.e., Microtip HL-200)	Tygon tubing

3. STARTUP/CALIBRATION PROCEDURE

The following describes startup and calibration procedures:

- Turn the instrument on by pressing the back of the power switch located on the handle of the Microtip.
- The message “Warming up now, please wait” will be displayed for up to 3 minutes. After normal display appears, the Microtip is ready for calibration.
- Fill a Tedlar bag with the desired calibration gas (usually 100 ppm isobutylene).
- Press SETUP button and select the desired Cal Memory using the arrow keys (normally set to 200 ppm). Press EXIT button to leave setup function.
- Press CAL button and expose Microtip to Zero Gas. (Usually clean outdoor air will be suitable. If any doubt exists as to the cleanliness of the background air, a commercial source of zero gas should be used.).
- The Microtip then asks for the Span Gas concentration. Enter the known span gas concentration and then connect the Tedlar bag containing the Span Gas.

NOTE: THE SPAN GAS CONCENTRATION IS DEPENDENT UPON BOTH THE CONCENTRATION OF THE SPAN GAS USED AND THE RATING OF THE ULTRAVIOLET LAMP IN THE MICROTIP AT TIME OF CALIBRATION. IF USING 100 ppm ISOBUTYLENE AND THE STANDARD 10.6 eV LAMP, THE SPAN GAS CONCENTRATION WILL BE 56 ppm.

- Press enter and the Microtip sets its sensitivity. Once the display reverts to normal, the Microtip is calibrated and ready for use. Remove the Span Gas from the inlet probe. The instrument should be calibrated at least once a day.

4. BATTERY CHARGING

The following is a summary of battery charging procedures:

- Ensure Microtip is off.
- Set the voltage selector switch on the bottom of the battery charger to the appropriate AC line voltage.
- Press the release button on the bottom of the Microtip and remove the battery pack by sliding it backwards.
- Plug charger into the battery pack and then into an AC outlet and allow the battery to charge for at least 8 hours.
- After charging, remove the charger, first from the outlet then from the battery pack, and slide the battery pack back onto the Microtip.

5. PRECAUTIONS

The following is a summary of precautions while using the Microtip:

- Microtip does not carry an Intrinsic Safety Rating and must not be used in a hazardous location where flammable concentrations of gases or vapors are constantly present.
- All calibration, maintenance, and servicing of this device, including battery charging, must be performed in a safe area away from hazardous locations.
- Do not open or mutilate battery cells.
- Do not defeat proper polarity orientation between the battery pack and battery charger.
- Substitution of components may affect safety rating.

6. REFERENCES

Microtip HL-200 User's Manual. February 1990.

Standard Operating Procedure No. 025
for
Soil Sampling

Prepared by

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CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for sampling surface and subsurface soils. Soil samples give an indication of the area and depth of site contamination, so a representative sample is very important.

2. MATERIALS

The following materials may be required:

Bucket auger or push tube sampler	Split-spoon, Shelby tube, or core barrel sampler
Drill rig and associated equipment	Stainless steel bowl
Personal protective equipment as required by the Health and Safety Plan	Stainless steel spoon, trowel, knife, spatula (as needed)

3. PROCEDURE

3.1 SUBSURFACE SAMPLES

Don personal protective equipment. Collect split-spoon, core barrel, or Shelby Tube samples during drilling. Upon opening sampler, or extruding sample, immediately screen soil for volatile organic compounds using either a photoionization detector or flame ionization detector. If sampling for volatile organic compounds, determining the area of highest concentration, use a stainless steel knife, trowel, or laboratory spatula to peel and sample this area. Log the sample in the Field Logbook while it is still in the sampler. Peel and transfer the remaining sample in a decontaminated stainless steel bowl. Mix thoroughly with a decontaminated stainless steel spoon or trowel. Place the sample into the required number of sample jars. Preserve samples as required. Discard any remaining sample into the drums being used for collection of cuttings. Decon sampling implements. All borings will be abandoned.

NOTE: If sample recoveries are poor, it may be necessary to composite samples before placing them in jars. In this case, the procedure will be the same, except that two split-spoon samples will be mixed together. The Field Logbook should clearly state that the samples have been composited, which samples were composited, and why the compositing was done.

Samples taken for geotechnical analysis will be undisturbed samples, collected using a thin-walled (Shelby tube) sampler.

3.2 SURFICIAL SOIL SAMPLES

Don personal protective equipment. Remove vegetative mat. Collect a sample from under the vegetative mat with a stainless steel trowel, push tube sampler, or bucket auger. If a representative sample is desired over the depth of a shallow hole or if several shallow samples are to be taken to represent an area, composite as follows:

- As each sample is collected, place a standard volume in a stainless steel bowl.
- After all samples from each hole or area are in the bucket, homogenize the sample thoroughly with a decontaminated stainless steel spoon or spatula.

If no compositing is to occur, place sample directly into the sample jars. Place the leftover soil in the auger borings and holes left by sampling. If necessary, add clean sand to bring the subsampling areas back to original grade. Replace the vegetative mat over the disturbed areas. Samples for volatile organic compounds will not be composited. A separate sample will be taken from a central location of the area being composited and transferred directly from the sampler to the sample container. Preserve samples as required. Decon sampling implements.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

Refer to the Health and Safety Plan.

Soil samples will not include vegetative matter, rocks, or pebbles, unless the latter are part of the overall soil matrix.

6. REFERENCES

ASTM International. Method D1586-84, Penetration Test and Split-Barrel Sampling of Soils.

———. Method D1587-83, Thin Walled Sampling of Soils.

Department of the Army, Office of the Chief of Engineers. 1972. Engineer Manual 1110-2-1907 Soil Sampling. 31 March.

Standard Operating Procedure No. 028
for
Well and Boring Abandonment

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to establish the protocols by which all wells and borings will be safely abandoned. The primary objective of well abandonment is to ensure that the abandoned well or boring does not provide a conduit for the vertical migration of contamination between aquifers.

2. MATERIALS

The following materials may be required:

Drill rig	Bentonite pellets (seal)
Filter pack material	Cement (Portland Type II)
Pure sodium bentonite with no additives (bentonite) powder (grout)	Approved water

3. PROCEDURE

The procedures used in boring abandonment will ideally accomplish two objectives: (1) protect aquifers from cross-contamination by sealing the borehole, and (2) restore the strata in the borehole to nearly original conditions by selective placement of fill material.

Any casing will be pulled, drilled out, or thoroughly pierced. Using tremie pipe, grout will be placed from the bottom of the hole to within 3 ft of the ground surface, and allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with "ABD," for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples, completion records, and abandonment procedures will be included in the records of work on the site or cluster.

If the hole is within 10 ft of a monitoring well in the same aquifer, or a replacement well is to be installed within 10 ft of the well, any temporary casing will be pulled, drilled out, or thoroughly pierced. Using tremie pipe, the hole will then be backfilled with filter pack material opposite sand strata and bentonite or grout opposite substantial (2 ft or thicker) clay and silt strata. Where sand as backfill approaches the ground surface, 2 ft of bentonite will be placed above the sand and a 3-ft concrete plug will be placed at the surface. Otherwise, backfill materials will be placed from the bottom of the hole to within 3 ft of the ground surface. These materials will be allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with "ABD," for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples, completion records, and abandonment procedures will be included in the records of work on the site cluster.

If the well is not within 10 ft of another monitoring well, or if there are no substantial, continuous sand bodies, and no replacement well is planned within 10 ft of the original well location, then the hole may be grouted from the bottom to the top.

3.1 GROUT

Grout used in construction will be composed by weight of:

- 20 parts cement (Portland cement, Type II or V)
- 0.4-1 part (maximum) (2-5 percent) bentonite
- 8 gal (maximum) approved water per 94-lb bag of cement.

Neither additives nor borehole cuttings will be mixed with the grout. Bentonite will be added after the required amount of cement is mixed with the water.

All grout material will be combined in an aboveground container and mechanically blended to produce a thick, lump-free mixture. The mixed grout will be recirculated through the grout pump prior to placement.

Grout placement will be performed using a commercially available grout pump and a rigid tremie pipe removal and grouting will be accomplished in stages, aquifer by aquifer, sealing the boring from the bottom to ground surface. This will be accomplished by placing a grout pipe to the bottom and pumping grout through the pipe until undiluted grout reaches the bottom of the next higher section of casing or, for the top-most section, until grout flows from the boring at ground surface. Efforts will be made to grout incrementally as the temporary casing is removed.

After 24 hours, the abandoned drilling site will be checked for grout settlement. On that day, any settlement depression will be filled with grout and rechecked 24 hours later. This process will be repeated until firm grout remains at the ground surface.

3.2 BORINGS

The term “Borings” as used in this Standard Operating Procedure applies to any drilled hole made during the course of a remedial investigation which is not completed as a well. This includes soil test borings, soil sampling borings, and deep stratigraphic borings. Whether completed to the planned depth or aborted for any reason prior to reaching that depth, borings will be grouted and normally closed within 4 hours, or within 4 hours or completion of logging of completion of logging.

3.2.1 Shallow Borings not Penetrating Water Table

Shallow borings made for the collection of subsurface soil samples will be abandoned by backfilling the hole with cuttings from the hole, **if and only if the boring does not penetrate the water table.** Clean sand will be used to make up any volume not filled by the cuttings.

3.2.2 Borings Penetrating the Water Table

Shallow borings made for the collection of subsurface soil samples **which penetrate the water table** will be abandoned by grouting the hole from the bottom to the top.

3.2.3 Deep Stratigraphic Borings

Deep stratigraphic borings will normally be located in areas which, by virtue of the historical record, are presumed relatively uncontaminated. Therefore, these borings are usually over 100 ft from any sampling well locations. Any boring located within 10 ft of a proposed well location, or located directly upgradient or downgradient (on anticipated flow line) of a proposed well location, will be abandoned by placing clean sand in the aquifer intervals and bentonite or grout in aquitard intervals as described above. If the boring is over 10 ft from and/or not upgradient of a proposed well location, the boring will be completely filled with grout.

3.3 WELLS

The following procedure applies to wells aborted prior to completion and existing wells determined to be ineffective or otherwise in need of closure.

Prior to abandoning any developed well, the proper well licensing body will be provided written notification along with an abandonment plan for that well.

If the well is within 10 ft of another monitoring well in the same aquifer, or a replacement well is to be installed within 10 ft of the well, casing will be pulled, drilled out, or thoroughly pierced. Using tremie pipe, the hole will then be backfilled with filter pack material opposite sand strata and bentonite or grout opposite substantial (2 ft or thicker) clay and silt strata. Where sand as backfill approaches the ground surface, 2 ft of bentonite will be placed above the sand and below the concrete plug near the surface. Backfill materials will be placed from the bottom of the hole to within 3 ft of the ground surface. These materials will be allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with “ABD,” for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples, completion records, and abandonment procedures will be included in the records of work on the site cluster.

If the well is not within 10 ft of another monitoring well, and is not to be replaced by another well within 10 ft of the original location, casing will be pulled, drilled out, or thoroughly pierced. Using tremie pipe, grout will be placed from the bottom of the hole to within 3 ft of the ground surface, and allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with “ABD,” for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples, completion records, and abandonment procedures will be included in the records of work on the site cluster.

4. REPLACEMENT WELLS

Replacement wells (if any) will normally be offset at least 10 ft from any abandoned well in a presumed upgradient or crossgradient groundwater direction. Site-specific conditions may necessitate variation to this placement.

5. PRECAUTIONS

None.

Standard Operating Procedure No. 031
for
Sample Container Cleaning

Prepared by

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CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to define laboratory protocols to be used in cleaning and preparing containers used to collect environmental samples.[1]

2. MATERIALS

The following materials may be required:

5 percent sodium hydroxide	Bottle caps
5 percent Ultrex nitric acid	Deionized water
40-ml vials	Hexane (Nanograde or equivalent)
Acetone	Methylene chloride
Alconox detergent	Polyethylene bottles
Amber glass bottles	Polytetrafluoroethelyne liners

3. PROCEDURE

3.1 POLYETHYLENE BOTTLES

Rinse bottles and lids sequentially with 5 percent sodium hydroxide, deionized water, and 5 percent nitric acid with deionized water. Drain and allow to air dry.

3.2 AMBER GLASS BOTTLES AND 40 ml VIALS

- Wash bottles in detergent and rinse with copious amounts of distilled water
- Rinse with acetone
- Rinse with methylene chloride
- Rinse with hexane
- Allow bottles to air dry
- Place bottles in a drying oven and heat to 200°C
- Allow bottles to cool prior to sealing with clean caps and polytetrafluoroethelyne liners.

3.3 BOTTLE CAPS

- If applicable, remove paper liners from caps
- Wash caps with detergent, followed by a distilled water rinse
- Dry caps in drying oven at 40°C.

1. This Standard Operating Procedure is included for completeness only. It is anticipated that sample containers will either be provided by the laboratory or that the sampling contractor will purchase new, certified clean sample containers.

3.4 POLYTETRAFLUOROETHELYNE LINERS

- Always handle liners with forceps or tweezers; never use fingers.
- Wash liners with detergent, followed by distilled water rinse.
- Rinse the liners with acetone, followed by hexane (Nanograde or equivalent).
- Allow liners to air dry prior to placing in clean caps, then heat liner and caps in drying oven at 40°C for 2 hours.
- Allow caps and liners to cool prior to placing on clean bottles.

A statistically representative number of randomly selected clean sample containers will be analyzed for Target Analyte List/Target Compound List analytes. Results of these analyses will be provided to the client.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

None.

Standard Operating Procedure No. 039
for
Sample Preservation and Container Requirements

Prepared by

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Revision 1
April 2012

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Page

1. PURPOSE AND SCOPE

The purpose of this Standard Operating Procedure (SOP) is to define the preservatives and techniques to be employed in preserving environmental samples between collection and analysis.

2. MATERIALS

The following materials may be required:

Containers (see Section 3 for description)	NaOH
HNO_3	Ice chests
H_2SO_4	Ice

3. DEFINITION OF CONTAINER TYPES

Listed below are the definitions of various container types.

Type	Container	Closure	Septum
A	80-ounce (oz) amber glass, ring handle bottle/jug, 38-millimeter (mm) neck finish	White polypropylene or black phenolic, baked polyethylene cap, 38-430 size, 0.015-mm polytetrafluoroethelyne (PTFE) liner	
B	40-mililiter (mL) glass vial, 24-mm neck finish	White polypropylene or black phenolic, open top, screw cap, 15-mm opening, 24-400 size	24-mm disc of 0.005-in. PTFE bonded to 0.120-in. silicon for total thickness of 0.125 in.
C	1-L high density polyethylene, cylinder-round bottle, 28-mm neck finish	White polyethylene cap, white ribbed, 28-410 size; F217 polyethylene liner	
D	120-mL wide mouth glass vial, 48-mm neck finish	White polyethylene cap, 40-480 size; 0.015-mm PTFE liner	
E	250-mL Boston round glass bottle	White polypropylene or black phenolic, open top, screw cap	Disc of 0.005-in. PTFE bonded to 0.120-in. silicon for total thickness of 0.125 in.
F	8-oz short, wide mouth, straight-sided, flint glass jar, 70-mm neck finish	White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.030-mm PTFE liner	
G	4-oz tall, wide mouth, straight - sided, flint glass jar, 48-mm neck finish	White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.015-mm PTFE liner	

Type	Container	Closure	Septum
H	1-L amber, Boston round, glass bottle, 33-mm pour-out neck finish	White polypropylene or black phenolic, baked polyethylene cap, 33-430 size; 0.015-mm PTFE liner	
K	4-L amber glass ring handle bottle/jug, 38-mm neck finish.	White polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm PTFE liner	
L	500-mL high-density polyethylene, cylinder bottle, 28-mm neck finish	White polypropylene, white ribbed, 28-410 size; F217 polyethylene liner	

4. PROCEDURE

All containers described in Section 3 must be certified clean, with copies of laboratory certification furnished upon request. There may be circumstances when alternative containers will be used (e.g., aluminum foil around tissue samples placed in plastic bags, plastic buckets for large soil/sediment samples, etc.) for which laboratory certification may not be available. Such containering should be appropriately decontaminated or verified appropriately clean prior to using.

Water samples will be collected into pre-preserved containers appropriate to the intended analyte as given in Quality Assurance Project Plan. Samples taken for volatile organic compounds will be collected in accordance with SOP No. 003, Section 3.3.8. Samples taken for metals analysis will be verified in the field to a pH <2. The container should be tightly capped, then swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure this operation may have generated. Samples taken for total phosphorous content will be verified in the field to a pH <2. The container should be tightly capped and swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure this operation may have generated. Samples taken for cyanide will be verified for a pH >12. Most other samples do not require added preservation; however, there are analytes that may require special preservation, i.e., sulfide which requires a zinc acetate preservation. Preservation must be performed as documented in the project-specific Quality Assurance Project Plan. These samples will be immediately placed on ice and cooled to 4±2°C.

Soil and sediment samples will be collected into containers appropriate to the intended analyte as given in the Quality Assurance Project Plan. Samples taken for volatile organic compound analysis will collected in accordance with the site-specific SOP. Samples taken for metals analysis will be tightly capped, placed on ice, and maintained at a temperature of 4°C. Samples taken for total phosphorous content will be tightly capped, placed on ice, and maintained at a temperature of 4°C. Under most circumstances, no preservatives will be added to any other soil samples; follow project-specific requirements as documented in the Quality Assurance Project Plan. These samples will be immediately placed on ice and cooled to 4±2°C.

5. MAINTENANCE

Not applicable.

6. PRECAUTIONS

Note that acidifying a sample containing cyanide may liberate HCN gas.

- Avoid breathing any fumes emanating from acidified samples.
- Acidify samples only in the open, rather than in closed spaces, i.e., a vehicle.
- Hold suspected HCN-generating sample away from body and downwind while manipulating it.
- See the Health and Safety Plan for other safety measures.

7. REFERENCES

U.S. Environmental Protection Agency (EPA). 1986. Test Methods for Evaluating Solid Waste, SW-846.

———. 1987. A Compendium of Superfund Field Operations Methods, EPA 540-P87-001.

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Standard Operating Procedure No. 043 for Multi-Probe Water Quality Monitoring Instruments

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Revision 1
April 2011

CONTENTS

1. PURPOSE AND SCOPE

The purpose of this Standard Operating Procedure is to delineate protocols for field operation of multi-probe water quality instruments. The instrument can monitor a variety of basic parameters including dissolved oxygen, percent saturation, temperature, pH, specific conductance, resistivity, salinity, total dissolved solids, oxidation reduction potential (ORP), level, and depth.

Use of brand names in this Standard Operating Procedure is not intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor will provide applicable and comparable standard operating procedures for the maintenance and calibration of same.

2. MATERIALS

The following materials may be required:

- Multi-probe instrument
- Probe/sonde with appropriate cables
- Appropriate standards
- Accessories (batteries, charger, case, etc.)
- Instrument logbook
- Manufacturer's Operations Manual.

3. CALIBRATION PROCEDURE

Calibration must be performed daily at a minimum before using the instrument. Calibration may be performed in the laboratory or in the field. Detailed step-by-step calibration procedures for the equipment described below are provided in the most recent version of the manufacturer's Operations Manual. Documentation includes at a minimum: time, date, analyst, standard, primary standard lot number, secondary standard lot number, and expiration dates of standards.

Fill the calibration cup with the appropriate standard as follows:

- Temperature: None required
- Specific Conductance: Conductivity standards
- pH: pH 7 buffer plus pH 4 and/or pH 10 buffer
- Dissolved Oxygen: Saturated air or saturated water
- ORP: Quinhydrone (Zobell's Solution)
- Turbidity: Nephelometric turbidity unit (NTU) standards
- Salinity: Calibration for specific conductance
- Depth/Level: Set zero in air.

3.1 CONDUCTIVITY CALIBRATION

Conductivity meters are calibrated at least once per day to at least one standard. The standard should be selected in accordance with the range expected to be measured (e.g., 1.0 µS/cm standard should not be used to calibrate meters being used in saltwater). See manufacturer's recommendations in the Operations Manual for additional information on calibration standard selection. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.2 pH CALIBRATION

The pH meters are calibrated at least once per day to a minimum of two standard buffers (pH 4 and 7, or pH 7 and 10) in accordance with the range expected to be measured. The calibration is verified using a fresh solution of pH 7 buffer post-calibration. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.3 DISSOLVED OXYGEN CALIBRATION

Dissolved oxygen meters are air calibrated at least once per day. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.4 OXIDATION REDUCTION POTENTIAL CALIBRATION

ORP meters are calibrated at least once per day to at least one standard. It is recommended that Zobell's Solution is used; however, another solution can be used as long as it meets the manufacturer's specifications for calibration. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.5 TURBIDITY CALIBRATION

The turbidity meters are calibrated at least once per day to a minimum of two standards (0 NTU and 100 or 126 NTUs recommended) in accordance with the range expected to be measured. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.6 DEPTH/LEVEL CALIBRATION

The depth and level calibration is performed with the depth sensor module in the air and not immersed in any solution. The appropriate correction for height above the water surface is inputted into the meter. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.7 ADDITIONAL CALIBRATIONS

Additional measurements may be taken with the multi-probe water quality instruments. For any of these measurements, the calibration procedures will be conducted in accordance with the manufacturer's specifications. Calibration information is recorded in conjunction with the data collected for that sampling event.

4. FIELD OPERATION

4.1 SETUP OF MULTI-PROBE WATER QUALITY INSTRUMENT

Post-calibration and prior to sampling, the multi-probe water quality instrument will be set up for data collection. If the cables have been unattached, they will be reconnected to the transmitter (if applicable) and the display. Once all cables are attached, the meter will be turned on and allowed to warm up for a few seconds in order to allow the display screen to load.

4.2 SURFACE WATER

Prior to sampling, check the condition of the probes before each deployment. When sampling in surface water, the sensor must be in an amount of water sufficient for all probes to be submerged. Data values displayed on the display screen are recorded in the field logbook and accepted into the instrument's data logger. Post-data collection, the sensor will be retrieved and rinsed for use at the next sample location. If travel time between sample locations is great, the display is to be turned off. When all sampling is completed, disconnect all equipment and return it to its proper storage location.

4.3 GROUNDWATER

Prior to sampling, check the condition of the probes before each deployment. When sampling groundwater, mount sampler on a flow-through sampler cup. Start sampler pump and allow pump/hose system to be purged of air bubbles. Sampling rate should be set to record all parameters each time 1-3 liters (unless otherwise specified in the sampling plan) have been removed from the well. Record all the monitored values in the appropriate field logbook to ensure against inadvertent data loss.

5. MAINTENANCE

All maintenance should be performed in accordance with the manufacturer's Operations Manual.

6. PRECAUTIONS

Check the condition of the probes frequently between sampling. Do not force pins into connections, note keying sequence. If field readings are outside the expected range, check for bubbles on, or damage to, the probes. If there are no bubbles or damage, recalibrate the sensor.

7. REFERENCES

Manufacturer's Operations Manual.

Standard Operating Procedure No. 047
Direct-Push Technology Sampling

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August 2007

CONTENTS

1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) establishes the protocol for using direct-push technology (DPT) in media sampling and performing subsurface characterization. This SOP includes the following DPT methods: Geoprobe®, Hydropunch®, Cone Penetrometer Testing (CPT), and Site Characterization and Analysis Penetrometer System (SCAPS).

2. MATERIALS

The following materials may be required:

Appropriately sized, all-terrain vehicle-skid-or track-mounted; DPT equipment; and supplies (i.e., hydraulic derrick and hammer assembly)	Personal protective equipment
Bentonite grout and clean sand for DPT hole abandonment	Phosphate-free, laboratory-grade detergent (e.g., Liquinox, Alconox, etc.)
DPT stainless steel rods	Source of approved water
Heavy plastic sheeting	Steam cleaner/sprayer and water obtained from approved source for decontaminating DPT equipment
Logbook	Steel drums for intrusion derived wastes (e.g., contaminated personal protective equipment, decon solutions, etc.)
Long-handled bristle brushes	Wash and rinse tubs
Mini-bailer or tubing and peristaltic pump (groundwater sampling only)	

3. GEOPROBE® AND HYDROPUNCH®

3.1 MATERIALS

Water sources for Geoprobe® and Hydropunch® activities, grouting, sealing, filter placement, well installation, and equipment decontamination must be approved by the Project Manager prior to arrival of the Geoprobe® and Hydropunch® equipment. Information required for the water source includes: water source, manufacturer/owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analysis over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives will be the only additive allowed, and its use must be approved by the Project Manager prior to the arrival of the Geoprobe® and Hydropunch® equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product number, product description, and intended use for the product.

Portland Type II cement will be used for grout (refer to SOP No. 019).

3.2 GROUNDWATER – HYDRAULIC PUSHING AND SAMPLING

The objective of the selected DPT sampling technique is to allow grab samples to be taken at a selected site to facilitate aquifer characterization and analysis of potential contaminants. The analytical results from sampling can also be used to determine the placement of monitoring wells.

A site geologist will be present during all sampling and installation procedures, and will fully document all procedures and soil characteristics in the Field Logbook (refer to SOP No. 016).

The site geologist will have on hand, at a minimum, a copy of the approved Health and Safety Plan, this SOP, the Field Investigation Work Plan, a hand lens (10X), a standard color chart, and a grain size chart.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole Geoprobe® and Hydropunch® equipment.

Surface runoff or other fluids will not be allowed to enter any DPT location or well during or after DPT activities.

The subcontractor will use the equipment specific guidelines for installation of the Geoprobe® DPT equipment. Probe rods will be forced into the ground by hydraulic means.

- Drive the sampler to the desired groundwater sampling interval. At the desired depth, insert extension rods down the inside diameter of the probe rods until the extension reaches the bottom of the screen. Remove the probe rods and sampler sheath while holding the screen in place.

- Collect the groundwater sample in the screen interval with a mini-bailer, peristaltic or vacuum pump, or other acceptable small diameter sampling device.

- The head of the rod may be equipped with a sensing device for characterization of soil properties or the contaminant content.

The subcontractor will use the equipment-specific guidelines for installation of the Hydropunch® equipment. Rods will be forced into the ground by hydraulic means.

- The Hydropunch® tool is a double cylinder, designed to be sealed until the desired sampling depth is reached. Upon reaching the desired sampling depth, the outer cylinder is pulled back, exposing a perforated, stainless steel sampling barrel covered with filter material.

- The water sample enters the barrel and the sample is retrieved by pulling the probe rods from the hole with the hydraulic derrick and hammer assembly. Groundwater is the only media that is sampled by Hydropunch® equipment.

- The head of the rod may be equipped with a sensing device for characterization of the soil properties or the contaminant content.

- The sample volume collected with this technique is approximately 500-1,000 ml. Larger sample volumes can be collected by inserting tubing attached to a peristaltic pump into the rods to obtain water samples.

If desired, a small diameter monitoring well may be installed at this point. Refer to SOP No. 019 (Monitoring Well Installation).

If a well will not be installed, the rods will be removed as the borehole is simultaneously filled with a bentonite/grout mixture. A polyvinyl chloride (PVC) tube fed into the rod casing will allow the addition of grout.

3.3 SUBSURFACE SOIL – HYDRAULIC PUSHING AND SAMPLING

The objective of the selected DPT sampling technique is to allow grab samples to be taken at a selected site for characterization of the stratigraphy and for analysis of potential contaminants. The analytical results from sampling can also be used to determine the placement of monitoring wells.

A site geologist will be present during all DPT sampling and soil characterization. All procedures and soil characteristics will be fully documented in the Field Logbook (refer to SOP No. 016).

The site geologist will have on hand, at a minimum, a copy of the approved Health and Safety Plan, this SOP, the Field Investigation Plan, a hand lens (10X), a standard color chart, and a grain-size chart.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole Geoprobe® equipment.

Surface runoff or other fluids will not be allowed to enter any DPT location or well during or after DPT activities.

The subcontractor will use the equipment specific guidelines for installation of the Geoprobe® DPT equipment. Probe rods will be forced into the ground by hydraulic means. Additional rods will be added in 3- to 4-ft increments until the leading edge of the sampler reaches the top of the desired sampling interval.

Once the desired sampling depth has been reached, insert extension rods down the inside diameter of the probe rods until it reaches the top of the sampler assembly. Attach the extension rod handle to the top extension rod. Turn the handle clockwise until the stop-pin detaches from the drive head. Remove the extension rods and the stop-pin. Attach a drive cap to the probe and drive the sampler approximately 2 ft using hydraulic derrick.

The DPT sampler can be retrieved by pulling the probe rods from the hole with the hydraulic derrick and hammer assembly.

The liner will be capped with Teflon® tape and vinyl end caps. The liners can be split open to remove samples for composition analysis or for transfer to other containers for shipment to the laboratory for analysis.

The head of the rod may be equipped with a sensing device for characterization of the soil properties or the contaminant content.

3.4 DECONTAMINATION

All Geoprobe® and Hydropunch® DPT equipment must be thoroughly cleaned before and after each use to allow retrieval of representative groundwater samples. Geoprobe® soil sample liners are disposed of after each use. Scrub all metal parts with a stiff, long bristle brush and a non-phosphate soap solution. Steam cleaning may be substituted where available. Rinse with distilled water and allow to air-dry before assembly.

After decontamination, a new clean liner will be installed and all parts will be inspected for wear or damage.

Refer to SOP No. 005 (Field Decontamination).

3.5 ABANDONMENT

Pure bentonite or a bentonite/grout mixture (20:1) will be used to fill the resulting borehole if the water table is penetrated. Boreholes that do not penetrate the water table will be backfilled with cuttings from the hole and topped with a bentonite seal. Clean sand will be used to fill any remaining volume in the borehole.

Abandonment of Geoprobe® and Hydropunch® generated DPT boreholes will meet the standards established under SOP No. 028 (Well and Boring Abandonment).

4. CONE PENETROMETER TESTING

4.1 MATERIALS

A CPT rig typically consists of an enclosed 20- to 40-ton truck equipped with vertical hydraulic rams that are used to force a sensor probe into the ground. The weight of the CPT rig is dependent upon the thrust required at the site. The majority of CPT rigs are mounted in heavy-duty trucks that are ballasted to a total dead weight of approximately 15 tons. Screw anchors are utilized to develop the extra reaction to reach the maximum thrust of 20 tons. The rig is separated into two separate workspaces: data acquisition and hydraulic push areas.

Water sources for CPT activities and decontamination must be approved by the Project Manager prior to arrival of the CPT equipment. Information required for the water source includes: water source, manufacturer/owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analysis over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives will be the only additive allowed, and its use must be approved by the Project Manager prior to the arrival of the DPT equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product number, product description, and intended use for the product.

Portland Type II cement will be used for grout (refer to SOP No. 019).

4.2 SUBSURFACE CHARACTERIZATION

The objective of this technology is to collect stratigraphic information using CPT equipment to determine subsurface stratigraphy and geotechnical properties at a particular site. CPT activities will be in accordance with American Society for Testing and Materials D 3441-86 and American Society for Testing and Materials D 5778-95. The stratigraphic information gathered can be used to facilitate the selection of DPT sampling screen intervals. At the same time, it is possible to install a 0.25-in. diameter pre-packed PVC monitoring well.

CPT rods are used to hydraulically push the CPT probe into the subsurface. Probes cannot be pushed into hard rock, and significant gravel or cobble content in the formation may impede or preclude penetration of the probe. The depth of penetration achievable depends on the type of formation, type of sampling probe, and size of the hydraulic equipment used.

The CPT probe includes the following components:

- A conical tip to measure vertical resistance beneath the tip.
- A friction sleeve to measure frictional resistance on the side of the probe, as a function of depth.

- Two internal strain gauge-type load cells, which independently measure the vertical resistance and side friction.

- A cone pressure gauge to measure the water pressure as the probe is pushed into the ground.

- Inclinometer to determine potential drifting of the probe (optional).

- Seismic transducers to perform downhole seismic surveys (optional). Therefore, stratigraphic data collected with the CPT include: tip resistance, local friction, friction ratio, pore pressure, and resistivity.

Data will be transferred from the probe to the data acquisition system or logger through an electrical cable. The hole will be advanced continuously at a rate of 0.6-1.0 in. per second. The data will be logged at every 0.4-0.8 in. of penetration. Monitor the probe's stratigraphic position will be monitored as it advances downward. Perform pore water pressure dissipation tests in representative hydrostratigraphic intervals. Record dissipated pore water pressures to represent hydraulic head values.

Once the confining unit underlying the surficial aquifer or the required depth has been reached, the CPT is pulled from the ground. Target interval samples can be collected during CPT hole advancement using direct push sampling techniques, i.e., Geoprobe® or Hydropunch® (Section 3).

4.3 DECONTAMINATION

All CPT equipment must be thoroughly cleaned before arrival at the work site, between test holes, and prior to being moved out of a work area. Scrub all metal parts with a stiff, long bristle brush and a non-phosphate soap solution. Steam cleaning may be substituted where available. Rinse with distilled water and allow to air-dry before assembly.

Refer to SOP No. 005 (Decontamination).

4 .4 ABANDONMENT

If the push hole was developed for the stratigraphic test only, once the testing is completed, grout the hole from bottom to top. If the hole has not collapsed after removing the CPT, PVC piping will be used to grout the hole. If the hole has collapsed after removing the CPT, then hollow CPT rods and a sacrificial tip will be used to grout the hole. The PVC pipe or CPT rods will be pushed to the bottom of the hole. Grout will then be pumped to the bottom of the hole as the PVC pipe or CPT rods are withdrawn.

Refer to SOP No. 028 (Well and Boring Abandonment).

5. SITE CHARACTERIZATION AND ANALYSIS PENETROMETER SYSTEM

5.1 MATERIALS

SCAPS cone penetrometer and laser induced fluorescence (LIF) technology requires the use of a specialized 20-ton truck. The truck has two separate enclosed compartments. Each compartment is temperature controlled and monitored for air quality. The two rooms are the data acquisition and processing room, and the hydraulic ram/rod handling room. Approximately 20 ft of overhead clearance is required to fully extend the hydraulic ram and allow for leveling jack movement.

All materials required to complete SCAPS analysis are provided by the subcontractor to include cone penetrometer equipment. All hydraulic equipment, SCAPS rods, nitrogen lasers, etc. are included within the vehicle. A decontamination water source and a source of water for mixing the grout are required.

Water sources for equipment decontamination must be approved by the Project Manager prior to arrival of the SCAPS equipment. Information required for the water source includes: water source, manufacturer/owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analysis over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives will be the only additive allowed, and its use must be approved by the Project Manager prior to the arrival of the SCAPS equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product number, product description, and intended use for the product.

Portland Type II cement will be used for grout (refer to SOP No. 019).

5.2 HYDRAULIC PUSHING AND SAMPLING

The objective of the SCAPS technique is to allow grab samples and stratigraphic information to be collected at a selected site to facilitate subsurface characterization and for analysis of potential contaminants. The analytical results obtained can also be used to determine the placement of monitoring wells. At the same time, it is possible to install a small diameter well for sampling purposes. Refer to SOP No. 019 (Monitoring Well Installation). If a well will not be installed, the borehole can be grouted as the equipment is removed.

A site geologist will be present during all installation and sampling procedures and will fully document all procedures and soil characteristics in the Field Logbook (refer to SOP No. 016).

The site geologist will have on hand, at a minimum, a copy of the approved Health and Safety Plan, this SOP, the Field Investigation Work Plan, a hand lens (10X), a standard color chart, and a grain-size chart.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole SCAPS equipment.

Surface runoff or other fluids will not be allowed to enter any DPT location or well during or after direct-push activities.

The subcontractor will use the equipment specific guidelines for installation of the SCAPS DPT equipment. Prior to SCAPS field activities, calibration soil samples will be collected and analyzed in order to determine the LIF sensor fluorescence threshold and detection limits for the site.

SCAPS LIF technology uses a pulsed nitrogen laser coupled with an optical detector to make fluorescence measurements via optical fibers. The LIF sensor is mounted on a cone penetrometer probe so that soil classification data and fluorescence data are collected simultaneously. The laser consumes nitrogen gas, which is supplied from cylinders stored on the accompanying trailer.

The SCAPS CPT sensors are used to gather stratigraphic information. See Section 4 for CPT operating procedures.

Target interval samples can be collected during SCAPS hole advancement using direct push sampling techniques such as Geoprobe® or Hydropunch® (Section 3).

5.3 DECONTAMINATION

Decontamination of SCAPS equipment is automated after initialization by a field team member. A pressurized hot water system is used to decontaminate the push rods as they are retracted from the ground. The SCAPS vehicle is equipped with a decontamination collar mounted to the bottom that cleans the rods. The decontamination water is removed by vacuum and transferred to a storage drum prior to disposal or treatment. A trailer attached to the back of the vehicle contains the water pump, heater for decontamination, and decontamination water containment drum.

Worker exposure is reduced by minimizing contact with contaminated media.

Refer to SOP No. 005 (Decontamination).

5.4 ABANDONMENT

SCAPS automatically grouts the penetrometer cavity as the rods are removed. The grout is pumped at high pressure through a 0.25-in. diameter tube in the center of the penetrometer rods. The tip is sacrificed at the bottom of the cavity to allow release of the grout.

A trailer attached to the back of the vehicle contains the 300-gal grout mixing bin and pump.

If the automatic grout feed does not work, the cavity will be manually filled with grout.

Abandonment of SCAPS generated borehole will meet the standards established under SOP No. 028 (Well and Boring Abandonment).

6. MAINTENANCE

Not applicable.

7. PRECAUTIONS

Refer to the site-specific Health and Safety Plan for discussion of hazards and preventive measures during intrusive activities.

8. REFERENCES

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Standard Operating Procedure No. 048
for
Low-Flow Sampling

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Revision 0
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CONTENTS

1. GROUNDWATER SAMPLING BY LOW-FLOW PURGE AND SAMPLING METHOD USING DEDICATED PUMPS

1.1 SCOPE OF APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to establish the protocol for collecting groundwater samples using dedicated pump systems. The procedure is designed to permit the collection of groundwater samples with minimum turbidity.

1.2 EQUIPMENT/MATERIALS

- Work Plan.
- Well construction data, location map, and field data from last sampling event.
- Field logbook and Field Record of Well Gauging, Purging, and Sampling forms (Figure SOP048-1).
- Electric water level measuring device, 0.01 ft accuracy for monitoring water level during pumping operations.
- Pumps: adjustable rate, submersible pumps constructed of stainless steel and Teflon®.
- Tubing: Teflon or Teflon-lined polyethylene must be used to collect samples for organic analysis. For samples collected for inorganics analysis, Teflon or Teflon-lined polyethylene tubing will be used.
- Flow measurement supplies (e.g., graduated cylinder and stop watch).
- Power source (generator, etc.).
- Water quality indicator parameter monitoring instruments—pH, turbidity, specific conductance, and temperature. Optional indicators—Eh and dissolved oxygen.
- Flow-through cell (preferred) or clean container for water quality probes.
- Decontamination supplies (for monitoring instrumentation).
- Sample bottles and sample preservation supplies (as required by the analytical methods).
- Sample tags or labels.
- Cooler with bagged ice for sample bottles.
- Drum for purge water containment.

1.3 PRELIMINARY SITE ACTIVITIES

The following site activities are required prior to performing well purging and groundwater sampling. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate: site name, project number, field personnel, well identification, weather conditions, date and time, equipment used, and quality assurance/quality control data for field instrumentation.

- Check well for damage or evidence of tampering, record pertinent observations in field logbook and sampling form.

- Lay out sheet of polyethylene for monitoring and sampling equipment.

- Unlock well and remove well cap (if applicable).

- Measure VOCs with an ionization detector (flame or photo) instrument at the rim of the well and in the breathing zone, and record the readings in the field logbook and the sampling form.

- Measure and record the height of protective casing above the concrete pad or ground surface, as appropriate. This reading is compared to that recorded during well installation as an indication of possible well damage or settling that may have occurred.

- Dedicated sampling pumps should be positioned with the pump intake mid-point in the screened interval. If non-dedicated equipment is used, care will be taken to position pump or sampling hose intake at the screen mid-point.

- Measure and record the depth to water (to 0.01 ft) in the well to be sampled before purging begins. If the well casing does not have a reference point (usually a v-cut or indelible mark in the well casing), make one. If a reference point is made, it will be noted in the field logbook. Care should be taken to minimize disturbance of any particulate attached to the sides or at the bottom of the well. The depth to well bottom will be measured following the completion of sampling because of the potential to stir up sediment at the bottom of the well.

- Prepare the pump by checking electrical connections, discharge tubing, and motor (Grundfos Redi-Flo2). Locate the generator (if applicable) downwind of the well; connect the power converter to the generator and to the pump.

1.4 WELL PURGING AND SAMPLING PROCEDURE

The following general procedure should be followed to obtain representative groundwater samples. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate, prior to purging: purge date and time, purge method, and total well depth.

- Connect the flow-through cell or clean container containing the instrumentation header to the pump discharge and begin purging the well at 0.2-0.5 L/min, unless a different purge rate has been previously established for that well. Fill the flow cell completely. Care should be taken not to cause entrapment of air in the system. Record the purge start time and purge rate.

- Establish that the water level has not dropped significantly such that the pump is dry (bubbles in discharge) or water is heard cascading down the inside of the well. Ideally, the pump rate should cause little or no water level drawdown in the well (>0.5 ft and the water level should stabilize). The water level should be monitored every 3-5 minutes (or as appropriate) during pumping. Record pumping rate adjustments and depths to water. Pumping rates should, if needed, be reduced to the minimum capabilities of the pump (e.g., 0.1-0.2 L/min) to avoid pumping the well dry and/or to ensure stabilization of indicator parameters. If water levels continue to drop with the pump on the lowest flow rate, the pump will be shut off and the well will be allowed to recharge to prevent the well from going dry. **The well will not be purged to dryness prior to sampling to prevent erroneous field parameters and groundwater samples.** Sampling will commence as soon as the well has recharged to a sufficient level to collect the appropriate volume of samples with the pump.

- During purging of the well, monitor the water quality indicator parameters (turbidity, temperature, specific conductance, pH, etc.) every 3-5 minutes (or as appropriate). Record purge rate, volume purged, depth to water, water quality indicator parameters values, and clock time at 3- to 5-minute intervals in field logbook and sampling record. Purging of the standing well water is considered complete when three consecutive readings of the water quality indicator parameters agree within approximately 10 percent. Turbidity readings consistently below 10 nephelometric turbidity units (NTU) are considered to represent stabilization of discharge water for this parameter. If the parameters have stabilized, but the turbidity is not in the range of the 10 NTU goal, the pump flow rate should be decreased and measurement of the parameters should continue every 3-5 minutes.

- Purge water at a well will be containerized if a well has exceeded the MEG or MCL in previous sampling events. Any purge water that is collected will be treated at the groundwater treatment plant.

- Prior to sampling, disconnect the discharge tubing from the flow-through cell. If the water discharged by the pump is silty, wait for the water to clear before sampling. Ensure that bubbles are not observed in the discharge tubing. Record pertinent observations in field logbook and sampling records.

- Begin filling sample containers by allowing the pump discharge to flow gently down the inside of the container with as little agitation or aeration as possible. Collect the samples in the order below, as applicable:

  — VOCs
  — Inorganics.

- VOC samples requiring pH adjustment will have their pH checked to assure that the proper pH has been obtained. This will require that a test sample be collected to determine the amount of preservative that needs to be added to the sample containers prior to sampling. Details on sample preservation are discussed in Section 1.5.

- Label each sample as collected. Those samples (VOCs, etc.) requiring cooling will be placed into an ice cooler for delivery to the laboratory. Inorganic samples, after preservation, do not need to be cooled.

- After collection of the samples, restore the dedicated pumping assembly to the well by hanging the tube, electric line, and support cable inside the well by the specially-designed PVC well cap assembly. Lock well.

- Complete remaining portions of Field Record of Well Gauging, Purging, and Sampling form (Figure SOP048-1) after each well is sampled, including sample date and time, total quantity of water removed, well sampling sequence, types of sample bottles used, sample identification numbers, preservatives used, parameters requested for analysis, and field observations of sampling event.

1.5 SAMPLE PRESERVATION

The following preservation procedures are examples of typical preservation protocols specific to the indicated analyses. Pre-preserved bottles will be used if possible. Minimum sample preservation requirements for each parameter group are summarized below:

- **VOCs—**Aqueous VOC samples must be collected as specified below. Each VOC sample is taken in duplicate:

  — Uncap the sample bottle, taking care not to touch the Teflon-faced septum. If the septum is contaminated in any way, it should be replaced.

  — Fill a sample bottle, preserve with HC1, and check the pH. Adjust the volume of HC1 to assure pH<2.

— Add the amount of HC1 determined in the above step, and fill the sample vial slowly from the tubing, minimizing air entrainment, until the vial slightly overflows.

— Place the Teflon-faced silicon rubber septum on the convex meniscus, Teflon side (shiny side) down and screw cap on.

— Invert the bottle, tap lightly, and check for air bubbles.

— If air bubbles are present, open the bottle, add sample to eliminate air bubbles, and reseal. Repeat this procedure until the bottle is filled and no air bubbles are detected.

— Place samples on ice until shipment.

- **Inorganics—**Fill the sample bottle, preserve the sample to pH<2 with nitric acid (HNO_3), seal container, and place sample on ice for shipment.

Disposable pipettes should be used to introduce chemicals into the samples if necessary. Chemicals used for preserving should be poured into a 150-ml beaker. They should not be drawn directly from the preservative bottles because the bottle may become contaminated. Measurements for pH and temperature should not be taken from the sample containers. When preserving samples to a required pH, pH paper should be used to check the resultant pH. The sample should be poured across the pH paper. Never place pH paper directly into sample.

NOTE: Shipping regulations limit the amount of preservative which can be added. For a 1-L sample, this is generally 1.5 ml of acid preservative.

1.6 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not affected the quality of the groundwater samples. All field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples will be collected for each sample delivery group (SDG) (an SDG may not exceed 20 samples) at the frequency noted:

- Field Duplicate—Required at a frequency of 10 percent per SDG.

- Matrix Spike/Matrix Spike Duplicate—Required at a frequency of 5 percent.

- Equipment Rinsate Blank—Required once prior to installation of dedicated pump systems.

- Source Water Blank—Required at a frequency of once per source per sampling event when equipment (rinsate) blank is required.

- Trip Blank—Required for VOC samples at a frequency of one per sample shipment.

1.7 DECONTAMINATION

Non-dedicated sampling equipment and field monitoring equipment will be decontaminated prior to use and following sampling of each well. This equipment will be decontaminated by the procedure listed below. Alternative procedures must be approved by the Project Manager prior to sampling event. Decontamination fluids will be collected in a 5-gal bucket and treated at the groundwater treatment plant.

The following decontamination procedure will be used:

- Flush the equipment with potable water
- Flush with non-phosphate detergent solution
- Flush with tap water to remove all of the detergent solution
- Flush with distilled/deionized water
- Flush with isopropyl alcohol
- Flush with distilled/deionized water.

It is recommended that the detergent and isopropyl alcohol used in the above sequence be used sparingly.

2. GROUNDWATER SAMPLING BY LOW-FLOW PURGE AND SAMPLING METHOD USING PERISTALTIC PUMPS

2.1 SCOPE OF APPLICATION

The purpose of this SOP is to establish the protocol for collecting groundwater samples using peristaltic pump systems. The procedure is designed to permit the collection of groundwater samples with minimum turbidity, and is intended to be used in conjunction with the analyses for the most common types of groundwater contaminants (VOCs and inorganic compounds).

2.2 EQUIPMENT/MATERIALS

- Work Plan.
- Well construction data, location map, field data from last sampling event.
- Field logbook and Field Record of Well Gauging, Purging, and Sampling forms (Figure SOP048-1).
- Water level measuring device, 0.01 ft accuracy (electronic preferred) for monitoring water level drawdown during pumping operations.
- Peristaltic pump.

- In-well tubing: Teflon or Teflon-lined polyethylene must be used to collect samples for organic analysis. For samples collected for inorganics analysis, Teflon or Teflon-lined polyethylene, PVC, Tygon, or polyethylene tubing may be used.

- Pump head tubing: Silicon tubing must be used to in the pump head assembly.

- Flow measurement supplies (e.g., graduated cylinder and stop watch).

- Power source (battery, etc.).

- Water quality indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Optional indicators – Eh and dissolved oxygen.

- Flow-through cell (preferred) or clean container for water quality probe.

- Decontamination supplies (for monitoring instrumentation).

- Sample bottles and sample preservation supplies (as required by the analytical methods).

- Sample tags or labels.

- Cooler with bagged ice for sample bottles.

- Drum for purge water containment.

2.3 PRELIMINARY SITE ACTIVITIES

The following site activities are required prior to performing well purging and groundwater sampling. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate: site name, project number, field personnel, well identification, weather conditions, date and time, equipment used, and quality assurance/quality control data for field instrumentation.

- Check well for damage or evidence of tampering, record pertinent observations in field logbook and sampling form.

- Unlock well and remove well cap (if applicable).

- Measure VOCs with an ionization detector (photo or flame) instrument at the rim of the well and in the breathing zone and record the readings in the field logbook and the sampling form.

- Measure and record the height of protective casing above the concrete pad, or ground surface, as appropriate. This reading is compared to that recorded during well installation as an indication of possible well damage or settling that may have occurred.

- Measure and record the depth to water (to 0.01 ft) in the well to be sampled before purging begins. If the well casing does not have a reference point (usually a v-cut or indelible mark in the well casing), make one. If a reference point is made, it will be noted in the field logbook. Care should be taken to minimize disturbance of any particulate attached to the sides or at the bottom of the well. The depth to well bottom will not be measured following the completion of sampling because of the potential to stir up sediment at the bottom of the well.

- Position the intake of the sampling hose at the mid-point of the screened interval.

- Prepare the pump by checking electrical connections and discharge tubing. Locate the battery downwind of the well; connect the peristaltic pump to the battery.

2.4 WELL PURGING AND SAMPLING PROCEDURES

The following general procedure should be followed to obtain representative groundwater samples. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate, prior to purging: purge date and time, purge method, and total well depth.

- Measure the water level with the pump in well before starting the pump. Begin purging the well at 0.3-0.5 L/min, unless a different purge rate has been previously established for that well.

- If well diameter permits, establish that the water level has not dropped significantly such that the pump is dry (air in discharge) or tubing suction is broken. Ideally, the pump rate should cause little or no water level drawdown in the well (>0.5 ft and the water level should stabilize). The water level should be monitored every 3-5 minutes (or as appropriate) during pumping. Care should be taken not to cause pump suction to be broken, or entrainment of air in the pump system. Record pumping rate adjustments and depths to water. Pumping rates should, if needed, be reduced to the minimum capabilities of the pump (e.g., 0.3 L/min) to avoid pumping the well dry and/or to ensure stabilization of indicator parameters. If water levels continue to drop with the pump on the lowest flow rate, the pump will be shut off and the well will be allowed to recharge to prevent the well from going dry. **The well will not be purged to dryness prior to sampling to prevent erroneous field parameters and groundwater samples.** Sampling will commence as soon as the well has recharged to a sufficient level to collected the appropriate volume of samples with the pump.

- During purging of the well, monitor the field indicator parameters (turbidity, temperature, specific conductance, pH, etc.) every 3-5 minutes (or as appropriate). Purging of the standing well water is considered complete when three consecutive readings of the water quality indicator parameters agree within approximately 10 percent. Turbidity readings consistently below 10 NTU are considered to represent stabilization of discharge water for this parameter. If the parameters have stabilized, but the turbidity is not in the range of the 10 NTU goal, the pump flow rate should be decreased and measurement of the parameters should continue every 3-5 minutes.

- Purge water at a well will be containerized if a well has exceeded the MEG or MCL in previous sampling events. Any purge water that is collected will be treated at the groundwater treatment plant.

- Prior to sampling, disconnect the sample discharge tubing from the flow-through cell. If the water discharged by the pump is silty, wait for the water to clear before sampling. Ensure that bubbles are not observed in the discharge tubing.

- Collect groundwater samples directly from the silicon tubing into preserved (when appropriate) sample containers. Begin filling sample containers from the pump discharge, allowing the water to fill the containers by allowing the pump discharge to flow gently down the inside of the container with as little agitation or aeration as possible. Collect the samples in the order below, as applicable:

  — VOCs
  — Inorganics.

- VOC samples requiring pH adjustment will have their pH checked to assure that the proper pH has been obtained. This will require that a test sample be collected to determine the amount of preservative that needs to be added to the sample containers prior to sampling. Detail on sample preservation are discussed in Section 2.5.

- Label each sample as collected. Those samples (VOCs, etc.) requiring cooling will be placed into an ice cooler for delivery to the laboratory. Inorganic samples, after preservation, do not need to be cooled.

- After collection of the samples, restore the dedicated tubing assembly to the well by hanging the tube inside the well by the specially-designed PVC well cap assembly. Lock well.

- Complete remaining portions of Field Record of Well Gauging, Purging, and Sampling form (Figure SOP048-1) after each well is sampled, including: sample date and time, total quantity of water removed, well sampling sequence, types of sample bottles used, sample identification numbers, preservatives used, parameters requested for analysis, and field observations of sampling event.

- The silicon tubing used in the peristaltic pump will be changed after use at each well.

2.5 SAMPLE PRESERVATION

The following preservation procedures are examples of typical preservation protocols specific to the indicated analyses. Pre-preserved bottles will be used if possible. Minimum sample preservation requirements for each parameter group are summarized below:

- **VOCs—**Aqueous VOC samples must be collected as specified below. Each VOC sample is taken in duplicate:

  — Uncap the sample bottle, taking care not to touch the Teflon-faced septum. If the septum is contaminated in any way, it should be replaced.

  — Fill a sample bottle, preserve with HCL, and check the pH. Adjust the volume of HCL to assure pH<2.

  — Add the amount of HCL determined in the above step, and fill the sample vial slowly from the tubing, minimizing air entrainment, until the vial slightly overflows.

  — Place the Teflon-faced silicon rubber septum on the convex meniscus, Teflon side (shiny side) down, and screw cap on.

  — Invert the bottle, tap lightly, and check for air bubbles.

  — If air bubbles are present, open the bottle, add sample to eliminate air bubbles, and reseal. Repeat this procedure until the bottle is filled and no air bubbles are detected.

  — Place samples on ice until shipment.

- **Inorganics—**Fill the sample bottle, preserve the sample to pH<2 with nitric acid (HNO_3), seal container, and place sample on ice for shipment.

Disposable pipettes should be used to introduce chemicals into the samples if necessary. Chemicals used for preserving should be poured into a 150-ml beaker. They should not be drawn directly from the preservative bottles because the bottle may become contaminated. Measurements for pH and temperature should not be taken from the sample containers. When preserving samples to a required pH, pH paper should be used to check the resultant pH. The sample should be poured across the pH paper. Never place pH paper directly into sample.

NOTE: Shipping regulations limit the amount of preservative which can be added. For a 1-L sample, this is generally 1.5 ml of acid preservative.

2.6 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not affected the quality of the groundwater samples. All field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples will be collected for each SDG (an SDG may not exceed 20 samples) at the frequency noted:

- Field Duplicate—Required at a frequency of 10 percent per SDG
- Matrix Spike/Matrix Spike Duplicate—Required at a frequency of 5 percent
- Equipment (Rinsate) Blank—Required once prior to installation of dedicated sample tubing
- Source Water Blank—Required at a frequency of one per source per sampling event
- Trip Blank—Required for VOC samples at a frequency of one per sample shipment.
- Temperature Blank—Required at a frequency of once per sample shipment container.

2.7 DECONTAMINATION

Non-dedicated sampling and field monitoring equipment will be decontaminated prior to use and following sampling of each well. This equipment will be decontaminated by the procedure listed below. Alternate procedures must be approved by the Project Manager prior to the sampling event. Decontamination fluids will be collected in a 5-gal bucket and treated at the groundwater treatment plant.

The following decontamination procedure will be used:

- Flush the equipment with potable water
- Flush with non-phosphate detergent solution
- Flush with tap water to remove all of the detergent solution
- Flush with distilled/deionized water
- Flush with isopropyl alcohol
- Flush with distilled/deionized water.

It is recommended that the detergent and isopropyl alcohol used in the above sequence be used sparingly.

3. SURFACE WATER AND LEACHATE SEEP SAMPLING PROCEDURE

3.1 SCOPE OF APPLICATION

The purpose of this SOP is to establish the protocol for collecting surface water and leachate seep samples. The procedure is designed to permit the collection of representative surface water and leachate seep samples, and has been adapted from the procedure outlined in the Work Plan. This SOP is suitable for collecting surface water and seep samples requiring analyses for the most common types of surface water contaminants (VOCs and inorganic compounds).

3.2 EQUIPMENT/MATERIALS

- Work Plan.
- Location map, field data from last sampling event.
- Field logbook and Field Record of Surface Water and Sediment Sampling forms (Figure SOP048-2).
- Water quality indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Optional indicators – Eh and dissolved oxygen.
- Decontamination supplies (for monitoring instrumentation).
- Dedicated, pre-cleaned 1-L wide-mouth or volatile organic analyte sample container (for sample collection).
- Sample bottles and sample preservation supplies (as required by the analytical methods).
- Sample tags or labels.
- Cooler with bagged ice for sample bottles.

3.3 PRELIMINARY SITE ACTIVITIES

The following site activities are required prior to performing surface water or leachate seep sampling. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate: site name, project number, field personnel, sample station identification, weather conditions, date and time, equipment used, and quality assurance/quality control data for field instrumentation.

- Visually inspect sample station for evidence of changes in physical condition; record pertinent observations in field logbook and sampling form.

- Measure VOCs with a flame ionization detector instrument in the breathing zone and record the reading in the field logbook and sampling form.

3.4 SAMPLING PROCEDURE

The technique for surface water and leachate seep sampling must be selected after addressing such items as:

- Depth of waterbody
- Depth of sample
- Stratification
- Seasonal variations
- Analytical parameters of interest.

The following general procedure should be used to obtain representative surface water and leachate seep samples. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate, prior to sampling: date and time, sample method, and sample depth.

- Collect the sample from the surface water, within several tenths of a foot of the streambed, by immersing a new, dedicated 1-L glass or volatile organic analyte sample container into the waterbody. If a stream is being sampled, collect the sample upstream of the sampler with the opening of the sampling device oriented upstream but avoiding floating debris.

- Directly fill the appropriate sample containers from the 1-L or volatile organic analyte sampling device.

- Collect the samples in the order below, as applicable:

  — VOCs
  — Inorganics.

- Water sample containers are generally filled directly from the source or sampler without special considerations. The exception is the collection of aqueous VOC samples requiring pH adjustment. VOC samples will have their pH checked to assure that the proper pH has been obtained. This will require that a test sample be collected to determine the amount of preservative that needs to be added to the sample containers prior to sampling. Details on sample preservation methods are discussed in Section 3.6.

- Label each sample as collected. Those samples (VOCs, etc.) requiring cooling will be placed into an ice cooler for delivery to the laboratory. Inorganic samples, after preservation, do not need to be cooled.

- Measure water quality indicator parameters, if possible, by direct immersion of instrument probes into the waterbody immediately following sample collection. If direct measurement is not possible, measure these parameters from water remaining in the sampling device or another sample bottle. Record this information in the field logbook and sample data record.

- Complete remaining portions of the Field Record of Surface Water and Sediment Sampling form (Figure SOP048-2) after each station is sampled, including: time of sample collection, types of sample bottles used, sample identification numbers, preservatives used, parameters requested for analysis, and field observations of sampling event.

3.5 SAMPLE PRESERVATION

The following preservation procedures are examples of typical preservation protocols specific to the indicated analyses. Minimum sample preservation requirements for each parameter group are summarized below:

- **VOCs—**Aqueous VOC samples must be collected as specified below. Each sample is taken in duplicate:

  — Uncap the sample bottle, taking care not to touch the Teflon-faced septum. If the septum is contaminated in any way, it should be replaced.

  — Fill a sample bottle, preserve with HC1, and check the pH. Adjust the volume of HC1 to assure pH<2.

  — Add the amount of HC1 determined in the above step, and fill the sample vial slowly from the 1-L container, minimizing air entrainment, until the vial slightly overflows.

  — Place the Teflon-faced silicon rubber septum on the convex meniscus, Teflon side (shiny side) down and screw cap on.

  — Invert the bottle, tap lightly, and check for air bubbles.

  — If air bubbles are present, open the bottle, add sample to eliminate air bubbles, and reseal. Repeat this procedure until the bottle is filled and no air bubbles are detected.

  — Place samples on ice until shipment.

- **Inorganics—**Fill the sample bottle, preserve the sample to pH<2 with nitric acid (HNO_3), seal container, and place sample on ice for shipment.

Disposable pipettes should be used to introduce chemicals into the samples. Chemicals used for preserving should be poured into a 150-ml beaker. They should not be drawn directly from the preservative bottles because the bottle may become contaminated. Measurements for pH and temperature should not be taken from the sample containers. When preserving samples to a required pH, pH paper should be used to check the resultant pH. The sample should be poured across the pH paper. Never place pH paper directly into sample.

NOTE: Shipping regulations limit the amount of preservative which can be added. For a 1-L sample, this is generally 1.5 ml of acid preservative.

3.6 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not affected the quality of the surface water and leachate seep samples. All field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples will be collected for each SDG (an SDG may not exceed 20 samples) at the frequency noted:

- Field Duplicate—Required at a frequency of 10 percent per SDG.
- Matrix Spike/Matrix Spike Duplicate—Required at a frequency of 5 percent.
- Equipment (Rinsate) Blank—Required at a frequency of once per day per media sampled.
- Source Water Blank—Required at a frequency of once per source per sampling event when equipment (rinsate) blank is required.
- Trip Blank—Required for VOC samples at a frequency of one per sample shipment.

3.7 DECONTAMINATION

Field monitoring equipment will be decontaminated prior to use and following sampling of each station by the procedure listed below. Laboratory pre-cleaned, dedicated 1-L glass sample collection containers are used once and discarded and, therefore, do not undergo any decontamination. Decontamination fluids will be collected in a 5-gal bucket and treated at the groundwater treatment plant.

The following decontamination procedure will be used:

- Flush the equipment with potable water
- Flush with non-phosphate detergent solution
- Flush with tap water to remove all of the detergent solution
- Flush with distilled/deionized water
- Flush with isopropyl alcohol
- Flush with distilled/deionized water.

It is recommended that the detergent and isopropyl alcohol used in the above sequence be used sparingly.

4. REFERENCES

U.S. Environmental Protection Agency. 1996. Groundwater Issue-Low Flow Sampling (Minimal Drawdown) Groundwater Sampling Procedures. April.

FIELD RECORD OF WELL GAUGING, PURGING, AND SAMPLING

Site Name:		Project Number:	
Well ID:		Well Lock Status:	
Well Condition:		Weather:	

Gauge Date:		Gauge Time:	
Sounding Method:		Measurement Ref:	
Stick Up/Down (ft):		Well Diameter (in.):	

Purge Date:		Purge Time:	
Purge Method:		Field Personnel:	
Ambient Air VOCs (ppm):		Well Mouth VOCs (ppm):	

WELL VOLUME

A. Well Depth (ft):		D. Well Volume/ft (L):	
B. Depth to Water (ft):		E. Well Volume (L) (C*D):	
C. Liquid Depth (ft) (A-B)		F. Three Well Volumes (L) (E*3):	

G. Measurable LNAPL? Yes _____ /ft No _____

Parameter	Beginning	1	2	3	4	5
Time (min.)						
Depth to Water (ft)						
Purge Rate (L/min)						
Volume Purged (L)						
pH						
Temperature (°C)						
Conductivity (µmhos/cm)						
Dissolved Oxygen (mg/L)						
Turbidity (NTU)						
eH (mV)						

Total Quantity of Water Removed (L):			
Samplers:		Sampling Time (Start/End):	
Sampling Date:		Decontamination Fluids Used:	
Sample Type:		Sample Preservatives:	
Sample Bottle IDs:			
Sample Parameters:			

Figure SOP048-1.

FIELD RECORD OF WELL GAUGING, PURGING, AND SAMPLING

Site Name:	Project Number:	Date:
Well ID:	Field Personnel:	

Parameter	6	7	8	9	10	11
Time (min.)						
Depth to Water (ft)						
Purge Rate (L/min)						
Volume Purged (L)						
pH						
Temperature (°C)						
Conductivity (µmhos/cm)						
Dissolved Oxygen (mg/L)						
Turbidity (NTU)						
eH (mV)						

Parameter	12	13	14	15	16	17
Time (min.)						
Depth to Water (ft)						
Purge Rate (L/min)						
Volume Purged (L)						
pH						
Temperature (°C)						
Conductivity (µmhos/cm)						
Dissolved Oxygen (mg/L)						
Turbidity (NTU)						
eH (mV)						

Comments and Observations:

Figure SOP048-1.

FIELD RECORD OF SURFACE WATER AND SEDIMENT SAMPLING

Site Name:			Project Number:
Sample Location ID:			Date:
Time:	Start:	End:	Sample Team Members:

SURFACE WATER INFORMATION

Type of Surface Water:
( ) Stream ( ) River
( ) Pond/Lake ( ) Seep

Water Depth and Sample Location _____ (ft)

Depth of Sample from Top of Water _____ (ft)

Equipment Used for Collection:
( ) None, Grab into Bottle
( ) Bomb Sampler
( ) Pump _____

Decontamination Fluids Used:
( ) Isopropyl Alcohol
( ) ASTM Type II Water
( ) Deionized Water
( ) Liquinox Solution
( ) Hexane
( ) HNO_3 Solution
( ) Potable Water
( ) None

Water Quality Parameters
( ) Temperature ____
( ) Conductivity ___ µmhs/cm
( ) pH ____ units
( ) Dissolved oxygen ____ mg/L
( ) Turbidity ____ NTU
( ) Eh ____ mv

Velocity Measurements Obtained? ( ) No ( ) Yes, See Flow Measurement Data Record

Field QC Data: ( ) Field Duplicate Collected Sample Location Sketch: Method Used:
Duplicate ID _____ ( ) Yes ( ) Winkler
( ) MS/MSD ( ) No ( ) Probe

SEDIMENT INFORMATION

Type of Sample Collected:
( ) Discrete
( ) Composite

Sediment Type:
( ) Clay
( ) Sand
( ) Organic
( ) Gravel

Equipment Used for Collection:
( ) Gravity Corer
( ) Stainless Steel Split Spoon
( ) Dredge
( ) Hand Spoon/Trowel
( ) Aluminum Pans
( ) Stainless Steel Bucket
( ) _____

Decontamination Fluids Used:
( ) Isopropyl Alcohol
( ) ASTM Type II Water
( ) Deionized Water
( ) Liquinox Solution
( ) Hexane
( ) HNO_3 Solution
( ) Potable Water
( ) None

Sample Observations:
( ) Odor _____
( ) Color

Field QC Data: ( ) Field Duplicate Collected ( ) MS/MSD
Duplicate ID _____

SAMPLES COLLECTED

Check if Required at this Location	Matrix: Surface Water	Matrix: Sediment	Check if Preserved with Acid/Base	Volume Required	Check if Sample Collected	Sample Bottle IDs			

NOTES/SKETCH

Figure SOP048-2.

Standard Operating Procedure No. 059
for
Field Logbook

Prepared by

EA Engineering, Science, and Technology, Inc.
225 Schilling Circle, Suite 400
Hunt Valley, Maryland 21031

Revision: 1
November 2012

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1. SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for recording field survey and sampling information in the Field Logbook.

2. MATERIALS

The following materials may be required:

- Field Logbook (Teledyne 415 Level Book, or equivalent)[1]
- Indelible ink pen (e.g., Sharpie®).

3. PROCEDURE

All information pertinent to a field survey or sampling effort will be recorded in a bound logbook. Each page/form will be consecutively numbered, dated, and signed. All entries will be made in indelible ink, and all corrections will consist of line-out deletions that are initialed and dated. The person making the correction will provide a brief explanation for the change. Entries are factual only. No personal opinions should be entered.

There should be no blank lines on a page. A single blank line or a partial blank line (i.e., at the end of a paragraph) should be lined to the end of the page. If only part of a page is used, the remainder of the page should have an "X" drawn across it. The bottom of each page must be signed and dated by the field personnel entering the information.

At a minimum, entries in the Field Logbook will include but not be limited to the following:

- Date.
- Project number and project name.
- Name and address of field contact.
- Identification of sample crew members.
- Documentation should include model numbers of equipment used (e.g., drilling rigs) and calibration (if applicable). Each day's entry should begin with time onsite, who is onsite (including observers other than the sampling crew), brief description of what work will be performed that day and how, and the weather.

[1] Pre-printed, bound forms are approved as well. See SOP No. 016 for recommended content and format.

- If samples are being taken in or near tidal waters, the time of high and low tide for the site should be determined from local gauges or tables and recorded.

- References such as maps of the sampling site.

- Times of key daily milestones should be entered (e.g., time borings began, times personnel arrived and left site, times subcontractors arrived and left site, etc.). Time should be recorded in the left-hand margin on the page in military time.

- Sample-specific information:

  — Unique, sequential field sample number

  — Purpose of sampling

  — Location, description, and log of photographs of each sampling point

  — Details of the sample site (e.g., elevation of the casing, casing diameter and depth, integrity of the casing, etc.)

  — Documentation of procedures for preparation of reagents or supplies which become an integral part of the sample (e.g., filters and absorbing reagents)

  — Type of media of sample (e.g., groundwater, surface water, soil, sediment, and product)

  — Suspected waste composition

  — Number and volume of sample taken

  — Sampling methodology, including distinction between grab and composite sample

  — Sample preservation

  — Date and time of collection

  — Collector's sample identification number(s)

  — Sample shipment (e.g., name of the laboratory and cartage agent: Federal Express, United Parcel Service, etc.)

  — Field observations (e.g., oily sheen on groundwater sample, incidental odors, soil color, grain size, plasticity, moisture content, layering, Unified Soil Classification System classification, etc.)

— Any field measurements made (e.g., pH, conductivity, explosivity, water depth, organic vapor analyzer readings, etc.)

— Signature and date by the personnel responsible for observations

— Decontamination procedures.

Sampling situations vary widely. No general rules can specify the extent of information that must be entered in a Field Logbook. However, records should contain sufficient information so that someone can reconstruct the sampling activity without relying on the sampler's memory. Further, the project work plan or field sampling plan should be reviewed to identify additional specific information or requirements that should be included in the Field Logbook.

The Project Manager will keep a master list of all Field Logbooks assigned to the Sampling Team Leaders. One Field Logbook kept by the Project Manager will be a master site log of daily activities and will contain the list of Field Logbooks assigned to Sampling Team Leaders.

Project name and number should be clearly marked on the outside cover using indelible ink. If more than one Field Logbook exists for the project, then the number of the Field Logbook should also be clearly marked on the outside cover.

4. MAINTENANCE

At the end of the field sampling effort, the Field Logbook should be scanned and filed in the electronic file for the project and maintained according to the EA Records Retention Policy or contract requirements.

5. PRECAUTIONS

None.

6. REFERENCES

EA Engineering, Science, and Technology, Inc. 2007. Standard Operating Procedure No. 016 for Surface Water, Groundwater, and Soil/Sediment Field Logbooks. August.

U.S. Environmental Protection Agency. 1980. *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans*, QAMS-005/80.

———. 1990. *Sampler's Guide to the Contract Laboratory Program*. EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C. December.

———. 1991. *User's Guide to the Contract Laboratory Program*. EPA/540/O-91/002, Directive 9240.0-01D. Office of Emergency and Remedial Response. January.

Standard Operating Procedure No. 061
for
Geographic Information System
Data Management and Deliverables

Prepared by

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Revision: 0
April 2013

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1. INTRODUCTION

1.1 PURPOSE

The purpose of this standard operating procedure is to provide EA personnel responsible for Geographic Information System (GIS) activities within the company the guidance necessary to maintain company-wide uniformity of GIS setup, data storage, file naming conventions, products, and deliverables.

1.2 SCOPE

This standard operating procedure covers EA's basic requirements for creating, managing, and deliverables for a GIS project or GIS subtask within a project.

1.3 DEFINITIONS

- FGDC = Federal Geographic Data Commission
- ESRI = Environmental Systems Research Institute
- MXD = ESRI GIS project file extension.

1.4 RESPONSIBILITY

EA personnel assigned GIS tasks will be responsible for adhering to the standards included in this standard operating procedure and for ensuring that all GIS deliverables meet any and all contract specifications. In accordance with EA Quality Control procedures and published Senior Technical Review guidance, GIS deliverables should be reviewed by an appropriate Senior Technical Reviewer.

1.5 RELATED STANDARD OPERATING PROCEDURES

None.

2. EQUIPMENT

2.1 SOFTWARE

ESRI software (ArcInfo, ArcDesktop, ArcMap, and ArcEditor) Version 9.3 or newer is EA's standard GIS software. This also includes the ESRI suite of extensions (spatial analyst, 3D analyst, Business analyst, Network analyst, etc.) and any client-specific software requirements for projects. It is EA's policy to wait to update to a new version of GIS software until the first service pack or major revision of that software is released. However, this policy is superseded when a client and/or project requires deliverables in a newer version of the software.

2.2 HARDWARE

Standard computer provided by EA.

3. PROCEDURE

There are multiple elements that must be considered when setting up an ESRI GIS project or GIS subtask within a project that will maintain uniformity and consistency for all projects company-wide. These elements look at how the project is stored, how it is set up, storage of data, what data are allowed in the GIS, and reliability of the data. Explanation of the above elements is provided in this section.

At the end of each section, there will be a "Required" and "Guidelines" entry. The "Required" entry will describe what shall be followed and the "Guidelines" entry will describe points to be considered but do not have to be followed.

3.1 GEOGRAPHIC INFORMATION SYSTEM DIRECTORY STRUCTURE

GIS project files reference the physical location of data (spatial and attribute) to correctly manage the information. Because of this, it is important that all spatial and attribute data be stored in a standardized manner. This will not only help ensure that GIS projects function years after they have been completed but also will help any individual who is new to the project.

The following examples illustrate the recommended directory tree and directory descriptions that should be used when setting up GIS projects. Not all directories and subdirectories included in the example need to be created if there will be no data of the type utilized by the project. Additionally, some clients may have their own requirements that may override the standard directory tree. This is the only scenario in which deviating from the standard is acceptable.

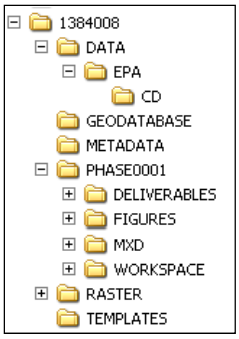
Required:

The example below represents EA's standard directory tree that shall be used to organize and create GIS projects.

GIS—This directory may be renamed according to how departments and offices store their projects.

GIS*PROJECT #*, e.g., GIS\1384008
GIS*PROJECT #*\DATA, e.g., GIS\1384008\DATA
GIS*PROJECT #*\DATA\CD, e.g., GIS\1384008\\DATA\CD
GIS*PROJECT #*\DATA\EMAIL, e.g., GIS\1384008\\DATA\EMAIL
GIS*PROJECT #*\DATA\WEB, e.g., GIS\1384008\\DATA\WEB
GIS*PROJECT #*\GEODATABASE, e.g., GIS\1384008\GEODATABASE
GIS*PROJECT #*\METADATA, e.g., GIS\1384008\METADATA
GIS*PROJECT #*\RASTER, e.g., GIS\1384008\RASTER
GIS*PROJECT #*\TEMPLATES, e.g., GIS\1384008\TEMPLATES
GIS*PROJECT #*\PHASE*NUMBER*, e.g., GIS\1384008\PHASE0001
GIS*PROJECT #*\PHASE*NUMBER*\DELIVERABLES, e.g., GIS\1384008\PHASE0001\DELIVERABLES

GIS*PROJECT #*\PHASE*NUMBER*\DELIVERABLES\DRAFT
– ex. GIS\1384008\PHASE0001\DELIVERABLES\DRAFT
GIS*PROJECT #*\PHASE*NUMBER*\DELIVERABLES\FINAL
– ex. GIS\1384008\PHASE0001\DELIVERABLES\FINAL
GIS*PROJECT #*\PHASE*NUMBER*\FIGURES
– ex. GIS\1384008\PHASE0001\FIGURES
GIS*PROJECT #*\PHASE*NUMBER*\FIGURES\PDF
– ex. GIS\1384008\PHASE0001\FIGURES\PDF
GIS*PROJECT #*\PHASE*NUMBER*\MXD
– ex. GIS\1384008\PHASE0001\MXD
GIS*PROJECT #*\PHASE*NUMBER*\WORKSPACE
– ex. GIS\1384008\PHASE0001\WORKSPACE.



A snapshot of what the directory tree would look like in Windows Explorer is displayed at right.

The following table describes the usage of the above directories.

Directory	Description	Note
GIS	GIS parent directory or directory where all projects are stored.	This directory may be renamed according to how departments and offices store their projects.
GIS**PROJECT#**	Subdirectory for a specific project.	This is the parent directory of the GIS project.
GIS\PROJECT#\ **GEODATABASE**	Geospatial database subdirectory. All features comprising project MXDs should be stored here.	This directory contains personal databases as well as file databases. This directory should only include final databases that are acceptable to be given to the client. This directory should not include any "working" files or data.
GIS\PROJECT#\ **RASTER**	Raster subdirectory. All raster types as well as geo-referenced imagery should be stored here.	This directory may contain subdirectories as needed. An example would be imagery broken out by year. NOTE: All imagery should be compressed.
GIS\PROJECT#\ **DATA**	DATA subdirectory. Incoming raw data, emails, etc. should be stored here.	This directory contains the original source files from clients or other sources. The directory should be organized in subdirectories based on source name, i.e., EPA. Subdirectories under source name should be organized by media to help in locating original files. An example would be EPA\CD\2010Wetland.zip.
GIS\PROJECT#\ **TEMPLATES**	Templates subdirectory. Stores project templates.	Templates stored here are used for figure creation.
GIS\PROJECT#\ **PHASE *number***	The PHASE *number* subdirectory. Organizes the project into its respective phases.	This is the parent directory for each phase.
GIS\PROJECT#\ PHASE *number***MXD**	The MXD subdirectory stores all MXDs used for DRAFT and FINAL products.	This directory stores the draft and final MXDs.
GIS\PROJECT#\ PHASE	Figures subdirectory. Organizes figures.	This is the parent directory for all figures.

Directory	Description	Note
*number***FIGURES**		
GIS\PROJECT#\ PHASE *number* \FIGURES**PDF**	PDF subdirectory. Contains PDF figures.	Contains PDF figures
GIS\PROJECT#\ PHASE *number*\ **DELIVERABLES**	Deliverables subdirectory. Is used for various stage deliverables to a client.	This directory contains the DRAFT and FINAL subdirectories for client deliverables.
GIS\PROJECT#\ PHASE *number*\ DELIVERABLES\ **DRAFT**	Draft subdirectory. Is used to compile draft deliverables.	This directory contains draft submittals to the client. Multiple drafts will have the submittal date incorporated in the zip file title.
GIS\PROJECT#\ PHASE *number*\ DELIVERABLES\ **FINAL**	Final subdirectory. Is used to compile final deliverables.	This directory contains final submittals to the client. Multiple finals will have the submittal date incorporated in the zip file title.
GIS\PROJECT#\ PHASE *number*\ **WORKSPACE**	The working files subdirectory. Working files, shape files, etc. to be deleted after incorporated into geospatial database should be stored here.	This directory is used for creating temporary files that are used to create final layers and data for the project draft and finals. Once files have been moved to MXDs and DATA storage areas they should be deleted from this directory.

Guidelines:

None.

3.2 GEOGRAPHIC INFORMATION SYSTEM PROJECT SETUP

There are three items required for every GIS project (MXD) creation before adding any data.

Required:

1. The MXD shall be set for storing the relative path to data sources rather than the full path. To store the relative path, go to the FILE pull down menu and click on "Document Properties." Click on "Data Source Option" and check the relative path box.

2. Data frames shall be set to the proper coordinate system dictated by the project. This ensures that data created or stored in the GIS will always have the correct coordinate system.

3. Third party extensions are not allowed if the final GIS deliverable includes the GIS MXD project file and requires the client to obtain/purchase the extension in order to recreate functionality and deliverables unless authorized by the client. An example would be the use of "Mapbooks," a third party extension, in the GIS project which automates the production of a tiled map series. Without Mapbooks, a client would not be able to reproduce a map series. This is only permitted when the client approves the extension and EA assists the client with obtaining the extension.

Guidelines:

- The use of multiple data frames and group layers is recommended for maintaining a logical easy-to-follow format in the MXD Table of Contents.

3.3 TEMPLATES

Templates are used to keep a consistent look across all maps and figures. EA has a set of standard templates that should be used on all projects company-wide (Appendix A). EA's standard templates should be used for all deliverables unless the client requires a specific layout. In cases where templates are customized for a specific client or projects, the template must be used for all deliverables to the project or client.

When creating templates for a project, the following is required.

Required:

- All templates should be stored in the TEMPLATES directory of the project.
- Each figure should include a North arrow, scale bar, file path, and a legend. The style and size of each should remain consistent throughout all figures.
- Source of data shall be included on all figures when the client requires.
- The scale will be set at an industry standard (i.e., 1 in. = 200 ft, 1 in. = 5 miles) and not an arbitrary scale (i.e., 1 in.:430 ft).
- Scale shall be consistent among figures in a report that depict geographical areas of the same size on paper of the same size, i.e., all figures in a report for the same geographical area presented on 8.5- × 11-in. paper should have the same scale.
- Label font size should be consistent within feature types regardless of the scale, i.e., all road names have the same font properties, and all stream names have the same font properties which may be different from the road layer. This means the font will be the same height per feature type on small and large scale figures when printed out.

Guidelines:

- The exterior neat line should allow for a minimum 0.5-in. margin for all sides if map is not intended to be bound in a report.
- A 0.75-in. margin is preferred on the binding edge.

3.4 GEOGRAPHIC INFORMATION SYSTEM DATA STORAGE

Regardless of the format that data are created or received in, it is required that a coordinate system be defined for all features whether they reside in a database, shape file, GRID, coverage, etc. Additionally, features, databases, and files shall be named in a fashion that is self describing to allow for easier identification of files.

All data, spatial or tabular, used in a GIS project for a final figure, map, or electronic deliverable are required to be stored in a geospatial database. The types of geospatial databases that can be used are Personal, File, or SDE.

Required:

- All data included in a final deliverable must be stored in one of the following geospatial database formats:

  — Personal (Microsoft Access)
  — File geospatial database
  — SDE geospatial database.

Guidelines:

- "Working" files can be in any format. A "working" file is any file that you create or are given to produce a new layer which is used in the final project.

- "Working" files will be discarded on a regular basis. Original or source files should not be altered but copied for revision.

- Many projects require a database schema specified by a client. An example would be the Spatial Data Standards, Facilities, Infrastructure, and Environment (SDSFIE). A client specification for data storage supersedes EA's standards.

3.5 ACQUIRING GEOGRAPHIC INFORMATION SYSTEM DATA

Large amounts of "acceptable" data can be found on the web at federal, state, and local websites. "Acceptable" data are data that include metadata describing the source, time of data collection, and how they were created. If metadata are not provided for a dataset, then the dataset should not be used. All data must be downloaded to the project directory for use in a project. Web links of GIS data through the web are not acceptable for any project that is final. Web-linked data can be used for working figures that will not be used for a deliverable.

Required:

- Data cannot be used if copyrighted, i.e., Google aerials.
- Data that are linked via the web have to be downloaded to the project directory.
- Data must have similar accuracy with overall GIS project.

Guidelines:

None.

3.6 METADATA

Metadata are an important aspect of any GIS project. Metadata become crucial in situations where a project's findings and/or data have been contested or questioned. It is required that all files used in a project will have metadata. If the file does not have an existing metadata file, then the GIS specialist will create one. The information to include is as follows.

Required:

- Source of data
- Coordinate and projection
- Description of data
- Time of data collection
- How it was created
- Who manipulated if applicable.

Guidelines:

This information is superseded when the client dictates the use of a metadata standard like the Federal Geographic Data Committee (FGDC) FGDC-STD-001-1998 adopted by all agencies. Additionally, this requirement can be disregarded for general maps, such as vicinity or location maps, that are created for ad-hoc purposes.

The exception to this is for projects that have general maps like "vicinity" or "location" maps and a basic site map. All EA generated layers must have metadata. Examples would be Global Positioning System collected data from the field, floodplain, or contour generation. All projects where GIS data are delivered to a client must have metadata

3.7 QUALITY ASSURANCE AND QUALITY CONTROL

Quality assurance and quality control should be integrated at all levels of the project. Specifics of EA's Quality Assurance/Quality Control Program can be found in the Corporate Quality Assurance Plan located on *Inside EA*. At a minimum, review by an appropriate Senior Technical Reviewer is required for all client deliverables. Additionally, the checks listed below should be followed:

Required:

CHECK	PDF	GIS FILES
Check directory structure	X	X
Check file naming conventions	X	X
Check files for projection	X	X
Check final files in data base	X	X
Check metadata	X	X
Check consistency between figures	X	X
Check source data are not streamed from external website		X

Guidelines:

With larger projects that span a large time period or have large and numerous datasets, PLTS should be considered for quality assurance/quality control reporting. PLTS can check data topology, data duplicates, polygon overlap, and database structure, to list a few. Other tools such as SDSFIE analyze tools can be used for quality assurance/quality control on SDSFIE data deliverables.

4. REFERENCES

Link to the Federal Geographic Data Committee standards
http://www.fgdc.gov/standards/projects/FGDC-standards-projects/metadata/base-metadata/index_html.

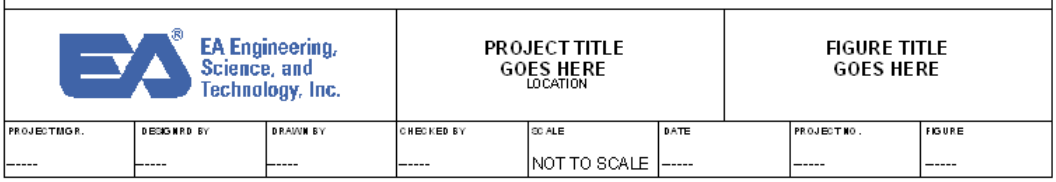
Appendix A

Engineering Style Templates

Engineering Style Portrait Template

Includes the following:

- EA Logo
- Project Title
- Figure Title
- Project Manager Initials
- Designed by Initials
- Drawn by Initials
- Checked by Initials Scale
- Date
- Project Number
- Figure Number
- File Path



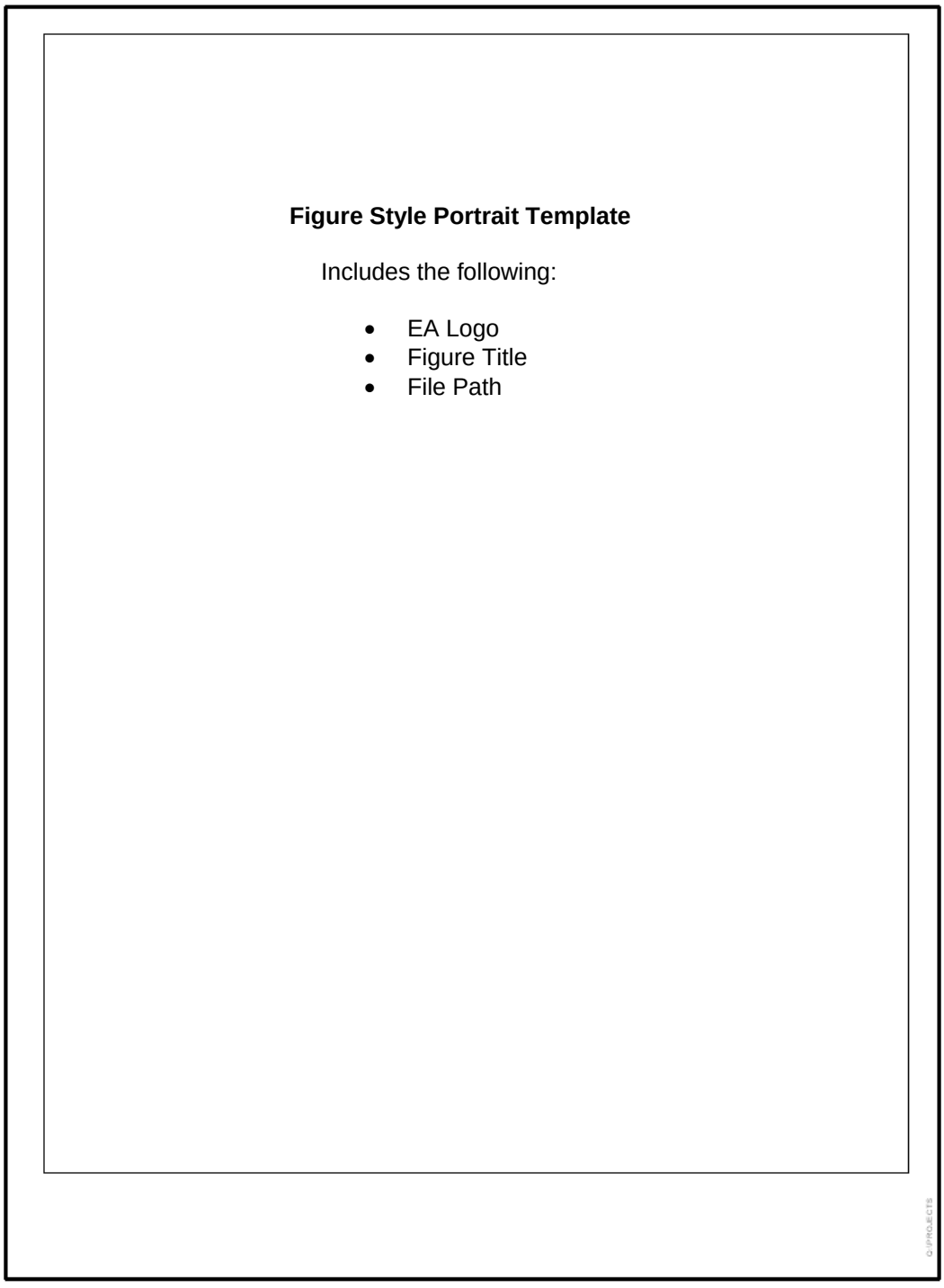


Figure Title

Engineering Style Landscape Template

Includes the following:

- EA Logo
- Project Title
- Figure Title
- Project Manager Initials
- Designed by Initials
- Drawn by Initials
- Checked by Initials Scale
- Date
- Project Number
- Figure Number
- File Path

EA Engineering, Science, and Technology, Inc.	PROJECT TITLES GOES HERE LOCATION	FIGURE TITLE GOES HERE	DESIGNED BY ----	DRAWN BY ----	DATE NOT TO SCALE	PROJECT NO. ----
			CHECKED BY ----	PROJECT MGR. ----	SCALE NOT TO SCALE	FIGURE ----

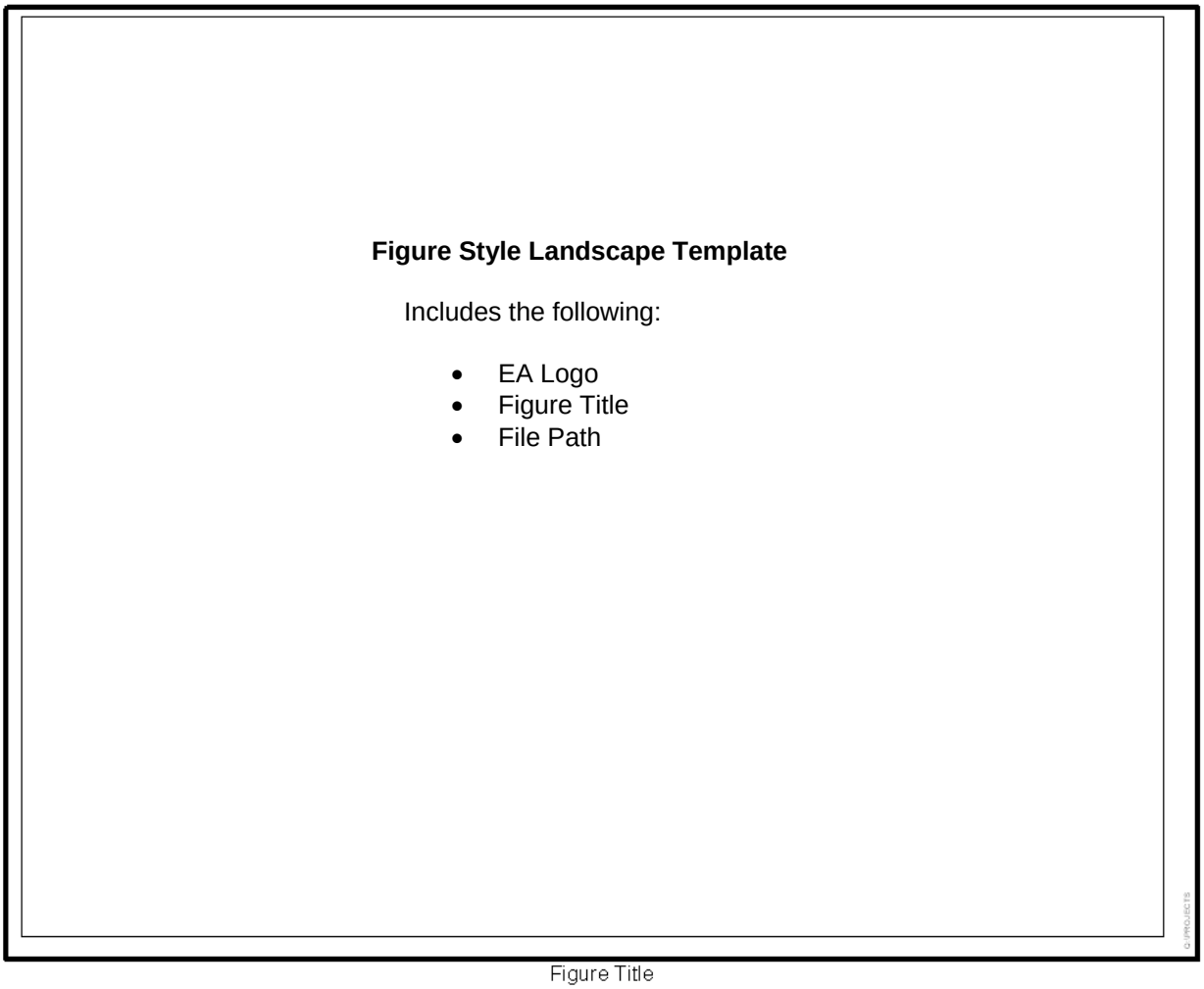
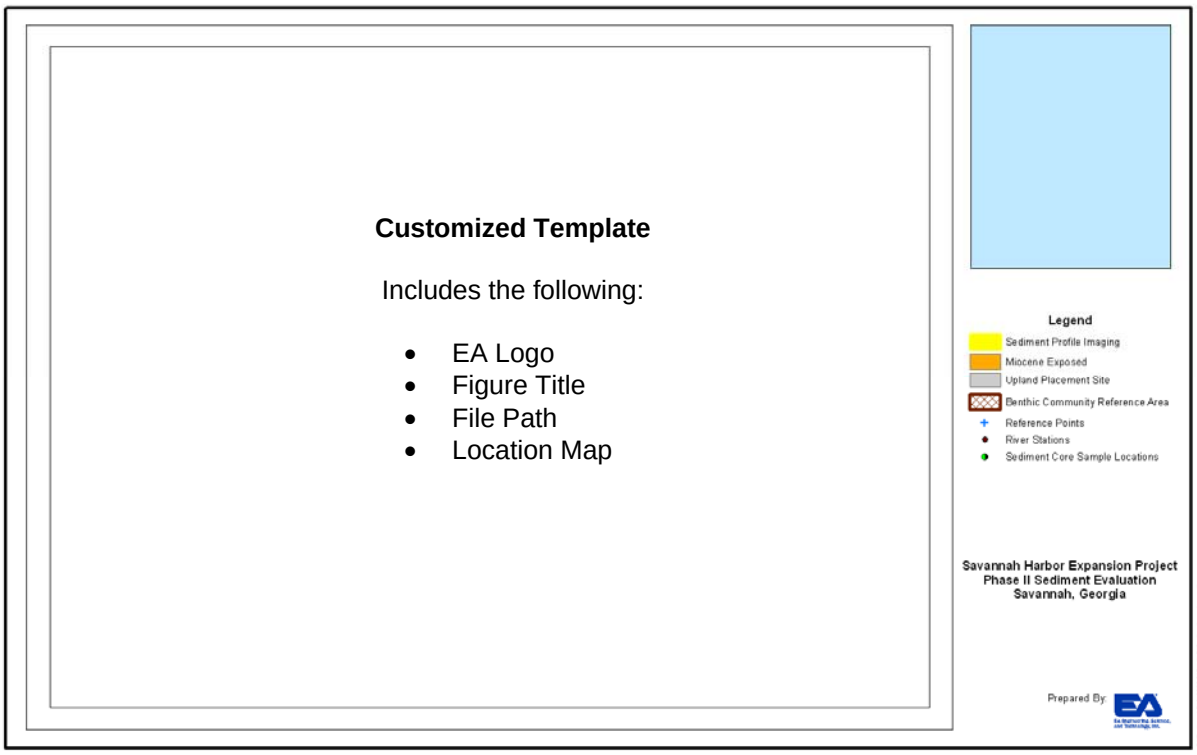


Figure Title


Customized Template
Includes the following:
EA Logo
Figure Title
File Path
Location Map
Legend
Sediment Profile Imaging
Miocene Exposed
Upland Placement Site
Benthic Community Reference Area
Reference Points
River Stations
Sediment Core Sample Locations
Savannah Harbor Expansion Project
Phase II Sediment Evaluation
Savannah, Georgia
Prepared By:

APPENDIX B-2
Spectrum Analytical
Standard Operating Procedures - Laboratory

Sulfuric Acid Cleanup

Method SW3665A

Contents SOP NO. 50.0031

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		3/29/10
Quality Assurance Director		3/29/10
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	Supervisor	2/7/12			
	Supervisor	3/18/13			

Revision Record

Revision Date	Revision Description	Comments	Initials
2/26/09	Lab name change		SBL
11/06/09	Corrected acid concentration	1:1 ratio required, not conc	SBL
	there are no restrictions for using concentrated sulfuric acid for the Method 3665A clean-up if you can	Per EHGS MICE 12/2009	
03/29/10	Revise SOP to match EPA SOM SOW	Per EPA	SBL
05/17/11	Reagents vendors removed		SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC

STANDARD OPERATING PROCEDURE

For

Sulfuric Acid Cleanup Method SW3665A

SOP No. 50.0031
Rev. 2

	Signature	Date
QA Director:	Mary S Lamb	3/29/10
Lab Director:		3/29/10
Effective Date:	4/5/10	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC

STANDARD OPERATING PROCEDURE

for

Sulfuric Acid Cleanup Method SW3665A

Rev. 2

1. Scope and Application

This method is suitable for the cleanup of samples prior to the analysis of Polychlorinated biphenyls (PCBs). This method should be used whenever elevated baselines or overly complex chromatograms prevent accurate quantitation of PCBs. This method is only allowed for PCB extracts since it will destroy most organic chemicals including pesticides.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Analysts and technicians are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. Supervisors and the Lab Manager are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. Supervisors or other senior management review the logbooks and data generated from this procedure and approve all reported results. The Laboratory Director or Project Manager evaluates all laboratory reports for reasonableness of the results and signs the reports. The QA Director reviews quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure

A hexane extract is treated with sulfuric acid prior to analysis for PCBs.

4. Sample Preservation, Containers, Handling, and Storage

Not Applicable

5. Interferences and Potential Problems

It is imperative that the extract be in hexane prior to this procedure.

6. Equipment and Apparatus

6.1. Pipettes, disposable

6.2. 10mL clear vials with Teflon lined screw caps

6.3. Clear autosampler vials with Teflon lined screw cap or crimp tops

7. Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used, provided the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1. Hexane: Pesticide Quality or better

7.2. H_2SO_4, concentrated, -ACS Trace Metals

7.3. DI H_2O (Organic-Free reagent water).

8. Procedure

8.1. Pipette 5.0mL of 1:1DI H_2O:H_2SO_4 into a clear 10mL vial. Mark the meniscus of the acid.

8.2. Use a disposable glass pipette to transfer an aliquot (approximately 1.5mL) of sample extract for acid cleanup.

8.3. Cap the vial and shake. Exercise care when handling H_2SO_4. Be sure not to shake too vigorously. The sample extract may be reacting with the acid and could potentially explode if the built up pressure is not relieved periodically.

8.4. Allow the acid and extract layers to separate for at least one minute. The top layer (hexane) should not be highly colored or have cloudiness or visible emulsion. If necessary use the centrifuge to assist with separation.

8.4.1. If the hexane layer is colored or an emulsion persists for several minutes, remove the acid layer and dispose of it properly. Add another 5.0mL of 1:1 H_2SO_4 into the vial and repeat **steps 8.3 through 8.4.**

8.5. Transfer the hexane top layer (extract) to a clean autosampler vial, taking care **not** to include any of the acid layer as acid can seriously affect GC/ECD performance. Mark the meniscus of the extract.

8.6. Document all cleanup procedures in the associated prep batch logbook and transfer the sample extracts to the analytical laboratory.

9. Data Reduction and Calculations

Not Applicable

10. Quality Assurance/Quality Control

Acid cleanup is mandatory for PCB/Aroclor sample extracts. All QC samples including blanks, lab control samples, matrix spikes and duplicate matrix spikes must be subjected to the same cleanup as the field samples.

11. Data Validation and Reporting

Document acid cleanup was performed, in the prep batch logbook.

12. Corrective Action Procedures

If interferences or elevated baselines are observed in the GC chromatogram, the GC analyst may request the aliquot undergo an additional acid cleanup.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, SW-846, Update III. Office of Solid Waste and Emergency Response, USEPA, Washington, D. C., Method 3665A, Sulfuric Acid/Permanganate Cleanup, Revision 1, December 1996.

USEPA. Statement of Work, Current Superfund Organic Methods (SOM) Exhibit D AROCLOR.

Sulfur Cleanup of Organic Extracts by modified Method SW3660B

Contents SOP NO. 50.0036

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		11/19/10
Quality Assurance Director		11/19/10
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
Timothy Mallard	Supervisor	2/7/12			
Jodie Warner	Supervisor	3/18/13			

Revision Record

Revision Date	Revision Description	Comments	Initials

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC

STANDARD OPERATING PROCEDURE

For

Sulfur Cleanup of Organic Extracts by modified Method SW3660B
SOP No. 50.0036
Rev. 0

	Signature	Date
QA Director:		11/19/10
Lab Director:		11/19/10
Effective Date:	11/26/10	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC

STANDARD OPERATING PROCEDURE

for
Sulfur Cleanup of Organic Extracts by modified Method SW3660B

1. Scope and Application

1.1. Elemental sulfur is encountered in many sediment samples, marine algae and some industrial wastes. The solubility of sulfur in various solvents is very similar to the organochlorine and organophosphate pesticides. Therefore the sulfur interference follows along with the pesticides through the normal extraction and cleanup steps.

1.2. Sulfur will be very evident in chromatograms obtained from a GC/ECD or GC/MS in SIM mode. If the GC is operated under normal conditions for pesticide analyses, the sulfur interference can completely mask the region from the solvent peak to Aldrin.

1.3. This SOP details the technique for sulfur elimination using copper powder. Copper may degrade organophosphorus and some organochlorine pesticides. This method is modified to use Sulfuric acid rather than Nitric. The activated copper is not dried under a stream of nitrogen, since exposure to air has negative consequences on the reactivity of the copper.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Analysts and technicians are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. Supervisors and the Lab Manager are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. Supervisors or other senior management review the logbooks and data generated from this procedure and approve all reported results. The Laboratory Director or Project Manager evaluates all laboratory reports for reasonableness of the results and signs the reports. The QA Director reviews quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure

The sample extract is mixed with activated copper. The mixture is shaken and allowed to settle. The extract is then sent to the Semivolatile Laboratory for analysis.

4. Sample Preservation, Containers, Handling, and Storage

Prior to Sulfur cleanup the 10mL hexane extract is stored in a 15mL vial. The meniscus is marked with a permanent marker.

5. Interferences and Potential Problems

The method requires the copper powder to be very reactive, as evidenced by a bright and shiny appearance. Care must be taken to remove all traces of acid used to prepare the copper in order to avoid potential degradation of specific analytes.

High levels of sulfur may result in crystallization in the extracts. If crystals are noted, centrifuge the extract or allow to crystals to settle, and remove the extract with a disposable pipette. Transfer the extract to a clean vial for further sulfur cleanup.

6. Equipment and Apparatus

6.1. Pipettes, disposable
6.2. VOA Vial, 60 mL with cap
6.3. Micro scoopula
6.4. Autosampler vials with Teflon/silicon lined crimp tops

7. Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test.

7.1. Methanol: pesticide quality or equivalent
7.2. Methylene Chloride: pesticide quality or equivalent
7.3. Acetone: pesticide quality or equivalent
7.4. Hexane: pesticide quality or equivalent
7.5. Copper, powder, fine granular Mallinckrodt 4649 or equivalent, requires activation
7.6. Copper Shots: granular copper (20-30 mesh)
7.7. H_2SO_4, concentrated, ACS Trace Metals
7.8. DI H_2O (Organic-Free reagent water).

8. Procedure

8.1. To activate copper:

8.1.1 Fill a 60 mL VOA vial half way with copper. Fill the vial with a 1:1 of DI H_2O: H_2SO_4. Screw cap on tight and shake the vial. If any heat builds up, uncap the vial. If it persists, decant the solution and add more DI H_2O.

8.1.2 Allow the copper to settle. Decant the aqueous layer without discarding any of the copper and minimizing exposure to air. Fill the vial with DI H_2O and shake. Allow the copper to settle. Exercise care to decant the DI H_2O without exposing the copper to air. Fill the vial with more DI H_2O. Shake and allow the copper to settle. Repeat the process until the water is neutral. Test with pH paper to verify.

8.1.3 Add methanol and shake. Decant and fill the vial with acetone. Shake and allow the copper to settle. Decant the Acetone and repeat. Ensure that the copper is free flowing. Any clumping indicates that water is still entrained in the copper mass and is not mixing with the more non-polar solvent. If the copper is going to be used in the extraction process then stop at this point and use as required. Ensure that the copper remains covered by acetone.

8.1.4 Continue the washing process with methylene chloride and then hexane if the copper is going to be used to treat extracts that are going on a GC/ECD. Always store the copper with a solvent layer on top to avoid exposure to air.

8.2 Extract Cleanup:

8.2.1 Use a glass disposable pipette to transfer approximately 1mL of the extract to a labeled autosampler vial. Add approximately a half of a micro scoopula full of activated copper powder or copper shots to the autosampler vial. Screw the cap on the vial and shake vigorously.

8.2.2 Allow the copper to settle.

8.2.3 If the copper turns black or the sulfur forms crystals, the procedure needs to be repeated. Use a glass, disposable pipette to transfer the extract to a clean autosampler vial without pipetting any of the spent copper and add new copper. Follow steps above.

8.2.4 Cap the vials with crimp caps. Crimp the caps and make sure the caps are on tight enough, i.e. the caps should not be able to twist on the vial. Mark the meniscus.

8.2.5 Document all cleanup procedures in the associated extraction logbook as well as the lot number of the copper. Document in logbook if multiple copper cleanups were needed. Transfer the sample extracts to the Semivolatile laboratory unless further cleanup is needed.

9. Data Reduction and Calculations

Not applicable

10. Quality Assurance/Quality Control

10.1. Sulfur cleanup is mandatory for all Pesticide/PCB sample extracts. All QC samples including blanks, lab control samples, matrix spikes and duplicate matrix spikes must be subjected to the same cleanup as the field samples.

10.2. If the copper begin to show any darkening or blackness it is becoming inactive and needs to go through the entire process again.

10.3. To aid in the evaluation of the copper activity a 60mL vial containing hexane that has had sulfur added to it can be used. Add copper to the solution be application to the samples. If it does not immediately turn black it is becoming inactive and may not be effective. Reactivate the copper.

11. Data Validation and Reporting

Not applicable

12. Corrective Action Procedures

Care must be taken to remove all traces of acid used to prepare the copper in order to avoid potential degradation of specific analytes. It has been noted that if acid has not been fully removed, use of the copper powder will degrade heptachlor quite readily. This will be noted in the LCS and/or MS/MSD samples. When this occurs, the set of extracts will need to be re-sulfur cleaned. The batch of copper powder must be reactivated.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, SW-846, Update III. Office of Solid Waste and Emergency Response, USEPA, Washington, D. C., Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid (Method 3520C)

Contents SOP NO. 50.0050

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		5/10/11
Quality Assurance Director	Shanna Stanley	4/28/11
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
Tim McDaniel	Dept Supervisor	4/6/12			

Revision Record

Revision Date	Revision Description	Comments	Initials
12/6/06	Added in TPH/DRO extraction		
01/25/07	Added sentence regarding pH to section 8.2.2.	TPH/DRO samples should be pH ≤ 2 prior to extraction for better recoveries.	SBL
4/27/07	Removed filtration and conc steps	Inserted reference to SOP 50.0054	SBL
01/03/08	Corrected surrogate and LCS volumes spiked, Changed Laboratory name	New conc of surrogate mixes used now.	SBL
6/27/08	Removed pH change to >11 second extraction, was **section 8.2.10.** Reorganized sections 8.2	LCS and surrogates had shown excellent recoveries using the single pH change as per SOM1.2 methodology for SV AQ ext.	SBL
4/20/09	TurboVap instead of RapidVaps		SBL
5/7/09	Add Mirex		SBL
02/02/10	Edited reagents grade/vendor	Full rev.	SBL
03/23/10	Procedural change BACK to two pH extractions per method	Full rev. Unable to make modification noted in 6/2008 since never did study. Had to remove most of section 5.4 also	SBL
10/29/10	Sodium sulfate added to round bottom during extraction	Minor: To remove water. Also removed re-ext log since no longer using	SBL
4/26/11	Revised TPH surrogate solutions and deleted MA EPH info; should use Sepf	Full rev. Only spiking OTP	SBL/TM
8/18/11	Include 1,4-dioxane option	Minor SIM	SBL
12/27/11	Standard storage temp	Minor; edit to refrigeration not freezer	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid (Method 3520C)

SOP No. 50.0050

Rev. 6

	Signature	Date
QA Director:	Sharon B Hawke	4/28/11
Lab Director:		5/10/11
Effective Date:	5/17/11	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid (Method 3520C)

Rev 6

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the operations for the preparation of aqueous samples for analysis by EPA SW846 8270, 8015, TPH/DRO, 8081, 8082 and EPA Methods 608 and 625 using continuous liquid-liquid extraction. USEPA SOM01.2 semivolatile liquid-liquid extraction is performed only at the acid pH, for a minimum of 18 hours. Discussions include sample extraction, sample cleanup references, and the preparation of standard spiking solutions.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 1-liter sample is solvent extracted for a minimum of 18 hours using a continuous liquid-liquid extractor. The extract is dried through granular anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using a Caliper TurboVap apparatus or Kuderna-Danish (KD) apparatus and nitrogen blow down.

4. Sample Preservation, Containers, Handling, and Storage

4.1 The sample holding times are as follows:

- Sample must be extracted within 7 days from the time of sample collection
- Sample extracts must be analyzed within 40 days of sample extract.

4.2. DRO samples are preserved in the field with H_2SO_4 to a pH < 2.

5. Interferences and Potential Problems

5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results for the analytes of interest.

5.3 Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation steps to minimize interferences from these compounds.

5.4 The decomposition of some semivolatile analytes has been demonstrated under basic extraction conditions required to separate analytes. In particular, phthalate esters may exchange, and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in method 3510 (separatory funnel extraction, SOP Number 50.0051). Method 3510 is preferred over Method 3520 for the analysis of these classes of compounds. The recovery of phenols may be optimized by using Method 3520 and performing the initial extraction at the acid pH.

6. Equipment and Apparatus

Equipment used in this preparation method includes:

6.1 Continuous liquid-liquid extractor.

6.2 Boiling flask – 500mL.

6.3 1L graduated cylinder.

6.4 Teflon boiling chips.

6.5 Wide range pH strips, Whatman or equivalent.

6.6 Narrow range pH strips, Whatman or equivalent.

6.7 Condensers, 45/50.

6.8 Heating mantles.

6.9 Glass syringes for delivering lab control spike, matrix and surrogate spike solutions.

6.10 Aluminum Foil (Industrial Grade)

6.12 Glass Pipettes

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. The following chemicals including solvent, standards and gasses are extensively used in the lab:

7.1 Methylene chloride: pesticide quality or equivalent, to be used for glassware rinsing and sample extraction.

7.2 Methanol: pesticide quality or equivalent, to be used for rinsing glassware and preparing standards.

7.3 Hexane: pesticide quality or equivalent, to be used for solvent exchange of samples.

7.4 Acetone: pesticide quality or equivalent, to be used for glassware rinsing.

7.5 Surrogate Standards: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials*.

- Semivolatile Full Scan: Restek BN Surrogate mix (Cat. No. 31086) at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
- Semivolatile SIM: Cambridge Isotopes Benzo (e) pyrene -d12 (Cat. No. DLM-257-S) at 200µg/mL.
- Diesel Range Organics (DRO)/TPH*:* o-Terphenyl at 10,000µg/mL (Made from neat source (Aldrich)).
- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB) (Cat. No. PPS-150) at 1000 µg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000µg/mL in Acetone.
- Semivolatile SIM Optional 1,4-Dioxane analysis: Cambridge Isotopes 1,4-Dioxane-d8 (Cat.No.DLM-28-5) in neat form.

7.6 Lab Control Sample and Matrix Spike: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials*.

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000µg/mL in Methylene Chloride; Restek 3, 3’ Dichlorobenzidine (Cat. No. 31026) at 2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1, 1’ Biphenyl, Caprolactam, Acetophenone, and Atrazine. Ultra 1, 4-Dioxane (Cat. No. RCC-180) in neat form.
- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL and Mirex (Ultra Scientific Cat. No.PST-720M100A01) at 100ug/mL. For the LCS/MS mix a 10.0ug/mL Intermediate standard is prepared for Mirex when required.
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
- TPH/DRO: LCS uses Diesel/gasoline.
- Semivolatile SIM Optional 1,4-Dioxane analysis: 1,4-Dioxane at 2000ug/mL (Restek Cat.No.31853)

7.7 H_2SO_4, concentrated, for sample pH adjustment.

7.8 10N NaOH for sample pH adjustment.

7.9 Anhydrous sodium sulfate: granular for drying the samples. Baked at 400°C for four hours.

8. Procedure

8.1 Standards are prepared in the semivolatile laboratory (GC or GC/MS) for the Organic Sample Preparation (OPREP) Lab. Standards are QC checked by the semivolatile laboratory (GC or GC/MS) prior to release to the OPREP Lab:

8.1.1 All primary standards received from vendors are logged into the LIMS Standard Logbook. These include standards for surrogates, LCS and matrix spike. The standards are labeled LPyymmddX, where:

L = S for Semivolatile, or P for Pesticides/PCB
P = Primary standard
yymmdd = date standard is received, and
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2 Preparation of Surrogate standard(**SS**):

Semivolatile Full Scan: The working surrogate standard is prepared by combining 2.5mL of the Base/Neutral primary standard in **section 7.5** and 1.075mL of the Acid primary standard in **section 7.5** and diluting to 250mL using methanol. The working standard contains the following compounds:

Compound	Concentration(µg/mL)
1, 2-Dichlorobenzene-d_4	50
2-Fluorobiphenyl	50
Nitrobenzene-d_5	50
p-terphenyl-d14	50
2-Chlorophenol-d_4	75
2-Fluorophenol	75
Phenol-d_6	75
2, 4, 6-Tribromophenol	75

The working surrogate spike standard is labeled LWyymmddX where:

L = S for Semivolatile, or P for Pesticides/PCB
W = working standard
yymmdd = date working standard is prepared,
X = the order that the working standard is prepared on that date, in increasing alphabetical order.

Semivolatile SIM surrogate standard is prepared by diluting 1.25mL of the 200ug/mL primary standard in **section 7.5** to 100mL in methanol. The resulting concentration is 2.5ug/mL of Benzo (e) pyrene -d12. Projects requiring 1,4-Dioxane will use a surrogate standard prepared by diluting 2.5mL of the 100ug/mL 1,4-Dioxane-d8 intermediate standard made from the primary in **section 7.5** to 100mL in methanol. The resulting concentration is 2.5ug/mL of1,4-Dioxane-d8.

The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **section 7.5**, and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

Compound	Concentration (µg/mL)
Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

The working TPH/DRO surrogate standard is prepared by taking 1mL of the o-Terphenyl primary standards in **section 7.5** and diluting to 100mL using methanol. The working standard contains o-Terphenyl at 100ug/mL.

A smaller or larger volume of the stock solution may be used to prepare the surrogate spike. The final volume of the surrogate solution will be adjusted accordingly to achieve the same spike working concentration.

The standard is placed in an amber bottle and stored in the refrigerator at 4± 2°C. It is stored in a separate location to make sure that there is no sample cross-contamination.

The expiration date for the surrogate standard is no longer than six months from the date of preparation.

8.1.3 Preparation of Lab Control Spike(**LCS**)and Matrix Spike Standard(**MS**):

The working Semivolatile Full Scan LCS/MS Standard is prepared by combining 5.0 mL of 8270 MegaMix, 2.5mL of 3,3' DCB, and 5.0 mL of 8270 add-on intermediate standard from **section 7.6**, and diluting to 50mL using methanol. The working LCS/MS Standard contains the full list of SW8270 compounds at 50 µg/mL.

The working Semivolatile SIM LCS/MS Standard is prepared by a further 20 times dilution of the above standard. When 1,4-Dioxane is required, an additional LCS/MS will be needed at a working concentration of 2.5µg/mL.

The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2 (and 1.0mL of Intermediate Mirex standard from **section 7.**6 when required), and diluting it to 50 mL in methanol. The working standard contains the full list of SW8081 individual response compounds. See Attachment 1 for a list of the individual compounds and their concentrations.

The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **section 7.6** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

Compounds	Concentration(µg/mL)
Aroclor 1016	4.0
Aroclor 1260	4.0

The working TPH/DRO LCS/MS Standard is prepared by first preparing an intermediate standard and making a dilution of it.

On an analytical balance, weigh out 5.00g of diesel gasoline into a 10mL volumetric flask and bring to volume with Methanol. This is the intermediate standard. The working LCS/MS Standard is prepared by taking 1mL of the intermediate standard and diluting to 100mL using Methanol. The working Standard contains Diesel Gasoline at 5000 µg/mL.

The Working LCS/MS Standards are labeled using the same approach discussed in **Section 8.1.2.**

A smaller or larger volume of the Lab Control Spike stock solutions may be used to prepare the LCS/MS Standard. The final volume will be adjusted accordingly to achieve the same working concentration.

The standard is placed in amber bottles and stored in the refrigerator at 4± 2°C. They are stored in a separate location to make sure that there is no sample and/or sample extract cross contamination.

The expiration date for the LCS/MS Standard is no longer than six months from the date of preparation.

All of the appropriate standard preparation information is to be recorded in the LIMS Standard Logbook.

NOTE: All standards made from a primary standard expire on or before the primary standard's expiration date.

8.2 Sample Extraction Procedure:

The liquid-liquid extractors are inspected periodically to ensure that there are no cracks in the glassware. After inspection of the glassware, it is rinsed twice using either methanol or acetone to remove any water and then rinsed twice using methylene chloride to remove any contaminants. This is to be done for both the liquid-liquid extractors and the boiling flasks.

8.2.1 The aqueous sample to be extracted is allowed to warm to ambient temperature prior to extraction. A representative 1L portion of the sample is measured by first marking the sample level on the bottle (which is not a Mitkem bottle). The contents of the container are poured into the liquid-liquid extractor. The marked bottle is then used to measure the sample. If the sample bottle is a Mitkem bottle, a bottle for each lot number will be calibrated and marked. This bottle will be used as a reference when determining initial sample volume.

8.2.2 The graduated cylinder or sample bottle is then rinsed with 50mL of methylene chloride, which is also added to the liquid-liquid extractor. The bottle is saved for volume determination if the sample volume was less than 1L.

8.2.3 If the sample volume is less than 1L, take the sample bottle with the meniscus marked and fill to the meniscus with tap water. Empty the sample bottle into a graduated cylinder. Record the volume in the Prep Lab logbook. Adjust all surrogates or spikes proportionally, if final volume is to be modified as well.

8.2.4 Use 1L of organic free water for the Blank and LCS.

8.2.5 The sample pH is taken.

- The Pesticide or PCB sample pH should be within 5 – 9. If pH is not correct, add 1:1 H_2SO_4 or 10N NaOH until the pH of the sample is within 5 – 9. Wide range pH paper may be used. Document any pH adjustment in the associated extraction logbook.

- The Semivolatile sample pH is taken and pH adjusted to **less than 2** with 1:1 H_2SO_4. Take the pH again with narrow range pH paper to verify the adjusted pH.

- The TPH/DRO sample pH should be **less than 2** since it was acidified in the field. If the sample is not acidic, note in logbook and contact the project manager as well. Acidify any samples which were not preserved in the field.

8.2.6 Add about 3 Teflon boiling chips to the boiling flask. Attach the continuous liquid-liquid extractor to the boiling flask and place the boiling flask in the heating mantle. Secure the continuous liquid-liquid extractor with the chain clamp such that it is completely vertical. Add 350mL of methylene chloride to the continuous liquid-liquid. Add 400mL of methylene chloride to the liquid-liquid extractor for the Blank and LCS.

8.2.7 Remove the surrogate standard and the lab control sample standard prepared in **Sections 8.1.2** and **8.1.3** from the refrigerator and allow them to reach room temperature.

- Use a syringe to add 1.0 mL surrogate standard into the Blank, LCS, sample and any MS/MSD sample that is in the continuous liquid-liquid extractor.

- Use another syringe to add 1.0 mL LCS standard into the LCS and any MS/MSD samples.

8.2.8 Place the condensers on the liquid-liquid extractors. Start the water flowing through the condensers. Allow the condensers to cool. Turn on the heating mantles. Measure the drip rate and document it in the extraction logbook.

8.2.9 Allow the samples to be extracted for a **minimum** of 18 hours.

8.2.9.1 For **Semivolatile samples only**, adjust the pH of the aqueous phase to >11 with 10N NaOH. Test the pH with narrow range pH paper to verify the adjusted pH. Reattach the boiling flask and chain clamps. Extract the sample again for a minimum of 18 hours.

8.2.10 Turn off the heating mantle and allow the liquid-liquid extractors to cool to room temperature. Hang up the condensers, while being sure to hold the boiling flask.

8.2.11 Un-clamp the liquid-liquid extractors from the chain clamp and siphon as much methylene chloride as possible into the boiling flask without getting water into the extract.

8.2.12 Detach the boiling flask. Add approximately 20-30 grams of sodium sulfate to the flask to absorb excess water in the extract. Cover the boiling flask with aluminum foil.

8.2.13 Dispose of the aqueous waste from the liquid-liquid extractors into the proper satellite receptacle.

8.2.14 Record all extraction information, including the Date and Time the liquid-liquid extraction began and ended, in the appropriate Prep Batch Logbook (**Figure 1 and 2**).

8.3 The sample extract from **section 8.2.12** is now ready for filtration and concentration. These procedures are found in SOP No. 50.0054, Extract Filtration and Concentration.

8.4 After concentration the extract may require cleanup. The following is a list of Cleanup Procedures**:**

8.4.1 **Sulfur cleanup** is mandatory for all Pesticide/PCB sample extracts containing sulfur. All QC samples including blanks, lab control samples, matrix spikes and duplicate matrix spikes must be subjected to the same cleanup as the field samples. Sulfur cleanup will be performed using activated copper powder. Refer to **SOP No. 50.0036** Method 3660B Sulfur Cleanup, for details on activating copper and using it in a sample extract.

8.4.2 **Acid cleanup (PCB extract only)** is mandatory for all PCB sample extracts. Refer to **SOP No. 50.0031** Method 3665A Sulfuric Acid Cleanup, for details on using the acid cleanup in a PCB sample extract.

8.4.3 **GPC cleanup** is useful for both Pesticide and semivolatile samples. Refer to SOP **No. 50.0032** Method 3640A GPC Cleanup, for details on the procedure for cleanup and quality control criteria for GPC.

8.4.4 Other cleanup methods may be found in SOP numbers **50.0033**(Florisil) and **50.0034** (Silica Gel).

8.5 The extracts are transferred to the Semivolatile lab. The extracts are stored in the refrigerator at 4°C until analysis. The storage location is recorded electronically by the analyst during LIMS batch transfer.

8.6 Sample and Extract Disposal:

Refer to SOPs 30.0003 and 30.0024 for details on sample and extract tracking and disposal.

9. Data Reduction and Calculations

9.1 Data reduction for calculation of standard preparation

$$\text{Concentration of working standard} = \frac{(\text{Concentration of ampule})(\text{amount used})}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. In order to trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and contains the manufacturer's reference number given in the standard preparation logbook or the LIMS logbook.

10.1 Method Blank – A method blank is a liter of organic free reagent water that is carried through the entire analytical procedure. It is used to determine the level of contamination associated with the analytical processing and analysis of samples.

10.1.1 Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2 Procedure for Method Blank

- The Method Blank is prepared in identical fashion as the associated samples.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.

- The Method Blank is labeled **MB** and is given a numerical value, which increases with every batch of twenty samples or less.

10.2 Surrogate – Surrogate standards are added to all samples including the Method Blank, Lab Control Sample and matrix spikes to assess the efficiency of the sample preparation and analysis procedures.

10.3 Lab Control Sample (LCS) – A Lab Control Sample is a liter of organic free reagent water that is spiked with all target analytes and the surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1 Frequency of LCS:

An LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.3.2 Procedure for LCS:

An LCS is prepared in identical fashion as the associated samples; in addition:

- An aliquot of surrogate solution prepared in **Section 8.1.2** and lab control spike prepared in **Section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the corresponding numerical value as the associated method blank.

10.4 Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix. For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received do not have any documented history, the lab will perform matrix spike and matrix spike duplicate.

10.4.1 Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Once every 20 samples, or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.4.2 Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples, in addition:

- An aliquot of surrogate solution prepared in **Section 8.1.2** and lab control spike prepared in **Section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.

11. **Data Validation and Reporting**

11.1 Data generated in the organic preparation laboratory will be reviewed by the supervisor. The QA Department will perform periodic and unscheduled reviews. These data consist of the standards preparation, volume of samples, and the volume and lot number of solvents used.

11.2 Reporting of the data will include review by the Organic Preparation Laboratory Supervisor of the data listed in **Section 11.1**, time of extraction, sampling handling procedures, and extract handling procedures.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1 Method Blank Analysis:

All samples associated with a non-compliant Method Blank are re-extracted and reanalyzed. The analysis laboratory will inform the Organic Preparatory Laboratory when method blanks have not met accepted criteria, and require re-extraction. The re-extracted samples will be labeled with the suffix RE.

12.2 Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects.

12.3 LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. Any sample(s) that is/are associated with a non-compliant LCS will require re-extraction and re-analysis. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS

recoveries have not met accepted criteria, and require re-extraction. The re-extracted samples will be labeled with the suffix RE.

12.4 Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Abbreviations

See sections 19.0 and 20.0 of the current Quality Assurance Plan.

15. References

Quality Assurance Plan, Mitkem Laboratories, A Division of Spectrum Analytical, Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.

Attachments:
Attachment 1: Pesticide MS/LCS list
Figure 1: Semivolatile Extraction Logbook
Figure 2: Pesticides/PCB Extraction Logbook

Attachment 1: Pesticide MS/LCS list

Analyte	Spike conc in ug/mL
4,4´-DDD	0.4
4,4´-DDE	0.4
4,4´-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2
Mirex	0.2

Figure 1: Semivolatile Extraction Logbook

MITKEM LABORATORIES **ORGANIC PREP - SAMPLE PREPARATION: SEMIVOLATILES**

Date:	Analysis:		Method #	Aq: 3510C (SepF) 3520C (Liq/Liq) Other: Soil: 3550B (Sonic) 3540C (Soxhlet) _____		Matrix Aqueous Soil Wipe Oil Other:_____		Sodium Sulfate Lot #:
Batch ID	LCS ID	Analyst	Spiked By	Witness	MeCl Lot#	Acetone Lot#	H_2SO_4 Lot#	NaOH Lot#	Time/Date Started: Time/Date Ended:

Lab ID	Bottle #	Sample Wt (g) / Vol (mL)	Initial pH	Surrogate Spike Added(uL)	Matrix Spike Added(uL)	H2SO4 pH<2	NaOH pH>11	Emulsion	KD Prior to GPC or Fractionation Date / Analyst	GPC or Fractionation Date / Analyst	Final Concentration Date / Analyst	Final Conc. Volume (mL)	Date Extract Trans.

Comments: _____

_____

_____

_____

Surrogate Std. ID: _____

Matrix Spike/LCS Std. ID: _____

Sonicator Tuned: Yes / No

Soxhlet Cycle/Hour:_____

Drip Rate_____

Water Bath Temp:_____/MT-___

Figure 2: Pesticides/PCB Extraction Logbook

MITKEM LABORATORIES ORGANIC PREP-SAMPLE PREPARATION:PEST/PCB

Date:	Prep Method	Aq: 3510C (SepF) 3520C (Liq/Liq) Soil: 3550B (Sonc) 3540C (Soxhlet) 3570(MSE) Other:_____	Matrix: Aqueous Soil Wipe Oil Other:_____	BATCH ID:	Date/Time Started:
Analyst	Spiked By	Witness	Solvent: Lot #	GPC Batch Number	Florisil Lot #	Analysis:	Date/Time Ended:

Lab ID	Bottle #	Sample Wt (g) / Vol (ml)	Initial pH	Emulsion?	Initial CONC Date/ Analyst TV	Extract volume (ml)	GPC Cleanup Date / Analyst	FLORISIL Cleanup Date / Analyst	ACID (PCB only) Cleanup Date / Analyst	SULFUR (Copper) Cleanup Date / Analyst	Final CONC Date/ Analyst TV	Final extract volume (ml)	Extract Transfer Date /By

Comments:

PEST Matrix Spike Std ID:	Vol. Spiked:		
PCB Matrix Spike Std ID:	Vol. Spiked:		
PEST Surrogate Spike Std ID:	Vol. Spiked:	Sodium Sulfate lot#	
PCB Surrogate Spike Std ID:	Vol. Spiked:	Hexane lot #	L/L Drip Rate:
PEST LCS Spike Std ID:	Vol. Spiked:	Sulfuric Acid lot#	Soxhlet Cycle/Hour:
PCB LCS Spike Std ID:	Vol. Spiked:	Sulfur cleanup Copper lot #	Sonicator Tuned? Yes/No

Logbook ID: 50.0149-03/10

Reviewed By:_____

Organic Preparation of Aqueous Samples by Separatory Funnel (Method 3510C)

Contents SOP NO. 50.0051

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		2/4/10
Quality Assurance Director		2/5/10
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	CAT	4/25/11			
Tim McDaniel	Lab. Supervisor	4/6/11			

Revision Record

Revision Date	Revision Description	Comments	Initials
11/3/06	Added Method 625 to list of tests		SBL
01/25/07	Added sentence to section 8.2.2	All TPH/DRO samples should be extracted at pH ≤ 2	SBL
4/24/07	Edited reference in section 8.3	SOP 50.0054	SBL
01/03/08	Edited surrogate and LCS volumes, and changed Lab name	Spike conc changed	SBL
4/20/09	TurboVap instead of RapidVap		SBL
5/7/09	Added Mirex		SBL
02/02/10	Standard and reagent purity comments		SBL
08/10/10	3D gyratory shaker		SBL
4/25/11	Edit to DRO/TPH Surrogate	OTP only	TM
12/27/11	Standard storage temp	Minor; edit to refrigeration not freezer	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Aqueous Samples by Separatory Funnel (Method 3510C)

SOP No. 50.0051

Rev. 2

	Signature	**Date**
QA Director:	Thomas B Lewis	2/2/10
Lab Director:	[signature]	2/4/10

Effective Date: 02/11/10

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Aqueous Samples by Separatory Funnel (Method 3510C)

Rev. 2

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the operations for the preparation of aqueous samples using separatory funnel extraction. Discussions include sample extraction, sample cleanup references, and sample concentration technique of aqueous samples for analysis by SW-846 Methods 8081, 8082, 8270, 8015(DRO/TPH), MA EPH and EPA Methods 608 and 625.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 1-liter sample is solvent extracted using a separatory funnel. The extract is dried through granular anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using a Caliper TurboVap apparatus or Kuderna-Danish (KD) apparatus, and nitrogen blow down.

4. Sample Preservation, Containers, Handling, and Storage

4.1. The sample holding times are as follows:

- Sample must be extracted within 7 days from the time of sample collection

- Sample extracts must be analyzed within 40 days of sample extraction.

4.1 Samples should be in 1-L amber glass jars.

4.2 Samples are stored at 4°C until time of extraction.

4.3 DRO samples are preserved in the field with H_2SO_4 to a pH < 2.

4.4 Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results for the analytes of interest. See Mitkem SOP Nos. 50.0030 through 50.0034 for cleanup methods.

5.3 Phthalate esters can contaminate sample extracts, and many products found in the laboratory contain these esters. Plastics in particular must be avoided during the preparation steps to minimize interferences and contamination from these compounds. Several phthalate esters are target compounds in the semivolatile analyses. Contamination of method blanks and samples with these phthalates poses additional quality issues with the resulting data.

6. Equipment and Apparatus

Instrumentation used in this preparation method include:

6.1 Separatory funnel – 2L, glass or Teflon.

6.2 Erlenmeyer flask – 500mL.

6.3 1L graduated cylinder.

6.4 Wide range pH strips, Whatman or equivalent.

6.5 Glass syringes for delivering lab control spike, matrix and surrogate spike solutions.

6.6 Volumetric flasks for making up surrogate and matrix spike solutions – 50mL, 250mL.

6.7 Kuderna-Danish apparatus with a 10mL or 15mL receiver tube.

6.8 Glass funnel.

6.9 Three ball Synder column.

6.10 Water bath, capable of maintaining 70 ^{o}C or 95^{o}C.

6.11 Boiling chips, carbon.

6.12 Nitrogen blowdown apparatus, N-EVAP Model No. 111.

6.13 Receiving vial with Teflon septa – 2mL.

6.14 Glass Pipettes.

6.15 20X150mm Disposable Culture Tubes.

6.16 Turbo Vap Collection Tubes.

6.17 Caliper TurboVap

6.18 Aluminum Foil (Industrial Grade).

6.19 3D Gyratory Shaker: three-directional shaker

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. The following chemicals including solvent, standards and gasses are extensively used in the lab:

7.1 Methylene chloride: pesticide quality or equivalent, to be used for glassware rinsing and sample extraction.

7.2 Methanol: pesticide quality or equivalent, to be used for preparing standards.

7.3 Acetone: pesticide quality or equivalent, to be used for rinsing glassware.

7.4 1:1 v/v H_2SO_4 to be used for pH adjustment.

7.5 10N NaOH to be used for pH adjustment.

7.6 Anhydrous sodium sulfate, granular for drying the sample extract.

7.7 Surrogate standards: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatiles Full Scan: Restek BN Surrogate mix (Cat. No. 31086)at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
- Semivolatiles SIM: Cambridge Benzo(e) pyrene d-12 surrogate (Cat. No. DLM-257-S) at 200µg/mL.
- Diesel Range Organics (DRO)/TPH: o-Terphenyl at 10,000µg/mL (Made from neat source (Aldrich)).
- MA EPH: 5-α Androstane at 10,000µg/mL (Made from neat source (Sigma)), and o-Terphenyl at 10,000µg/mL (Made from neat source (Aldrich)).
- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 µg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000µg/mL in Acetone.

7.8 Lab Control Sample and Matrix Spike: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000µg/mL in Methylene Chloride; Restek 3, 3' Dichlorobenzidine (Cat. No. 31026) at 2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine.
- DRO/TPH: Diesel/gasoline.
- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL and Mirex (Ultra Scientific Cat. No.PST-720M100A01) at 100ug/mL, if required. For the LCS/MS mix a 10.0ug/mL Intermediate standard is prepared for Mirex.
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
- MA EPH: An independent vendor than that used for the MA EPH calibration standards is used. The LCS consists of the 17 Aromatic Hydrocarbon compounds plus the 14 normal Aliphatic Hydrocarbons at 1000µg/mL in Methylene Chloride.

8. Procedure

8.1 Standards Preparation:

All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

8.1.1 All primary standards received from vendors are logged into the LIMS Standard Logbook. These include standards for surrogates, LCS and matrix spike. The standards are labeled ZPyymmddX, where:

Z= S for Semivolatile and DRO, P for Pest/PCB
P = Primary standard
yymmdd = date standard is received, and
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2 Preparation of Surrogate standard:

Semivolatile Full Scan: The working surrogate standard is prepared by combining 2.5mL of the Base/Neutral primary standard and 1.075mL of the Acid primary standard in **Section 7.7** and diluting to 250mL using methanol. The working standard contains the following compounds:

Compound	Concentration(µg/mL)
1,2-Dichlorobenzene-d_4	50
2-Fluorobiphenyl	50
Nitrobenzene-d_5	50
p-terphenyl-d_{14}	50
2-Chlorophenol-d_4	75
2-Fluorophenol	75
Phenol-d_6	75
2,4,6-Tribromophenol	75

Semivolatile SIM: The working surrogate standard is prepared by diluting 1.25mL of the 200ug/mL primary standard in Section **7.7** to 50mL in methanol. The resulting concentration is 5ug/mL of Benzo (e) pyrene d-12.

DRO/TPH: The working surrogate standard is prepared by taking 1.0 mL of the o-Terphenyl primary standards in **Section 7.7** and diluting to 200mL using methanol. The working standard contains o-Terphenyl at 50 µg/mL.

MA EPH: The working surrogate standard is prepared by taking 1mL of the 5-α Androstane and 1.0 mL of the o-Terphenyl primary standards in **Section 7.7** and diluting to 200mL using methanol. The working standard contains the following compounds:

Compound	Concentration(µg/mL)
o-Terphenyl	50
5-α Androstane	50

Pesticide/PCB: The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.7**, and

diluting them to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

Compound	Concentration(µg/mL)
Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

The working surrogate spike standard is labeled ZWyymmddX where:

Z= S for Semivolatile and DRO/TPH, P for Pest/PCB
W = working standard
yymmdd = date working standard is prepared,
X = the order that the working standard is prepared on that date, in increasing alphabetical order.

A smaller or larger volume of the stock solution may be used to prepare the surrogate spike. The final volume of the surrogate solution will be adjusted accordingly to achieve the same spike working concentration.

The standard is placed in amber screw top bottles and stored in the refrigerator at 4± 2°C. They are stored in a separate location from sample extracts to make sure that there is no sample and/or sample extract cross contamination.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3 Preparation of Lab Control Spike (LCS) and Matrix Spike (MS) Standard:

Semivolatiles Full Scan: The working Semivolatile LCS/MS Standard is prepared by combining 5.0 mL of 8270 MegaMix, 2.5mL of 3,3' DCB, and 5.0 mL of 8270 add-on intermediate standard in **Section 7.8** and diluting to 50mL using methanol. The working LCS/MS Standard contains the full list of SW8270 compounds at 50 µg/mL.

Semivolatile SIM: The working LCS/MS Standard is prepared by a further 10 times dilution of the above standard.

DRO/TPH: The working LCS/MS Standard is prepared by first preparing an intermediate standard and making a dilution of it.

On an analytical balance, weigh out 5.00g of diesel gasoline into a 10mL volumetric flask and bring to volume with Methanol. This is the intermediate standard. The working LCS/MS Standard is prepared by taking 1mL of the intermediate standard and diluting to 100mL using Methanol. The working LCS/MS Standard contains the following compounds:

Compounds	Concentration (µg/mL)

Diesel Gasoline	5000

Pesticides: The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2 and 1.0mL of Intermediate Mirex standard in **Section 7.8,** if required, and diluting it to 50 mL in methanol. The working LCS/MS Standard contains the full list of SW8081 individual response compounds. See Attachment 1 for a list of the individual compounds and their concentrations.

PCB: The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.8** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

Compounds	Concentration (µg/mL)
Aroclor 1016	4.0
Aroclor 1260	4.0

MA EPH :The working MA EPH LCS/MS Standard is prepared by taking 2.5mL of the Aliphatic Hydrocarbons standard in Section **7.8** and 2.5mL of the Aromatic Hydrocarbon standard in Section **7.8** and diluting to 250mL in 1:1 methylene chloride: acetone mixture . The final concentration will be 50 ug/ml for each of the individual components.

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2**.

A smaller or larger volume of the stock solution may be used to prepare the LCS/MS Standard. The final volume will be adjusted accordingly to achieve the same working concentration.

The standard is placed in amber screw top bottles and stored in the refrigerator at 4± 2°C. They are stored in a separate location from sample extracts to make sure that there is no sample and/or sample extract cross contamination.

The expiration date for the ampulated solutions is discussed in **Section 8.1.1.**

All of the appropriate standard preparation information is to be recorded in the LIMS Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2 Sample Extraction Procedure:

Aqueous samples are solvent extracted using a 2-L separatory funnel. The glass separatory funnels must be inspected periodically to ensure that there are no cracks. The

Teflon stoppers must also be inspected to make sure there are no scratches. The Teflon separatory funnels must be inspected periodically to ensure there are no scratches.

8.2.1 The aqueous sample to be extracted is allowed to warm to ambient temperature prior to extraction. A representative 1L portion of the sample is measured by first marking the sample level on the bottle (which is not a Mitkem bottle). The contents of the container are poured into the separatory funnel. The marked bottle is then used to measure the sample. If the sample bottle is a Mitkem bottle, a bottle for each lot number will be calibrated and marked. This bottle will be used as a reference when determining initial sample volume. An alternate option is to use a clean 1 L Class A graduated cylinder to measure the sample.

8.2.2 The sample pH is taken and pH adjusted to 5 – 9 if necessary. Add 1:1 H_2SO_4 or 10N NaOH as needed. The 1L aliquot is then transferred to the separatory funnel. The pH may be taken using a clean glass stir rod or a glass pipette. Another option is to open the stopcock on the sample for a moment and allow a drop or two to drop onto the pH paper directly from the separatory funnel. This must be done BEFORE the addition of solvent. For semivolatile extractions, the sample is acidified to a ph<2. DRO samples should have been acidified in the field. Acidify any DRO/TPH samples which were not field preserved. Document the pH adjustment of any Pesticide/PCB and DRO/TPH samples in the associated Prep Batch logbook.

8.2.3 The graduated cylinder or sample bottle is rinsed with 60mL of methylene chloride, which is also added to the separatory funnel. Use 1L of organic free water is necessary for the Blank and LCS.

8.2.4 Remove the surrogate standard and the lab control sample standard prepared in **Sections 8.1.2** and **8.1.3** from the refrigerator and allow them to reach room temperature.

- Use a syringe to add 1.0 mL of the surrogate standard into the Blank, LCS, sample and any MS/MSD sample that is in a separatory funnel. The standards should mix with the aqueous sample immediately since the solvent is methanol.
- Use another syringe to add 1.0 mL of the lab control sample standard into the LCS and any MS/MSD samples.

8.2.5 Transfer the rinsate from the graduated cylinder or the sample bottle, in **Section 8.2.3** to the separatory funnel. The bottle is saved for volume determination. For the Blank and LCS, add 60mL of methylene chloride to the separatory funnel.

8.2.6 Stopper the separatory funnel and invert. Vent the separatory funnel immediately to release the built up vapor. Shake the separatory funnel for 3 minutes, being sure to vent periodically to release the built up vapor.

8.2.7. At the end of the extraction, the separatory funnel is allowed to settle with the stopper removed. The separatory funnel should be allowed to sit undisturbed for at least 10 minutes to allow for phase separation. If an emulsion develops, use any

mechanical means, i.e. glass rod, available to break up the emulsion. If emulsions persist and do not break up with mechanical means, ask the supervisor for further instructions (may include preparation using continuous Liquid/ Liquid extraction).

8.2.8. Decant the bottom methylene chloride layer into a 500mL flask.

8.2.9. Repeat the extraction two more times with 60mL of methylene chloride each time.

8.2.10. Decant the bottom methylene chloride layer to the 500mL flask with care to limit the amount of water in the extract.

8.2.11. For Semivolatile sample extraction, the sample is then made basic with 10N NaOH to a pH > 10. Add 60mL of methylene chloride and shake for 3 minutes venting periodically.

8.2.12. Decant the bottom layer of methylene chloride into the 500mL flask. Repeat the extraction 2 more times.

8.2.13. Cover the extracts with aluminum foil until sample extract concentration.

8.2.14. Record all extraction information including prep start time in the Prep Lab logbook (**Figures 1 and 2**).

8.3. Sample Filtration and Concentration:

The sample extract is now ready for filtration and concentration. These procedures are found in SOP No. 50.0054, Extract Filtration and Concentration. Cleanup procedures can be found in SOP No. 50.0030 through 50.0034, and 50.0036.

8.4. The extracts are transferred to the Semivolatile lab. The extracts are stored in the refrigerator at 4^{o}C until analysis. The storage location is recorded electronically by the analyst during LIMS batch transfer.

8.5. Sample and Extract Disposal:

All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.

8.5.1. Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of.

8.5.2. Sample Extracts – All sample extracts are kept for 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of.

9. Data Reduction and Calculations

9.1 Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{(\text{Concentration of ampule})(\text{amount used})}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. In order to trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and contain the manufacturer's reference number given in the standard preparation log book.

10.1 Method Blank – A method blank is a volume of organic free reagent water that is carried through the entire analytical procedure. It is used to determine the level of contamination associated with the analytical processing and analysis of samples.

10.1.1 Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2 Procedure for Method Blank:

- The Method Blank is prepared in identical fashion as the associated samples.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled MB and is assigned a batch number in increasing order for each new batch of twenty samples or less.

10.1.3 Acceptance criteria for Method Blank:

All samples associated with a non-compliant Method Blank are re-extracted and reanalyzed. See the associated analytical SOP.

10.2 Surrogate – Surrogate standards are added to all samples including the Method Blank, Lab Control Sample and matrix spikes to assess the efficiency of the sample preparation and analysis procedures.

10.3. Lab Control Sample (LCS) – A Lab Control Sample is a volume of organic free reagent water that is spiked with all the target analytes and surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1. Frequency of LCS:

An LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.3.2. Procedure for LCS:

- The LCS is prepared in identical fashion as the associated samples; in addition the appropriate amount of surrogate solution prepared in **8.1.2** and appropriate amount of the lab control spike prepared in **8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled LCS and is given the corresponding batch number that is associated with the method blank.

Any sample(s) that is/are associated with a non-compliant LCS will require re-extraction and re-analysis. See the associated analytical SOP.

10.4. Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.4.1. Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Once every 20 samples, or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.4.2. Procedures for Duplicate Matrix Spikes:

- The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition
- 1.0mL of the surrogate solution prepared in **8.1.2** and 1.0mL of lab control spike prepared in **8.1.3** are added to the duplicate matrix spike samples.

- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.

11. Data Validation and Reporting

11.1 The supervisor will review data generated in the organic preparation laboratory. The Quality Control Officer will perform periodic and unscheduled reviews. These data consist of the standards preparation, volume of samples, and the volume of solvent used.

11.2 Reporting of the data will a include review by the Organic Preparation Laboratory Supervisor of the data listed in **11.1**, as well as time of extraction, sampling handling procedures, and extract handling procedures.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1. Method Blank Analysis:

All samples associated with a non-compliant Method Blank are re-extracted and reanalyzed. The analysis laboratory will inform the Organic Preparatory Laboratory when method blanks have not met accepted criteria, and require re-extraction. A re-extraction will be requested. The re-extracted samples will be labeled with the suffix RE.

12.2. Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A re-extraction will be requested The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects.

12.3. LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. Any sample(s) that is/are associated with a non-compliant LCS will require re-extraction and re-analysis. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A re-extraction will be requested.

12.4. Matrix Spike Recovery and RPD

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan: Mitkem Laboratories, A Division of Spectrum Analytical, Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

Attachments:

Attachment 1: Pesticide MS/LCS list
Figure 1: Semivolatile Extraction Logbook
Figure 2: Pesticides/PCB Extraction Logbook

Attachment 1: Pesticide MS/LCS list

Analyte	Spike conc in ug/mL
4,4´-DDD	0.4
4,4´-DDE	0.4
4,4´-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2
Mirex	0.2

Figure 1: Semivolatile Extraction Logbook

MITKEM LABORATORIES **ORGANIC PREP - SAMPLE PREPARATION: SEMIVOLATILES**

Date:	Analysis:		Method #	Aq: 3510C (SepF) 3520C (Liq/Liq) Other: Soil: 3550B (Sonic) 3540C (Soxhlet) _____		Matrix Aqueous Soil Wipe Oil Other:_____		Sodium Sulfate Lot #:	
Batch ID	LCS ID	Analyst	Spiked By	Witness	MeCl Lot#	Acetone Lot#	H_2SO_4 Lot#	NaOH Lot#	Time/Date Started: Time/Date Ended:

Lab ID	Bottle #	Sample Wt (g) / Vol (mL)	Initial pH	Surrogate Spike Added(uL)	Matrix Spike Added(uL)	H2SO4 pH<2	NaOH pH>11	Emulsion	KD/RV Prior to GPC or Fractionation Date / Analyst		GPC or Fractionation Date / Analyst		Final Concentration Date / Analyst		Final Conc. Volume (mL)	Date Extract Trans.

Comments: _____

_____

_____

_____

Logbook ID: 50.0147-01/10

Surrogate Std. ID: _____

Matrix Spike/LCS Std. ID: _____

Sonicator Tuned: Yes / No

Soxhet Cycle/Hour:_____

Drip Rate_____

Water Bath Temp:_____

Reviewed By:_____

Figure 2: Pesticides/PCB Extraction Logbook

MITKEM LABORATORIES ORGANIC PREP-SAMPLE PREPARATION:PEST/PCB

Date:	Prep Method	Aq: 3510C (SepF) 3520C (Liq/Liq) Soil: 3550B (Sonc) 3540C (Soxhlet) 3570(MSE) Other:_____	Matrix: Aqueous Soil Wipe Oil Other:_____	BATCH ID:	Date/Time Started:
Analyst	Spiked By	Witness	Solvent: Lot #	GPC Batch Number	Florisil Lot #	Analysis:	Date/Time Ended:

	Lab ID	Bottle #	Sample Wt (g) / Vol (ml)	Initial pH	Emulsion?	Initial CONC Date/ Analyst TV/ KD	Extract volume (ml)	GPC Cleanup Date / Analyst	FLORISIL Cleanup Date / Analyst	ACID (PCB only) Cleanup Date / Analyst	SULFUR (Copper) Cleanup Date / Analyst	Final CONC Date/ Analyst TV/ KD	Final extract volume (ml)	Extract Transfer Date /By

Comments:

PEST Matrix Spike Std ID:	Vol. Spiked:		
PCB Matrix Spike Std ID:	Vol. Spiked:		
PEST Surrogate Spike Std ID:	Vol. Spiked:	Sodium Sulfate lot#	
PCB Surrogate Spike Std ID:	Vol. Spiked:	Hexane lot #	L/L Drip Rate:
PEST LCS Spike Std ID:	Vol. Spiked:	Sulfuric Acid lot#	Soxhlet Cycle/Hour:
PCB LCS Spike Std ID:	Vol. Spiked:	Sulfur cleanup Copper lot #	Sonicator Tuned? Yes/No

Organic Preparation of Soil Samples by Sonication (Method 3550B)

Contents SOP NO. 50.0052

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		2/4/10
Quality Assurance Director		2/2/10
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	QAD	4/5/11			
Tim McDaniel	Prep. Supervisor	4/6/11			

Revision Record

Revision Date	Revision Description	Comments	Initials
12/12/06	Included TPH/DRO		SBL
05/08/07	Added micro tip sonic probe info, removed filtration and conc steps	Reference to SOP 50.0054	SBL
01/03/08	Surrogate and LCS spike volumes edited.	Changed conc.	SBL
4/20/09	Reference to RapidVap		SBL
5/21/09	Corrected section on LCS prep for add-on cmpds. Added more horn tuning info.		SBL
02/02/10	Revised reagent section to update grades, added sonc tuning instructions as attachment	Reagent, pesticide quality Full rev	SBL
05/10/10	Noted different wt options	Minor edit	SBL
11/24/10	Edited sulfur clean up SOP#, removed re-ext log use.	minor	SBL
4/25/11	Edited TPH/DRO surrogates	OTP only, minor	TM
12/27/11	Standard storage temp	Minor; edit to refrigeration not freezer	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Sonication (Method 3550B)

SOP No. 50.0052
Rev 3

	Signature	Date
QA Director:	Shawn B Lewis	2/2/10
Lab Director:		2/4/10
Effective Date:	02/11/10	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Sonication (Method 3550B) Rev 3

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of soil and sediment samples using ultrasonic extraction for analysis by EPA SW846 methods SW8081, SW8082, SW8015 and SW8270. Discussions include sample extraction, sample cleanup references, and the preparation of standard spiking solutions for the analysis of Total Petroleum Hydrocarbons (TPH), Diesel Range Organics (DRO), pesticide/PCB or semivolatile organic compounds in soil samples. Highly contaminated soils for MA DEP EPH analyses may also use this method.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts in the Organic Preparation Laboratory (OPREP). Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 30-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. The sample is solvent extracted three times using ultrasonic extraction. The extract is separated from the sample by gravity filtration. The extract is dried through powdered anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using TurboVap or Kuderna-Danish (KD) apparatus followed by Nitrogen blowdown. In addition, this method can be used for Medium/High Concentration Semivolatile samples. A 1-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. This is solvent extracted once using ultrasonic extraction. The extract is separated from the sample by gravity filtration. The extract is dried through anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration.

4. Sample Preservation, Containers, Handling, and Storage

4.1. The sample holding times are as follows:

- Sample must be extracted within 14 days from the time of sample collection.
- Sample extracts must be analyzed within 40 days of sample extraction.

4.2. Sediment/Soil samples – Decant and discard any water layer on the sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. Refer to **SOP No. 110.0039** for more detail on sub sampling techniques.

4.3. Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

5.1. Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2. Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results of analysis of the analytes of interest.

5.3. Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation steps to minimize interferences from these compounds.

6. Equipment and Apparatus

Instrumentation used in this preparation method include:

6.1. Ultrasonic Disrupter – pulsing horn type with 375 watts maximum and ¾" standard solid disrupter horn and ⅛" standard tapered microtip probe (medium soils only). Fisher Scientific Sonic Dismembrator Models 500 and 550.

6.2. Sonabox – ultrasonic disrupter box designed to reduce exposure to ultrasonic sound.

6.3. Beakers – 400mL

6.4. 500mL Erlenmeyer flask

6.5. Dessicator

6.6. Balance capable of weighing +/- 0.1 gram.

6.7. Glass syringes for delivering spike and surrogate solutions – 0.25mL, 0.5mL, and 1.0mL.

6.8. Volumetric flasks for making up surrogate and matrix spike solutions – 50mL, 250mL.

6.9. Aluminum Foil (Industrial Grade)

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. The following chemicals including solvents and standards are extensively used in the lab:

7.1. Methylene chloride: pesticide quality or equivalent to be used for glassware rinsing and sample extraction.

7.2. Methanol: pesticide quality or equivalent, to be used for rinsing glassware and preparing standards.

7.3. Acetone: pesticide quality or equivalent, to be used for rinsing glassware.

7.4. 1:1 v/v methylene chloride/acetone mixture, to be use for sample extraction.

7.5. Hexane: pesticide quality or equivalent , to be used for solvent exchange of samples.

7.6. H_2O, deionized.

7.7. H_2SO_4, concentrated, for sample pH adjustment.

7.8. Surrogate Standards *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatiles Full Scan: Restek BN Surrogate mix (Cat. No. 31086) at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
- Semivolatiles SIM: Cambridge Benzo (e) pyrene d-12 surrogate (Cat. No. DLM-257-S) at 200µg/mL.
- Diesel Range Organics (DRO)/TPH: o-Terphenyl at 10,000µg/mL (Made from neat source (Aldrich)).
- MA EPH: 5-α Androstane at 10,000µg/mL (Made from neat source (Sigma)), and o-Terphenyl at 10,000µg/mL (Made from neat source (Aldrich)).
- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 µg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000µg/mL in Acetone.

7.9. Lab Control Sample and Matrix Spike: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000µg/mL in Methylene Chloride; Restek 3,3' Dichlorobenzidine (Cat. No. 31026) at 2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine.
- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL.
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
- TPH/DRO: LCS uses Diesel/gasoline.
- MA EPH: An independent vendor than that used for the MA EPH calibration standards is used. The LCS consists of the 17 Aromatic Hydrocarbon compounds plus the 14 normal Aliphatic Hydrocarbons at 1000µg/mL in Methylene Chloride.

7.10. Anhydrous sodium sulfate: granular for drying the samples and sample extracts. Baked at 400°C for four hours.

8. Procedure

8.1. Standards Preparation: All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

8.1.1. All **primary standards** received from vendors are logged into the LIMS Standard Logbook. These include standards for surrogates, LCS and matrix spikes. The standards are labeled ZPyymmddX, where:

Z = S for Semivolatile or TPH/DRO, P for Pesticides or PCB
P= Primary standard
yymmdd = date standard is received, and
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2. Surrogate standard:

8.1.2.1. Semivolatile Full Scan: The Semivolatile working surrogate standard is prepared by combining 2.5mL of the Base/Neutral primary standard in **Section 7.8** and 2.5mL of the Acid primary standard in **Section 7.8** and diluting to 250mL using methanol.

The working standard contains the following compounds:

Compound	Concentration(µg/mL)
1,2-Dichlorobenzene-d_4	50
2-Fluorobiphenyl	50
Nitrobenzene-d_5	50
p-terphenyl-d_{14}	50
2-Chlorophenol-d_4	75
2-Fluorophenol	75
Phenol-d_6	75
2,4,6-Tribromophenol	75

Semivolatile SIM surrogate standard is prepared by diluting 1.25mL of the 200ug/mL primary standard in **Section 7.8** to 50mL in methanol. The resulting concentration is 5ug/mL of Benzo (e) pyrene d-12.

The working surrogate spike standard is labeled SWyymmddX where:
SW = Semivolatile working standard
yymmdd = date working standard is prepared,
X = the order that the working standard is prepared on that date, in increasing alphabetical order.

8.1.2.2. The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.8**, and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

Compound	Concentration(µg/mL)
Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

8.1.2.3. The MA EPH working surrogate standard is prepared by taking 1mL of the 5-α Androstane and 1mL of the o-Terphenyl primary standards in **Section 7.8** and diluting to 200 mL using methanol. The working standard contains the following compound:

Compound	Concentration(µg/mL)

5-α Androstane	50
o-Terphenyl	50

8.1.2.4. The TPH/DRO working surrogate standard is prepared by taking 1mL of the o-Terphenyl primary standard in **Section 7.8** and diluting to 200 mL using methanol. The working standard contains 50 µg/mL of o-Terphenyl.

A smaller or larger amount may be used to prepare the surrogate. The final volume of the surrogate solution will be adjusted accordingly.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3. Lab Control Sample (LCS) and Matrix Spike (MS) Standard:

8.1.3.1. The Semivolatile Full Scan working LCS/MS Standard is prepared by combining 5.0 mL of 8270 MegaMix, 2.5mL of 3,3' DCB, and 5.0 mL of 8270 add-on intermediate standard from **section 7.9**, and diluting to 50mL using methanol. The resulting concentration will be 50ug/mL for all SW8270 compounds. The working Semivolatile SIM LCS/MS Standard is prepared by a further 10 times dilution of the above standard.

8.1.3.2. The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2, and diluting it to 50 mL in methanol. The working standard contains the full list of SW8081 individual response compounds at 2-20 µg/mL. See **Attachment 1** for a list of the individual compounds and their concentrations.

8.1.3.3. The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.9** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

Compounds	Concentration (µg/mL)
Aroclor 1016	4.0
Aroclor 1260	4.0

8.1.3.5. The TPH/DRO working LCS/MS Standard is prepared by first preparing an intermediate standard and making a dilution of it.

On an analytical balance, weigh out 5.00g of diesel fuel into a 10mL Class A volumetric flask and bring to volume with Methanol.

The intermediate spike standard is labeled SIyymmddX

where:
SI = Semivolatile intermediate standard
yymmdd = date intermediate standard is prepared,
X = the order that the intermediate standard is prepared on that date, in increasing alphabetical order.

The working LCS/MS Standard is prepared by taking 1mL of the intermediate standard above, and diluting to 100mL using Methanol. The working LCS/MS Standard contains the following:

Compounds	Concentration(µg/mL)
Diesel fuel	5000

The working MA EPH LCS/MS Standard is prepared by taking 2.5mL of the Aliphatic Hydrocarbons standard in Section **7.9** and 2.5mL of the Aromatic Hydrocarbon standard in Section **7.9** and diluting to 250mL in 1:1 methylene chloride: acetone mixture . The final concentration will be 50 ug/ml for each of the individual components.

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2.1.**

A smaller or larger amount may be used to prepare the Lab Control Sample and Matrix Spike Standard. The final volume will be adjusted accordingly.

The standard solution is placed in amber bottles and stored in the freezer in the GC or GC/MS lab at –10 to –20^{o}C. One bottle is transferred to the OPREP Lab and stored in the refrigerator at 4± 2°C. The bottles are stored in a separate location from samples or sample extracts to make sure that there is no cross contamination.

The expiration date for the ampulated solutions is discussed in **Section 8.1.1.**

All of the appropriate standard preparation information is to be recorded in the appropriate Lab's working Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2. Sample Extraction Procedure:

8.2.1. Low Level Method: Unless Mitkem has documented history that the samples contain high concentration of target analytes (>10,000µg/Kg for the individual analyte) all of the semivolatile samples are prepared using the low-level approach.

Before starting:

- The disrupter has a minimum of 375 watts power and must be inspected periodically to ensure that the ¾ inch tip has not experienced excessive wear.
- The disrupter is tuned (or verified as with the Fisher 500) each day of use. Follow manufacturer's guidance for tuning procedures (see instructions posted by Sonicator). The tuning is documented in the Sonicator Horn Tuning Logbook, **Figure 4**. After tuning, note in extraction logbook as well.
- Remove the surrogate and LCS/MS spiking standards from the refrigerator and allow them to reach room temperature.

8.2.1.1. The sample is mixed to ensure sample homogeneity. A representative portion of the sample is measured into a pre-weighed 400mL glass beaker. A 30 gram ± 0.5 gram sample is transferred into the beaker using a clean stainless steel scoopula. The weight measurement is recorded to the nearest 0.1 gram. Calibrate the balance prior to use. Refer to **SOP No. 110.0007** for direction.

NOTE: A smaller sample size may be used as long as all surrogates and final volumes are modified appropriately. Associated QC samples should be adjusted similarly. Where appropriate or allowed, using a smaller sample volume can help to reduce solvent use and thereby minimize solvent waste.

8.2.1.2. Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. When mixed with the anhydrous sodium sulfate, the solid material must be a free flowing; however, it should not contain an excessive amount of anhydrous sodium sulfate. If a sample matrix presents difficulty, discuss the issue with the OPREP Supervisor or Mitkem's Technical Director before proceeding.

8.2.1.3. According to the test method being used, add the following spikes to the samples in beakers:

- Use a syringe to add 1.0mL of the surrogate standard to the Blank, LCS, samples and any MS/MSD sample.

- Use a syringe to add 1.0mL of the LCS/MS standard to the LCS, and any MS/MSD sample.

8.2.1.4 Add 100mL of the 1:1 v/v methylene chloride/acetone mixture to the Blank, LCS, samples, and any MS/MSD for Semivolatile or Pesticide/PCB extraction, OR add 100mL of methylene chloride to the Blank, LCS, samples, and any MS/MSD for TPH/DRO extraction.

8.2.1.5 Place the bottom surface of the ¾ inch tip of the disrupter about ½ inch

below the surface of the solvent, but above the solid layer.

8.2.1.6 Extract ultrasonically for 3 minutes, with the output control set at full power and the mode switch on Pulse, using a 50% duty cycle.

8.2.1.7 Remove the beaker from the disrupter. Decant the solvent layer into a 500mL Erlenmeyer flask, leaving the solid in the beaker.

8.2.1.8 Extract the sample two more times with 100mL of solvent and a 3 minute sonication time. Decant the solvent aliquots into the 500mL Erlenmeyer flask. Cover the flask with aluminum foil.

8.3. Semivolatile Medium Level Method: (Also refer to **SOP No. 50.0100**, Method SW846 3570 Microscale Solvent Extraction for another soil extraction option using small sample volumes.)

8.3.1. The sample is mixed to ensure sample homogeneity. As representative as possible, weigh about a 1 gram portion of the sample into a pre-weighed 15mL vial. The weight measurement is recorded to the nearest 0.1 gram.

8.3.2. Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. Mix the sample and anhydrous sodium sulfate to achieve a free flowing mixture.

8.3.3. Use a syringe to add 1.0mL of the surrogate standard into the Blank, LCS, sample and any MS/MSD sample that is in the 40mL vial.

8.3.4. Use a syringe to add 1.0mL of the lab control sample standard into the LCS and any MS/MSD samples.

8.3.5. Add 9mL of methylene chloride to the Blank and samples. Add 8mL of methylene chloride to the LCS and any MS/MSD.

8.3.6. Sonicate the sample with the ⅛ inch tapered Microtip for 2 minutes at output control setting at 5 in the continuous mode.

8.3.7. Cap the vial.

8.3.8. Record all extraction information including prep start time in the Prep Lab bench sheet (see **Figures 1 and 2**).

8.4. The sample extract is now ready for filtration and concentration. These procedures are found in SOP No. 50.0054, Extract Filtration and Concentration.

8.5 After concentration the extract may require cleanup. The following is a list of Cleanup Procedures**:**

8.5.1. **Sulfur cleanup** is mandatory for all Pesticide/PCB sample extracts containing sulfur. All QC samples including blanks, lab control samples, matrix spikes and duplicate matrix spikes must be subjected to the same cleanup as the field samples. Sulfur cleanup will be performed using activated copper powder. Refer to SOP No. 50.0036 Method 3660B Sulfur Cleanup, for details on activating copper and using it in a sample extract.

8.5.2. **Acid cleanup (PCB extract only)** is mandatory for all PCB sample extracts. Refer to SOP No. 50.0031 Method 3665A Sulfuric Acid Cleanup, for details on using the acid cleanup in a PCB sample extract.

8.5.3. **GPC cleanup** is useful for both Pesticide and semivolatile samples. Refer to SOP No. 50.0032 Method 3640A GPC Cleanup, for details on the procedure for cleanup and quality control criteria for GPC.

8.5.4. Other cleanup methods may be found in SOP numbers 50.0033(Florisil) and 50.0034 (Silica Gel).

8.6 The extracts are transferred to the Semivolatile lab. The extracts are stored in the refrigerator at 4^{o}C until analysis. The storage location is recorded electronically by the analyst during LIMS batch transfer.

8.7 Sample and Extract Disposal:

All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.

8.7.1. Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of by the Sample Custodian or his/her designee.

8.7.2. Sample Extracts – All sample extracts are kept for at least 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of by the GC or GC/MS labs.

9. Data Reduction and Calculations

Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{(\text{Concentration of ampule})(\text{Amount used})}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. To trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and the manufacturer's reference number is documented in the standard preparation log book.

10.1. Method Blank – A method blank is a weight of a clean reference matrix (granular sodium sulfate) that is carried through the entire analytical procedure.

10.1.1. Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2. Procedure for Method Blank:

- The Method Blank is prepared in identical fashion to the associated samples.
- An aliquot of the surrogate standard prepared in **section 8.1.2** is added to the Method Blank.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a sequential number for every batch of twenty samples or less.

10.2. Lab Control Sample (LCS) – A Lab Control Sample is a weight of a clean reference matrix (granular sodium sulfate) that is spiked with all appropriate target analytes and surrogate spikes, and carried through the entire analytical procedure.

10.2.1. Frequency of LCS:

An LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.2.2. Procedure for LCS:

The LCS is prepared in identical fashion as the associated samples; and in addition:

- An aliquot of the surrogate standard prepared in **section 8.1.2** and the lab control standard prepared in **section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the same numerical value as the corresponding method blank.
- The LCS is analyzed and the results are calculated for the recovery of all spiked analytes in the LCS.

10.3. Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.3.1. Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.3.2. Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition:

- An aliquot of the surrogate standard prepared in **section 8.1.2** and the lab control standard prepared in **section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.
- The duplicate matrix spikes are analyzed and the results calculated for the recovery of the spiked analytes in the duplicate matrix spike.

11. Data Validation and Reporting

11.1. Data generated in the organic preparation laboratory will be reviewed by the supervisor. These data consist of, but are not limited to, extraction/preparation logbook entries, balance calibration logbooks, weights for soil samples, volumes and lot numbers of

solvent used. The Quality Control Officer will perform periodic and unscheduled reviews.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1. Method Blank Analysis:

All samples that are prepared with a non-compliant Method Blank will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when method blank contamination has occurred.

12.2. Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction.

12.3. LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction.

12.4. Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan; Mitkem Laboratories, A Division of Spectrum Analytical Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

Fisher Scientific Instruction Manual Model 500 Ultrasonic Dismembrator.

Fisher Scientific Instruction Manual Model 550 Ultrasonic Dismembrator.

Attachments:

Attachment 1: Pesticide LCS/MS List
Attachment 2: Sonicator Tuning instructions
Figure 1: Semivolatile Extraction Log
Figure 2: Pesticides/PCB Extraction Log
Figure 3: Sonicator Tuning Log

Attachment 1: Pesticide LCS/MS List

Analyte	Spike conc in ug/mL
4,4´-DDD	0.4
4,4´-DDE	0.4
4,4´-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2

Attachment 2
Sonicator Tuning instructions

The User I/O may be used to remotely control the system. If this is the case, you must design in whatever safety precautions are appropriate to your User I/O circuit design to prevent unexpected start-up, which can cause personal injury and can cause equipment damage.

3.8 Ultrasonic Test

The Test button on the front panel of the Model 500 is used to verify that the unit is functioning (providing ultrasonic energy to the converter and horn). You can also run another test on the system for your particular experiment as described later. Before testing the Model 500, always make sure that the horn is not touching anything. The system will perform several self-tests when it is first turned on.

Step	Do this...	To obtain this result
1	Set up the Model 500 following the instructions in this manual. If no horn is currently installed, mount a 1/2" disruptor horn to the converter.	Prepare the Model 500 to operate, if it was not previously assembled.
2	After you have connected the converter/horn to the converter cable, verified all other connections are as desired: Turn the unit on, and observe the self-test displays.	Verify that the system passes all its self-tests, observing that there are no error messages on the front panel display. The Model 500 advances to the ready mode and shows the normal "Ready" display.
3	Adjust the amplitude control to approximately 50% (observe the value on the front panel display).	Ensures that ultrasonic energy will be at some mid-range value, and will not cause damage if you were using a microtip (must be less than 70%).
4	Verify that the horn is not touching anything. Press the test button on the front panel. Observe the front panel display.	Verifies the ultrasonic output of the system. You may hear a soft, high-pitched sound. The bargraph display will show some output value. The test will run for 2 seconds, then stop.
5	If the system showed readings on the display during the test, you may either proceed with your experiments or turn the unit off.	Verification that the Model 500 Dismembrator is operating and is ready to be set up for your experiment.

Sonic Dismembrator
Fisher Scientific
Model 500

F. TUNING INSTRUCTIONS - (for standard probes, Q-horns and cuphorns)

To assure optimum operation tune the generator in accordance to the following procedure each time a new probe is changed.

The probe or microtip should not be immersed in the liquid or come in contact with the work surface when tuning.

When operating with liquids at extreme temperatures, immerse the probe in the liquid for a few minutes, remove from the liquid then tune.

1. Turn OUTPUT CONTROL knob counter-clockwise to zero.

2. Press POWER SWITCH to ON (up) position. The switch will illuminate.

3. When the prompt [for tuning procedure refer to manual] appears, press TUNE key. Screen will read:
   [TUNING - - - PROBE ACTIVE].

4. Turn the Output Control Knob towards setting 3.
   a) Note the position of the Bar Graph on the LCD Display Screen. Do NOT exceed 70%.
   b) Rotate the Tuning Knob clockwise or counterclockwise until a minimum (not maximum) reading (usually less than 20%) is obtained.

5. Turn Output Control Knob towards setting 6.
   a) Again, note the position of the Bar Graph and do not exceed 70%.
   b) Rotate the tuning knob till you obtain a meter reading of 20% or below.

6. Repeat step 5 on power setting 10. Minimize the reading one last time to 20% or less.

7. Press the TUNE key to display prompt for programmed or continuous operation.

YOUR SONIC DISMEMBRATOR IS NOW TUNED

SPECIAL NOTE FOR TUNING MICROTIP™ PROBES AND OTHER HORNS

MICROTIPS

The above procedure must be followed with the exception that the OUTPUT CONTROL should NEVER exceed the MICROTIP LIMIT (5). Tuning at the MICROTIP LIMIT should be done as quickly as possible. Prolonged operation in air at the limit can cause MICROTIP failure.

CUPHORNS

Drain filled cups down to outlet fitting level, tune, and refill.

Figure 1

Semivolatile Extraction Log

MITKEM LABORATORIES **ORGANIC PREP - SAMPLE PREPARATION: SEMIVOLATILES**

Date:	Analysis:		Method #	Aq: 3510C (SepF) 3520C (Liq/Liq) Other: Soil: 3550B (Sonic) 3540C (Soxhlet) _____		Matrix Aqueous Soil Wipe Oil Other:_____			Sodium Sulfate Lot #:
Batch ID	LCS ID	Analyst	Spiked By	Witness	MeCl Lot#	Acetone Lot#	H_2SO_4 Lot#	NaOH Lot#	Time/Date Started: Time/Date Ended:

Lab ID	Bottle #	Sample Wt (g) / Vol (mL)	Initial pH	Surrogate Spike Added(uL)	Matrix Spike Added(uL)	H2SO4 pH<2	NaOH pH>11	Emulsion	KD/RV Prior to GPC or Fractionation Date / Analyst		GPC or Fractionation Date / Analyst		Final Concentration Date / Analyst		Final Conc. Volume (mL)	Date Extract Trans.

Comments: _____

Surrogate Std. ID: _____

Matrix Spike/LCS Std. ID: _____

Sonicator Tuned: Yes / No

Soxhet Cycle/Hour:_____

Drip Rate_____

Water Bath Temp:_____

Figure 2
Pest/PCB Extraction Log

MITKEM LABORATORIES ORGANIC PREP-SAMPLE PREPARATION:PEST/PCB

Date:	Prep Method	Aq: 3510C (SepF) 3520C (Liq/Liq) Soil: 3550B (Sonc) 3540C (Soxhlet) 3570(MSE) Other:_____	Matrix: Aqueous Soil Wipe Oil Other:_____	BATCH ID:	Date/Time Started:
Analyst	Spiked By	Witness	Solvent: Lot #	GPC Batch Number	Florisil Lot #	Analysis:	Date/Time Ended:

	Lab ID	Bottle #	Sample Wt (g) / Vol (ml)	Initial pH	Emulsion?	Initial CONC Date/ Analyst TV/ KD	Extract volume (ml)	GPC Cleanup Date / Analyst	FLORISIL Cleanup Date / Analyst	ACID (PCB only) Cleanup Date / Analyst	SULFUR (Copper) Cleanup Date / Analyst	Final CONC Date/ Analyst TV/ KD	Final extract volume (ml)	Extract Transfer Date /By

Comments:

PEST Matrix Spike Std ID:	Vol. Spiked:		
PCB Matrix Spike Std ID:	Vol. Spiked:		
PEST Surrogate Spike Std ID:	Vol. Spiked:	Sodium Sulfate lot#	
PCB Surrogate Spike Std ID:	Vol. Spiked:	Hexane lot #	L/L Drip Rate:
PEST LCS Spike Std ID:	Vol. Spiked:	Sulfuric Acid lot#	Soxhlet Cycle/Hour:
PCB LCS Spike Std ID:	Vol. Spiked:	Sulfur cleanup Copper lot #	Sonicator Tuned? Yes/No

Figure 3:
Sonicator Tuning Log

Mitkem Laboratories Organic Prep Sonicator Horn Tuning Logbook

See individual Manufacturer Manuals* for instructions on tuning, and passing criteria.

Date	Horn ID	Pass	Fail	Comments	Analyst

Logbook ID: 50.0014-09/09 **Reviewed by:** _____

*POSTED BESIDE HOOD

Organic Extract Filtration and Concentration Techniques

Contents SOP NO. 50.0054

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		8/19/11
Quality Assurance Director		8/19/11
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
Tom McDaniel	Supervisor	8/10/12			

Revision Record

Revision Date	Revision Description	Comments	Initials
4/24/08	Lab Name change, removed specific revision letters of the SW846 3500 and 3600 series, updated to SOM01.2		SBL
2/26/09	Replaced Rapid Vaps with Turbo Vaps		SBL
11/6/09	Corrected SV intermediate extract volume as 1ml and pest/PCB as below 5		SBL
02/02/10	Updated reagents and solvent grades	Section 7	SBL
05/10/10	Minor edit: added sentence regarding different initial volume:final vol scenarios.	Section 8	SBL
8/17/11	Added special 1,4-dioxane concentration step. Revised QA/QC and CA sections.	New section 12, rest are renumbered. Lab name change.	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

Spectrum Analytical, Inc.

Featuring Hanibal Technology
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Organic Extract Filtration and Concentration Techniques

Rev 3

	Signature	Date
QA Director:	Shawn B Lamb	8/19/11
Lab Director:		8/19/11
Effective Date:	8/26/11	

Spectrum Analytical, Inc.

Featuring Hanibal Technology
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Organic Extract Filtration and Concentration Techniques

Rev. 3

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the filtration and concentration of organic sample extracts that were prepared using EPA water and SW-846 solid hazardous waste methods, as well as OLC3.2, OLM4.3 and the current SOM version procedures.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

The sample extract is dried through granular anhydrous sodium sulfate and is made ready for cleanup and/or analysis following concentration using Kuderna-Danish (KD) apparatus or Caliper TurboVap apparatus and nitrogen blow down.

4. Sample Preservation, Containers, Handling, and Storage

Not Applicable

5. Interferences and Potential Problems

5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation and concentration; therefore, these materials

must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results for the analytes of interest. See SOP Nos. 50.0030 through 50.0034 for cleanup methods.

5.3 Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation and concentration steps to minimize interferences from these compounds.

6. Equipment and Apparatus

Equipment and apparatus used in this method include:

6.1 Teflon boiling chips.

6.2 Kuderna-Danish (KD) apparatus with a 10 or 15ml receiver tube.

6.3 80mm Glass funnel.

6.4 Glass wool.

6.5 Three ball Snyder column.

6.6 Water bath, capable of maintaining 60 ^{o}C to 90^{o}C.

6.7 Boiling chips, carbon.

6.8 Nitrogen blow-down apparatus, N-EVAP Model No. 111.

6.9 Receiving vial with Teflon septa – 2ml.

6.10 15ml storage vials with screw caps.

6.11 Caliper TurboVap apparatus with 200mL collection tube

6.12 Aluminum Foil (Industrial Grade)

6.13 Glass Pipettes

6.14 20X150mm Disposable Culture Tubes

6.15 Thermometer to check temperature of water bath and Nitrogen blow-down apparatus.

7. Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. The following chemicals including solvent, standards and gasses are extensively used in the lab:

7.1. Methylene chloride: pesticide quality or equivalent, to be used for glassware rinsing and sample extraction

7.2. Hexane: pesticide quality or equivalent, to be used for solvent exchange of samples.

7.3. Anhydrous sodium sulfate, granular for drying the sample extract. Baked at 400°C for at least four hours.

7.4. Acetone: pesticide quality or equivalent, to be used for glassware rinsing.

8. Procedure

Note: Final extract volumes given below are those associated with 1000 ml or 30 g initial volume/wt samples. The final extract volumes for smaller initial volume/wt samples would generally be subjected to further concentration to maintain the same initial: final volume ratio. For example, a 15g PCB sample would be concentrated to a final 5 ml volume instead of 30g to 10 ml.

8.1 Sample Extract Filtration:

All sample extracts are dried through anhydrous sodium sulfate prior to further sample concentration.

8.1.1. A drying funnel is first cleaned with methylene chloride to remove any contaminants. The drying funnel is then prepared by plugging an 80mm diameter filtering funnel with glass wool and adding approximately 20 grams of anhydrous granular sodium sulfate.

8.1.2. Sample extracts are dried through the drying funnels and collected directly into the KD concentrators or Caliper TurboVap collection tubes depending on which method of concentration is going to be used. Rinse the empty boiling flask and drying funnel with methylene chloride after the initial transfer to ensure the qualitative transfer of the extract.

8.1.3. The KD concentrator apparatus consist of a 500ml KD flask and a 10 or 15ml concentrator tube. The concentrator tube is attached to the flask by placing it on and then giving a gentle twist. The concentrator tube should be on snug. Place a blue Keck clamp to ensure the tube will not fall off. Continue with **Section 8.2.1**.

8.1.4. The Caliper TurboVap 200mL collection tubes are placed underneath the drying funnel and the extract is collected directly into it. Continue with **Section 8.2.2**.

8.2. Sample Extract Concentration:

8.2.1. Procedure for concentration of sample extracts using **KD** apparatus without further Gel Permeation Chromatography (GPC) cleanup.

8.2.1.1. Add 2 boiling chips to the KD flask containing the extraction fluid.

8.2.1.2. Attach a three-ball Snyder column to the KD flask and submerge the evaporator into the hot water bath set at medium heat (recommended temperatures are: 60-70 ^{o}C for SV (or DRO) and as high as 80-90^{o}C for Pest/PCB during and after hexane exchange) with the tip of the KD concentrator tube in the water.

8.2.1.2.1. Concentrate the **SV (or TPH/DRO)** extract down to less than 5ml.

8.2.1.2.2. Concentrate the **Pest/PCB** extract down to less than 5ml and then add approximately 20ml (or more) of hexane through the top of the Snyder column to exchange the solvent. Continue to concentrate the extract to less than 5ml. REPEAT.

Caution: It is important that the extract in the KD apparatus not be allowed to go dry to minimize volatilization of the more volatile target compounds.

8.2.1.3. Remove the evaporator from the water bath. Let the evaporator cool for at least 10 minutes, allowing the solvent in the Snyder column to drain back into the KD concentrator tube. Rinse the KD flask with a small volume of methylene chloride.

8.2.1.4. Remove the three-ball Snyder column and the KD flask from the concentrator tube. Transfer the extract into a 20x150mm disposable culture tube. Rinse the lower joint of the KD flask and the concentrator tube with another small amount of methylene chloride to ensure a full transfer of the extract.

8.2.1.5. Place the culture tube into the nitrogen blow down bath, set at 30-35^{o}C (recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8.

8.2.1.5.1. For samples prepared for 1,4-dioxane analysis, do NOT place culture tube into nitrogen blow down bath. The heat of the water bath will result in loss of the target analyte.

8.2.1.6. Start a low flow stream of nitrogen across the surface of the extraction solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulate inside the tube.

Caution: it is important that the extract in the culture tube not be allowed to go dry to minimize loss of the more volatile target compounds.

8.2.1.7. Concentrate the **SV (or TPH/DRO)** extract to between 0.5 and 0.8ml. Pull the extract up with a 1.0ml syringe. Rinse the collection tube with a small volume of methylene chloride and pull up the rinsate filling the syringe to the 1.0ml mark. Transfer the 1.0ml extract to a 2ml auto-sampler vial. Mark the meniscus with a permanent marker.

8.2.1.8. Concentrate the **Pest/PCB** extract to 5ml. Transfer the extract to a 15ml vial marked at the 10ml mark. Rinse the concentrator tube with 1ml of hexane and add to vial. Rinse the concentrator tube once more. Bring the final extract volume up to 10ml with hexane. Mark the meniscus with a permanent marker.

8.2.2. Procedure for concentration of sample extracts using TurboVap (TV) apparatus without Gel Permeation Chromatography (GPC) cleanup:

8.2.2.1. Place the 200mL TV collection tube into the TurboVap. Set the nitrogen to 10 psi, and the temperature to 60 ^{o}C (recommended).

8.2.2.1.1. Concentrate the **Semivolatile** extracts to 0.5 – 0.8mL. Allow the collection tube to stand without nitrogen flow to cool. Using a 1.0 ml syringe pull up the extract and rinse the interior of the collection tube with the extract. Pull up the extract again with the 1.0 ml syringe. Rinse the collection tube with additional methylene chloride and pull up in the syringe. If needed, add additional methylene chloride to bring the extract to 1.0 ml. Transfer the extract to a 2 ml crimp top autosampler vial. Mark the meniscus with a permanent marker.

8.2.2.1.2. Concentrate the **Pesticide/PCB extract** to about 4ml and then add 20ml of hexane. Raise the temperature to 80 ^{o}C (recommended), if necessary, to complete the concentration. Concentrate to about 4 ml again, and add 20 ml of hexane. Continue to concentrate the extract as in **Section 8.2.1.5.2.**

Caution: it is important that the extract in the collection tube not be allowed to go dry to minimize volatilization of the more volatile target compounds.

8.2.3. Procedure for concentrating sample extracts using the **KD** apparatus with further Gel Permeation Chromatography (GPC) cleanup:

8.2.3.1. Add 2 boiling chips to the KD flask containing the extraction fluid.

8.2.3.2. Attach a three-ball Snyder column to the flask and submerge the evaporator into the water bath set at medium heat (recommended temperatures are: 60-70 ^{o}C for SV (or DRO) and as high as 80-90^{o}C for Pest/PCB (during and after hexane exchange) with the tip of the KD concentrator tube in the water.

8.2.3.3. Concentrate the extract down to less than 5ml and remove the evaporator from the water bath.

8.2.3.4. Let the evaporator cool for at least 10 minutes, and allow the solvent in the Snyder column to drain back into the concentrator tube. Rinse the KD flask with a small volume of methylene chloride to ensure a full transfer of the extract.

8.2.3.5. Remove the three-ball Snyder column and the KD flask from the concentrator tube. Transfer the extract into a disposable culture tube and place the culture tube into the nitrogen blow down bath, with the temperature set at 30-35^{o}C(recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8. Rinse the lower joint of the KD flask and the concentrator tube with a small amount of methylene chloride to ensure a full transfer of the extract.

8.2.3.6. Start a low flow stream of nitrogen across the surface of the extract solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulates inside the tube. Concentrate the samples to 2ml. This is to reduce the concentration of acetone in the Pre-GPC soil extract.

8.2.3.7. Transfer the 2ml extract into a 15ml vial with a line at the 10ml mark. Rinse the concentrator tube with 2ml of methylene chloride and combine the rinsate into the vial. Rinse the concentrator tube one more time with 2ml of methylene chloride and pour into the vial. Bring up to the 10ml mark with methylene chloride.

8.2.3.8. The extract is now ready for GPC cleanup. Refer to SOP 50.0032 for how to perform GPC.

8.2.4. Procedure for concentrating sample extracts using the **TV** apparatus with further Gel Permeation Chromatography (GPC) cleanup:

8.2.4.1. Place the 200mL TV collection tube into the TurboVap. Set the nitrogen to 10 psi, and the temperature to 60 ^{o}C (recommended).

8.2.4.2. Concentrate the extract down to about 2-3 ml and remove the collection tube from the nitrogen blow down bath. Allow to cool without nitrogen flow.

8.2.4.3. Pipet or carefully pour the extract into a 15mL vial marked with a line at the 10ml mark. Rinse the concentrator tube with 2mL of methylene chloride and pour into the vial. Rinse the concentrator tube one more time with 2mL of methylene chloride and pour into the vial. Bring up to the 10mL mark with methylene chloride.

8.2.4.4. The extract is now ready for Gel Permeation Chromatography (GPC) cleanup. Refer to SOP 50.0032 for how to perform GPC Method SW3640.

8.2.5. Post GPC the extracts require additional concentration steps.

8.2.5.1. For **Semivolatile** extracts, 5ml of the original 10 ml extract was used for cleanup and the final extract volume must be 0.5ml in methylene chloride.

8.2.5.1.1. Semivolatile extracts (TPH/DRO samples do not undergo GPC Cleanup) are re-concentrated using the KD until approximately 4ml.

8.2.5.1.2. The extract is then transferred to 20x150mm disposable culture tubes and placed into the nitrogen blow down bath, set at 30-35^{o}C(recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8.

8.2.5.1.3. Start a low flow stream of nitrogen across the surface of the extraction solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulate inside the tube.

Caution: it is important that the extract in the collection tube not be allowed to go dry to minimize volatilization of the more volatile target compounds.

8.2.5.1.4. Concentrate the **SV** extract to a final volume of 0.5ml. Pull the extract up with a 1.0ml syringe. Rinse the collection tube with a small volume of methylene chloride and pull up the rinsate filling the syringe to the 0.5ml mark. Transfer the extract to a 2ml auto-sampler vial. Mark the meniscus with a permanent marker.

8.2.5.2. For **Pesticide/PCB** extracts, 5ml of the original 10 ml extract was diluted to a 10ml final volume using methylene chloride, and was used for GPC cleanup. The final extract should be solvent exchanged to hexane and concentrated to 5ml.

8.2.5.2.1. The post GPC extract is transferred to a TV apparatus with the temperature set to 60 ^{o}C (recommended). Concentrate to about 4 ml. Raise the temperature to 80 ^{o}C (recommended), if necessary, to complete the concentration. Add 20ml of hexane to exchange the solvent. REPEAT. Continue to concentrate the extract until it has

reached 2-3ml. Transfer the extract to a 15mL vial marked at the 5mL mark. Rinse the collection tube with hexane several times to ensure a full transfer. The final volume must be 5ml. Mark the meniscus with a permanent marker.

8.2.5.2.2. The extract is ready for florisil cleanup. Refer to SOP 50.0033 for detailed instructions on florisil cleanup, Method SW3620.

8.2.5.2.3. Other cleanup methods may be employed if needed. Possible procedures may be: Silica Gel Cleanup**,** Method SW3630 (SOP 50.0034), Acid Cleanup, Method SW3665 (SOP 50.0031) or Sulfur Cleanup, Method SW3660 (SOP 50.0030).

8.2.6. The extracts are transferred to the GC or GC/MS lab. The extracts are stored in the appropriate refrigerator, at 4^{o}C ± 2 ^{o}C until analysis. The storage location is documented in the Prep Batch logbook electronically.

8.2.7. Excess volume of extracts are stored in screw cap vials and labeled with color coded lab tape. The color of the tape records the extract holding time until disposal. See chart in laboratory.

8.3. Sample and Extract Disposal:

All sample extracts are disposed of in accordance with applicable OSHA and state regulations. All sample extracts must be protected from light and stored at 4°C ± 2°C.

8.3.1. Sample Extracts – All sample extracts are kept for 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of.

8.3.2. EPA CLP/SOM sample extracts- All sample extracts are kept until 365 days after delivery of a reconciled, complete data package.

9. Data Reduction and Calculations

Not Applicable

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. See OPREP extraction method SOPs for details of QC samples and their appropriate frequency and procedures. QC samples are filtered and concentrated in the same manner as field samples.

11. Data Validation and Reporting

11.1 Data generated in the organic preparation laboratory will be reviewed by the supervisor. These data consist of the final volume of sample extracts, the volume and lot number of solvents used, and extract transfer dates. The QA Director will perform periodic and unscheduled reviews.

11.2 Reporting of the data will include review by the Organic Preparation Laboratory Supervisor of the data listed in **Section 11.1**, time of extraction, sampling handling procedures, and extract handling procedures.

12. Data Management and Records Management

12.1 All preparation information is documented in the individual Prep Batch Logbook regardless of sample acceptance. Batch sheets are scanned to the network once completed. No batches are deleted from the logbooks.

12.2 Electronic data generated from the preparation of organic extracts (QC, samples, batch sheets…) is saved and managed per SOP 110.0029 Electronic Data Management.

13. Corrective Action Procedures

Discrepancy reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a discrepancy report for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007 Corrective Action Procedures. Corrective actions are recorded in the LIMS system in the Quality Control section/corrective action reports. All employees have access to LIMS and may initiate a corrective action. If help is needed, see the QA Director for assistance.

14. Health and Safety

The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined. Each chemical should be treated as a potential health hazard. Health and safety hazards in the Semivolatile Organic Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

15. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 (Waste Management) and 20.0 (Definitions, Acronyms, and Abbreviations) of the current Quality Assurance Plan.

16. References

Quality Assurance Plan, Spectrum Analytical, Inc RI Division (current version).

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, IV, or On-line Revisions of 3500 and 3600 series for Organic Extraction, Sample Preparation and Cleanup.

USEPA Statement of Work, Current OLC, OLM and SOM Methods.

Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron Capture Detector (GC/ECD) Analysis using SW846 Method 8082A

Contents SOP NO. 60.0003

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		4/12/11
Quality Assurance Director		4/11/11
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	Analyst	4/20/12			

Minor Revision Record

Revision Date	Revision Description	Comments	Initials
01/17/03	Added control page and renamed SOP		
07/25/03	Removed Tables 3 and 4 and Figure 12.		
9/08/03	Minor text changes		
3/27/08	Lab name changes, 8082A updates		SBL
9/12/08	Standard info changes to match SOM		SBL
1/5/09	RSD calculation for Form X/ LIMS form option	Using SOM %D	SBL
11/24/09	Added in QSM4.1 info	Attachment 2 replaced	SBL
07/29/10	Calibration edits, lowered calibration range	Revised tables for DoD, chromatograms of all Aroclors included. Full revision	SBL
04/11/11	Revised RT window info, LIMS std log	Full	SBL
07/01/11	Revised standard preparation scheme slightly. Added new sect 12	Removed E1 from use, minor edit	SBL
4/17/12	Second source standards changed. 3 min initial GC hold	Minor	SBL
03/29/13	Update of instruments	Minor	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron Capture Detector (GC/ECD) Analysis using SW846 Method 8082A

SOP No. 60.0003

Rev. 10

	Signature	Date
QA Director:	Sharon B Fowle	4/11/11
Lab Director:		4/12/11
Effective Date:	4/19/11	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron Capture Detector (GC/ECD)

Analysis using SW846 Method 8082A

Rev. 10

1. Scope and Application

This SOP describes the procedures applicable to the analysis of the compounds listed in **Table 1**. This SOP describes the analysis of polychlorinated biphenyls (PCB) as Aroclors in solid or aqueous sample extracts, using a gas chromatograph equipped with an electron capture detector. This SOP gives specific information to perform the analysis according to protocols discussed in USEPA SW846 Test Methods for Evaluating Solid Waste Method 8082A, and Department of Defense Quality System Manual for Environmental Laboratories, Final Version 4.1. All matrices including ground water, aqueous samples, TCLP and SPLP extracts, petroleum oil, wipes, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require extraction and/or clean-up prior to analysis. **Section 8.2.1** provides the SOP references for sample extraction and clean-up procedures to be used with this analytical procedure. **Section 10** provides the quality control (QC) requirements required by Method 8082A. A list of acronyms used in this SOP is included in **Table 2**.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that this SOP is accurate and up-to-date, and that it is implemented appropriately. **Supervisors and Peer analysts** review the logbooks and data generated from this procedure and approve all reported results. The **Data Reviewer** evaluates all laboratory reports for reasonableness of the results. The **Project Manager** reviews the final report and signs the narrative. The **QA Director** reviews all quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure

3.1. A sample extract is analyzed by injecting a 1-2µL aliquot into a gas chromatograph (GC) using an auto-sampler. Concentrations of various PCB Aroclors are determined by separation of the analytes using a GC equipped with fused silica, open-tubular, megabore columns and electron capture detectors (ECD). Hewlett Packard's Chem-Station (G1034C Version C03.00) is used to handle data acquisition. Target software from ThruPut (Revision 4.14) is used for data reduction and forms generation is through LIMS.

3.2. While SW846 Method 8082 is also applicable to determining polychlorinated biphenyls as congeners, this SOP specifically analyzes PCB as Aroclors.

3.3. PCB Aroclors are determined using dual-column system with dissimilar phases. The method allows for the option of dual columns joined to a single injection port and individually connected to two ECDs

4. Sample Preservation, Containers, Handling and Storage

4.1. Samples are collected by clients and submitted for analysis in pre-cleaned sample containers provided by the laboratory. In some instances, clients provide their own containers. For PCB analysis by USEPA SW846 Method 8082A, water samples are collected in 1-liter amber glass bottles with no preservation added to the samples. Solid samples are collected in 8-ounce amber glass containers with no preservation needed. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may be required for the analysis of laboratory QC samples.

4.2. All sample extracts are stored at 4°C ± 2°C until analyzed.

4.3 Sample extract holding time for PCB analysis by the method is 40 days from date of extraction to date of analysis. The holding time for sample extraction is covered in the corresponding extraction SOP.

4.4 The sample extracts are transferred from Organic Prep Laboratory with all appropriate sample prep information in 2ml auto-sampler vials with Teflon lined crimp cap. All vials should have a meniscus to mark the level of the extracts.

4.5 Samples, sample extracts and standards must be stored separately.

5. Interferences and Potential Problems

5.1 Sources of interference in this method can be grouped into three broad categories: (1) contaminated solvents, reagents, or sample processing hardware; (2) contaminated GC carrier gas, parts, column surfaces or detector surfaces; and (3) the presence of co-eluting compounds in the sample matrix to which the

ECD will respond. Interferences co-extracted from the samples will vary considerably from waste to waste. While general clean-up techniques are referenced or provided as part of this method, unique samples may require additional clean-up approaches to achieve the desired degree of discrimination and quantitation.

5.2. Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determination. These materials may be removed prior to analysis using Gel Permeation Clean-up (Method 3640). Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and batch analyzing the solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

5.3 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. The solvent rinse should be followed by detergent wash with hot water and rinsed with tap water. Drain the glassware and dry overnight. Store dry glassware in a clean environment. Prior to use, rinse the glassware in methanol and methylene chloride respectively.

5.4. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting PCB Aroclors. Sulfur contamination may be expected with sediment samples. Method 3660 is suggested for removal of sulfur.

5.5. Waxes, lipids, and other high molecular weight materials may interfere with the analysis of PCB Aroclors and can be removed by Gel-Permeation Clean-up (GPC) (Method 3640). Co-eluting chlorophenols can be removed by florisil clean-up (Method 3620).

6. Equipment and Apparatus

6.1. Gas Chromatograph/Electron Capture Detector

There are 4 GC/ECD in the laboratory. Three are GC/µ-ECD are labeled as E4, E5 and E6. GC E2 is a Hewlett Packard (HP) Model 5890 Series II with electron Pressure Control. GC E4, E5 and E6 are HP Model 6890 Series II with u-Electron Pressure Control. All GC's are temperature programmable instruments with single injection port and dual electron capture detectors, and are equipped with HP Model 7673A Auto-sampler. A single gooseneck splitless injection liner is used in the injection port and connected to a piece (up to a 5m long) of uncoated megabore column (0.53mm id) which serves as a guard column. A

guard column is connected to the tee split. Each of two columns is connected to the split tee also. HP 3365 Chemstation software is used in conjunction with EnviroQuant software to handle data acquisition and processing. All GC's are interfaced to the network workstation. LIMS software is used for reports.

6.1.1 HP Vectra PC.

6.1.2 HP 3365, EnviroQuant and Target software.

6.1.3 HP Model 7673A auto-sampler.

6.1.4 Chromatographic columns used in the laboratory:

RTX-CLPesticides (Restek) 30 m x 0.53 mm id (0.5 um film thickness) and RTX-CLPesticides II (Restek) 30 m x 0.53 mm id (0.42 um film thickness).

6.1.5 Gooseneck splitless injection liner, Cat #20799 from Restek or equivalent

6.1.6 Uncoated megabore guard column (0.53 mm id, up to 5 m long). Cat # 10028 from Restek or equivalent.

6.1.7 Universal "Y" Press-tight tee split Cat # 20406 from Restek or equivalent.

6.2 Glassware:

6.2.1 Class "A" volumetric flasks:
10 ml, 25 ml, 50 ml, 100 ml, and 250 ml.

6.2.2 Syringes:
10 µl, 25 µl, 50 µl, 100 µl, 500 µl, 1 ml, 2.5 ml (accuracy to ± 1% per vendor's specification).

7. Reagents and Standards.

7.1. n-Hexane: pesticide residue grade or better.

7.2. The standards used in the method are discussed below. Please note that standards from other vendors could be used as long as the standards are of high purity (>96%) and traceable to reference materials. The laboratory will at all time archive or have on order one complete set of un-opened ampulated standards.

7.3. The list of primary (ampulated) calibration standards are obtained from Restek:

- Aroclor 1016 and 1260 (Cat. # 32039) at 1000ug/mL.

- Aroclor 1221 (Cat. # 32007) at 1000ug/mL.
- Aroclor 1232 (Cat. # 32008) at 1000ug/mL.
- Aroclor 1242 (Cat. # 32009) at 1000ug/mL.
- Aroclor 1248 (Cat. # 32010) at 1000ug/mL.
- Aroclor 1254 (Cat. # 32011) at 1000ug/mL.
- Aroclor 1262 (Cat. # 32409) at 1000ug/mL.
- Aroclor 1268 (Cat. # 32410) at 1000ug/mL.
- Tetrachloro-m-xylene (TCX) (Cat.# 32027) at 200 ug/mL.
- Decachlorobiphenyl (DCB), (Cat.# 32029) at 200 ug/mL

7.4. The list of second source standards are obtained from Ultra:

- Aroclor 1016 (Cat. #PP282) at 1000ug/mL.
- Aroclor 1260 (Cat. #PP361) at 1000ug/mL.
- Aroclor 1262 (Cat. #PP372) at 1000ug/mL.
- Aroclor 1268 (Cat. #EPA 1382) at 1000ug/mL.

7.5. All of the above primary standards are logged into the LIMS Standard Logbook on receipt and labeled as **PP*yymmddX***

Where:

PP = Pesticides/PCB primary standard
yymmdd = date the standard is received
X = the order the standard is logged into the logbook on that date, in alphabetical order.

The expiration date for ampulated standards shall not exceed the manufacturer's expiration date. All primary standards are stored according to manufacturer's recommendation. All vials containing primary standards must be labeled according to the current version of SOP No. 80.0001 Standard Preparation, Equivalency and Traceability.

7.6. Intermediate Standards

All intermediate standards are labeled **PI*yymmddX***

where:

PI = Pesticide Intermediate Standard.
yymmdd = date the intermediate standard is prepared.
X = the order that the intermediate standard is prepared on that date in alphabetical order.

Smaller or larger volumes may be used and the final volume adjusted to keep the concentration the same.

All intermediate standards are stored in amber glass bottles under refrigeration at 4°C or below. All standards are stored separately from samples and sample extracts

All intermediate standard solutions must be prepared every six months, or sooner, if the solution has degraded or concentrated.

All of the appropriate standard preparations are recorded in the LIMS Standard Logbook.

Analyst must allow all intermediate standard solutions to equilibrate to room temperature before use.

7.6.1. Intermediate Pesticide/PCB Surrogate Mix:

The intermediate stock standard is made by diluting 0.5mL of stock TCX and 1.0 mL of stock DCB and diluting to 50 mL with hexane. Intermediate Pesticide/PCB Surrogate Mix contains TCX at 2 ug/mL and DCB at 4ug/mL.

7.6.2. Intermediate AR1660 (AR1016+AR1260) Stock Solution:

The two Aroclors –Aroclor1016 and Aroclor 1260 have different chromatographic patterns and can be combined for analysis. When the two Aroclors are combined, they are labeled as Aroclor 1660.

The mixture of the two Aroclors is prepared by diluting 1mL of primary Aroclor standard at 1000ug/mL (AR 1016 + AR1260) Mix into 100mL of hexane.

Concentration of the intermediate AR1660 standard solution is10ug/mL

7.6.3. Additional Aroclor Intermediate Stock Solutions:

Intermediate stock solutions of each of the Aroclors are prepared by diluting 1mL of the primary standards (at 1000ug/mL concentration) to 50 or 100mL using hexane. There will be one intermediate solution for each of the Aroclors listed above, each at a concentration listed below.

- Aroclor 1221 at 20µg/mL.
- Aroclor 1232 at 20µg/mL.
- Aroclor 1242 at 10µg/mL.
- Aroclor 1248 at 10µg/mL.
- Aroclor 1254 at 10µg/mL.
- Aroclor 1262 at 20µg/mL.
- Aroclor 1268 at 20µg/mL

Please note that the following preparation procedures pertain to the use of the primary stock standards listed in **Sections 7.3** and **7.4**. Different preparation schemes are needed if different stocks are used.

7.7. Working Standards

Working standards are labeled as PW*yymmddX*

where:

PW = Pesticides/PCB working standard
yymmdd = date the standard is prepared
X = the order the standard is prepared on that date, in alphabetical order

All working standards are stored in amber containers under refrigeration at 4°C or below. The standards are stored in separate location from the samples and/or extracts to minimize cross contamination.

The expiration date for the standards is six months from the date of preparation or whenever the primary standard expires, whichever comes first.

The working 6 levels calibration standards for each of the individual Aroclors is prepared as follows:

Level 5: 8mL of the intermediate stock Aroclor prepared **in Section 7.6.2** and 2mL of the intermediate surrogate standard prepared in **Section 7.6.1**.are diluted to 50mL using hexane.

Note: The preparation volumes could be adjusted to make smaller or larger volume of standards as long as the final working concentrations are the same.

Level 4: 4mL of the intermediate stock Aroclor and 1mL of the intermediate surrogate standard are diluted to 50mL using hexane.

Level 3: 8mL of the intermediate stock Aroclor and 2mL of the intermediate surrogate standard are diluted to 200mL using hexane.

Level 2: 1mL of the intermediate stock Aroclor and 0.25mL of the intermediate surrogate standard are diluted to 50mL using hexane.

Level 1: 0.5mL of the intermediate stock Aroclor and 0.125mL of the intermediate surrogate standard are diluted to 50mL using hexane.

Level 6: 0.25mL of the intermediate stock Aroclor and 62.5ul of the intermediate surrogate standard are diluted to 50mL using hexane.

7.7.1. The 6 levels of Aroclor 1660 standards contain the following (concentration in ug/mL):

	Aroclor 1016	Aroclor 1260	TCMX	DCB
Level 1	0.1	0.1	0.005	0.01
Level 2	0.2	0.2	0.01	0.02
Level 3	0.4	0.4	0.02	0.04
Level 4	0.8	0.8	0.04	0.08
Level 5	1.6	1.6	0.08	0.16
Level 6	0.05	0.05	0.0025	0.005

7.7.2. The working 6 level of each of Aroclors contain the following (concentration in ug/ml):

	Aroclor	TCMX	DCB
Level 1	0.1	0.005	0.01
Level 2	0.2	0.01	0.02
Level 3	0.4	0.02	0.04
Level 4	0.8	0.04	0.08
Level 5	1.6	0.08	0.16
Level 6	0.05	0.0025	0.005

7.7.3. Second source standards:

Aroclors 1016 and 1260 are prepared at 0.4 ug/mL as a mixed standard. 0.2mL of each primary standard is diluted to 50mL with hexane.

Please note that surrogate standards may or may not be added to the second source standards.

8. Procedure

8.1. Preparation – The methods in USEPA SW-846 for sample extraction are as follows:

Method 3510 (SOP# 50.0051) extracts aqueous samples for water-insoluble and slight water-soluble organics. The samples are serially extracted with methylene chloride using a separatory funnel.

Method 3520 (SOP# 50.0050) extracts aqueous samples for water-insoluble and slightly water-soluble organics. The samples are placed in continuous liquid-liquid extractor and extracted with methylene chloride for 18 hours.

Method 3540 (SOP# 50.0053) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed

with anhydrous sodium sulfate to form a free-flowing mixture, and then extracted using 1:1 (v/v) methylene chloride/acetone in a soxhlet extraction.

Method 3550 (SOP# 50.0052) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate to form a free-flowing mixture, and then extracted using ultrasonic extraction in 1:1 (v/v) methylene chloride/acetone.

Method 3570 (SOP# 50.0100) is the extraction of soil, sediment, tissues, biota and any sample considered solid. A 2-20gram sample is solvent extracted first with acetone, and then with hexane by either a manual shake approach or via rotation or spinning of the sample.

Method 3545 (SOP# 50.0101) is the extraction of soil or sediment. A 15gram sample is mixed with diatomaceous earth to form a free-flowing mixture. This is added to the sample extraction cell. The cell is loaded on the PFE extractor to perform the extraction. The resulting extract is then filtered through anhydrous sodium sulfate, and concentrated to the appropriate final volume.

In addition, clean-up procedures are employed to remove co-eluting interferences. The lab uses GPC clean-up (Method 3640) and sulfur clean-up (Method 3660).

8.2. Instrument conditions

The GC operating conditions are as follows:

Carrier Gas	Helium (99.999%)
Column Flow	5-10 mL/min., independent of temperature
Make-up Gas	5% methane/95% argon (P-5)
Make-up Gas Flow	80-100 mL/min.
Injector	Split-less
Injector Temperature	200°C
Initial GC Temperature	120-170°C
Initial GC Hold	3 min.
Temperature Ramp	6-8°C/min
Final Temperature	265-300°C
Final GC Hold	2-15 min.
Detector Temperature	300°C

In the event that these conditions are changed, Enviroquant Data Acquisition methods containing the actual GC operating conditions are copied and sent to the network along with all GC/ECD raw data files. They are located in a folder in the sequence batch called “Zacq”.

8.2.1. Optimize GC operating conditions for analytes separation and sensitivity. Once optimized the same conditions must be used for analysis of all standards, samples, blanks and MS/, MSD.

The auto-sampler makes 2µl injection for the analysis of all standards and sample extracts.

8.2.2. Preventative Maintenance.

The injector septum is replaced every time the instrument is set up to perform a sequence of analyses when a leak develops, or when initial and/or continuing calibrations fail to meet the method requirements.

The injection liner is replaced every time the instrument is set up to perform a sequence of analyses, when a leak develops or when initial and/or continuing calibrations fail to meet the method requirements. The gold seal will be replaced and the columns will be trimmed every time before a new calibration is run.

The column will be replaced if standard chromatograms show excessive peak tailing or initial and/or continuing calibrations repeatedly fail to meet the method requirements.

8.2.3. Major instrument maintenance must be documented in the LIMS maintenance logbook, regular (daily) maintenance can be documented in Instrument Run Logbook. Also refer to SOP 110.0040 Instrument Maintenance and Documentation for additional information.

8.2.4. Initial calibration is performed by analyzing 6 level standards of the Aroclors listed below, and at least one level (midpoint) of Aroclors 1221, 1232, 1262 and 1268. If detected in samples, a full calibration would be run:

- Aroclor 1242
- Aroclor 1248
- Aroclor 1254
- Aroclor 1660

All analytical runs need to be documented in the appropriate Instrument Run Log. In addition to listing the data file, the associated working standard ID and standard name should be noted.

The chromatograms for each of the target Aroclor standards are presented in **Attachment 3** for both the RTXCLPest and RTXCLPest II columns.

8.2.5. From the initial calibration, the following parameters are determined:

8.2.5.1. Retention time and retention time window:

It is our experience that the GC retention times operating under EPC conditions are very tight. Using the 72 hour approach to determine retention time windows at times had resulted in extremely narrow retention time windows. This may result in false negatives due to slight retention time shifts, especially when sample extracts are subjected to co-eluting interferences. Therefore, the procedures for establishing analyte RT and their windows have been taken from the USEPA SOM Statement of Work.

The retention time window is established by evaluating the retention time of the Aroclor 1260 standards (at 1ug/mL) that were analyzed in the initial calibration. Please note that the laboratory does not need to perform retention time window studies for the other Aroclors.

From the standard analysis, three peaks are selected for each of Aroclor 1016 and Aroclor 1260. The retention times for the six peaks are measured and the mean RT is calculated as the average value for each of these peaks. For Aroclors 1221, 1232, 1242, 1248, 1254, 1262, and 1268 a RT is measured for each of the peaks from a single-point calibration standard unless a full calibration study was performed. If a full calibration was done, the average RT is used as done for Aroclors 1016/1260. A RT is measured for the surrogates in each of the calibration standards and the mean RT is calculated as the average of the values.

An RT window is calculated for the major peaks (a minimum of 3) of each Aroclor and for each surrogate using the RT window listed below. Analytes are identified when peaks are observed in the RT window for the compound on both GC columns.

Compounds	Retention Time Windows (minutes)
Aroclors	±0.07
Tetrachloro-m-xylene	±0.05
Decachlorobiphenyl	±0.10

8.2.5.2. The lab chooses to use external quantitation for instrument calibration and sample quantitation. The following equation is used to calculate the calibration factor (CF):

$$CF = \frac{\text{Peak area (or height) of standard}}{\text{---------------------------------------}}$$

Mass injected (ng)

The above equation is used to determine the calibration factor for both the single component and multi-component standards. Please note that either peak area or peak height can be used for the calculation.

The lab chooses to use peak height for single component analytes such as the surrogate compounds and for the multi-component PCB.

In calibrating for the Aroclors, the laboratory selects three peaks that are characteristic of each of the Aroclors. The analyst will choose peaks in each Aroclor standards that are at least 25% of the height of the largest Aroclor peak. Also, it is preferable that one of the three peaks selected is unique to that Aroclor.

8.2.5.3. Linearity - linearity is used to evaluate the dynamic range of the calibration factor for each of the single component surrogate standards. Linearity, as measured by the % Relative Standard Deviation (RSD) is calculated using:

$$\% \text{ RSD} = \frac{SD_{CF}}{CF_{av}} \times 100$$

Where: $SD_{CF} = \sqrt{\sum (CF_i - CF_{av})^2 / (n-1)}$

CF_{av} = average calibration factor

CF_i = calibration factor

n = total number of values = 5

Please note that there are three sets of calibration factors for each of the Aroclors.

8.2.5.4. %RSD Acceptance criteria for linearity:

The acceptance limit for linearity check is that the % RSD for the individual Aroclor calibration factors must be less than or equal to 20%. If this condition is met, the instrument is determined to be linear within the calibration range.

Given the large number of individual compound calibration factors, it is likely that some Aroclors may exceed the 20% acceptance limit. For Aroclors that exceeded the 20% limit, the linearity check is still acceptable if the following least square regression conditions are met:

8.2.5.5 Alternate Least Square Regression criteria for Linearity:

The analyst may employ a regression equation for the Aroclor(s) that do(es) not pass using the earlier approach. The regression will produce the slope and intercept terms for a linear equation in the following form:

$$y = mx + b$$

Where y = instrument response (peak area or height)
m = slope of the line
x = concentration of the calibration standard
b = intercept

It is important that the origin (0, 0) is not included as a calibration point and that the above equation is not forced through the origin. The linear regression is deemed acceptable if the correlation coefficient $r \geq 0.995$ ($r^2 \geq 0.990$)

8.2.6. Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. The calibration is performed by analyzing standard Aroclor 1660 standard prepared in **Section 8.1.8**. The acceptance criteria are as follows:

The calculated value of the analyte in the ICV should be 80 **–** 120% of the expected value. If the ICV does not meet the criteria, see corrective action guidelines in **Attachment 1** of this SOP. Results of this evaluation should be documented in the Instrument run log.

If the above criteria are not met, the analyst must evaluate the integrity of the primary and confirmation standards. If needed, re-preparation and re-analysis of the initial calibration is required.

8.2.7. Initial calibration acceptance criteria must be met before any sample, blanks or LCS is to be analyzed.

8.2.8. Corrective Action for Initial Calibration – see **Attachment 1** for corrective action guidelines and documentation.

8.2.9. Original initial calibration raw data must be archived in the company organics analysis calibration (OCAL) database. See Mitkem SOP # 10.0009 (Application Xtender) for scanned archive information.

8.3. Continuing Calibration Verification and Sample Analysis – Method 8082 requires the analysis of Aroclors 1016/1260 as the continuing calibration verification (CCV) every time samples are to be analyzed to ensure that the

GC/ECD system continues to meet instrument sensitivity and linearity requirements. Mitkem may also analyze Aroclors 1242, 1248 and 1254 as part of the calibration verification

8.3.1. Frequency of Continuing Calibration (CCV) - The CCV must be performed if the instrument has been idle for more than 12 hours. A continuing calibration standard must also be injected at intervals of not less than once every twenty field samples (the method recommends the frequency to be once every ten samples, to minimize reanalysis due to unacceptable CCVs) and at the end of the analysis sequence.

DoD requires CCV standards must be injected at intervals of not less than once every ten field samples.

The continuing calibration is performed using the Level 3 (0.4 ug/ml) Aroclor 1660 standard (as well as Aroclors 1242, 1248 and 1254 where appropriate). While the method suggests alternating the use of high and low calibration mixtures, the lab performs the continuing calibration using the mid-point standard. When the additional Aroclors 1242, 1248 and 1254 are analyzed, they will also be evaluated using the same approach as Aroclors 1016/1260.

8.3.2. Evaluation of the Calibration Verification:

8.3.2.1. *% Difference:* Calculate the % difference between the continuing calibration CF and those from the most recent initial calibration.

The % difference is determined as follow:

$$\% \text{ Difference} = \frac{CF_c - CF_i}{CF_i} \times 100$$

In which:

CF_c = calibration factor from continuing calibration
CF_i = mean calibration factor from the most recent initial calibration

8.3.2.2. *% Drift:* Use % drift when using linear regression calibration.

$$\% \text{ Drift} = \frac{Conc_c - Conc_t}{Conc_t} \times 100$$

In which: $Conc_c$ = concentration obtained from continuing calibration
$Conc_t$ = theoretical concentration of standard

8.3.2.3. *% Difference* (*or % Drift*) between the continuing calibration factor and that of the initial calibration should be no greater than ± 20% at least on one column. If this criterion is met then the calibration has been verified and sample analysis can proceed. The analyst should mark whether the CCV passed on one or both columns in the instrument logbook. Checkmarks or the term "OK" are acceptable entries.

8.3.2.4. Comparison of the retention time window: the retention time of each Aroclor and surrogate peak in the calibration standards should fall within the retention time window established from the initial calibration. Due to the need to perform routine column maintenance (clipping off a small length of the guard and/or analytical columns), the retention time might be shortened. The retention time windows can then be updated using the first continuing calibration verification's absolute RTs to set the midpoint of the window, or a new curve must be analyzed. All subsequent closing and opening CCV analyses must fall within the new retention time windows to be valid.

8.3.3. Corrective Action for opening CCV- The %D (or % Drift) between the individual compound calibration verification factors and those from the initial calibration should be no greater than ±20% on at least one column. If this criterion is exceeded for any peak, the calibration has not been verified.

If the continuing calibration does not meet the criteria, a second injection of the calibration verification is allowed. If the second injection fails, the analytical sequence is stopped. Corrective actions must be performed. This may include preparing new standards, performing inlet maintenance and/or GC column maintenance. If the response of the analyte is still not within the above criteria, then a new initial calibration must be performed.

8.3.4. Corrective Action for closing CCV: When a calibration verification standard fails to meet the QC criteria on both columns, all samples that were injected after the last compliant standard must be evaluated to ensure the data is valid. The analyte(s) which fail(s) the 20%D must be addressed if the analyte affected is one that was detected in project sample(s). The analyst needs to note all QC issues in the instrument run log and on all associated project data review checklists. This information needs to be addressed in the associated project(s) narrative. See **Attachment 1** for corrective action guidelines and documentation.

8.3.4.1. If the non-compliant standard analyzed **after** a group of samples exhibits an elevated response for an analyte that is greater than 20% D (**positive bias**), and the analyte **was not** detected in the

group of samples analyzed between the compliant and non-compliant bracketing standards, the group of samples is deemed acceptable and does not require re-analysis.

8.3.4.2. If the non-compliant standard analyzed **after** a group of samples exhibits an elevated response for an analyte that is greater than 20% D (**positive bias**) on both columns, and the analyte **was** detected in the group of samples analyzed between the compliant and non-compliant bracketing standards, then re-injection of all samples are required to ensure accurate quantitation.

8.3.4.3. If the non-compliant standard analyzed **after** a group of samples exhibits an elevated response for an analyte that is greater than 20% D (**positive bias**) on one column, and the analyte **was** detected in the group of samples analyzed between the compliant and non-compliant bracketing standards, and the associated samples are detects, the reported values must come from the compliant column, even if this is the higher result.

8.3.4.4. If the non-compliant standard analyzed **after** a group of samples exhibits a lower response for an analyte that is greater than 20% D (**negative bias**), then re-injection of all samples are required to ensure accurate quantitation.

Sample injections may continue as long as the calibration verification standards meet the instrument QC requirements.

All standard analyses are to be documented in the appropriate Instrument Run Log with associated working standard IDs.

8.3.5. The determination of PCB Aroclors is accomplished by comparing the sample chromatogram to that of the most similar Aroclor standard. The use of PCB overlays is extremely helpful, either by using hardcopies of chromatograms or by utilizing the Target software. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample. Both retention time and pattern are important when determining PCBs in a sample.

It is important that if Aroclors are detected in the samples, the analysis of these sample extracts should be accompanied by the appropriate Aroclor calibration verification standard as part of the analytical sequence. If this is not done, the sample extracts have to be re-injected with the appropriate Aroclor standards to ensure the PCB pattern is similar.

Samples that contained weathered PCB present special analytical challenges. Weathering could alter the Aroclor pattern to the extent that different peaks have to be selected for quantitation. Samples that contained more than one Aroclor present similar problems. For these samples, the analyst may have to consider selecting the earlier eluting peaks for the lower boiling Aroclor and selecting the later eluting peaks for the higher boiling Aroclors to minimize overlapping peaks. In these instances, the analyst may need request the assistance of someone with more expertise in determining the presence of PCB Aroclors.

8.3.6. The concentration of the target compounds and surrogates are calculated separately for each of the three Aroclor peaks for both columns using the following equation.

$$\text{For aqueous samples, concentration } ug/L = \frac{(Ax)\,(Vt)\,(DF)}{(CF)\,(Vo)\,(Vi)}$$

$$\text{For soil samples, concentration } ug/kg = \frac{(Ax)\,(Vt)\,(DF)}{(CF)\,(Ws)\,(Vi)\,(S)}$$

in which: Ax = area/height of the peak for the compound to be determined

CF = calibration factor
Vo = volume of water extracted, in mL
Ws = weight of soil sample, in gram
Vi = volume of extract injected, in µL
Vt = volume of extract, in µL
DF = dilution factor
S = solid content, expressed in decimal

For the multi-component Aroclors, the analyte concentration is calculated by taking the average of the concentration determined for each of the three peaks selected for that analyte.

The sample concentration is determined for both columns. The lower of the two values is reported unless special circumstances exist such as co-elution or state/government programs require otherwise.

8.3.6.1. In instances where the %D discussed in **section 8.3.4** passes for only one of the two columns, the analytical value is reported from the passing column.

8.3.6.2. The difference between the two determined values is calculated using the equation listed below. The analyst should evaluate the chromatogram to see if the discrepancy might be due to co-eluting interferences. If the difference is greater than 40% between the columns, the analytical values will be flagged with a "P" qualifier on the sample data report form (Form I) to notify the data user that one of the concentrations might be biased due to co-eluting interferences. Results from both GC columns are reported on the Form X.

$$\%D = \frac{C_H - C_L}{C_L} \times 100\%$$

Where C_H = Higher Concentration
C_L= Lower Concentration

Please note that this formula is taken from the determinative EPA SOM methods and not SW 846 Method 8000. Method 8000 suggests using the average of the two concentrations as the denominator. The above calculation provides a more conservative approach to the RPD calculation. An additional data report sheet is available through LIMS comparing the two GC column results using the SW-846 calculation if needed.

8.3.7. Manual integrations are performed, reviewed and documented per SOP No. 110.0008 Manual Integration of IC, GC and GC/MS Chromatograms.

8.3.8. The concentrations of the surrogate analytes are calculated and reported separately for each of the two columns. The acceptance limits for the recoveries are discussed in **Section 10**.

8.3.9. The analyte recoveries of the LCS and matrix spike compounds are determined and reported. The acceptance limits for the recoveries are discussed in **Section 10**.

8.4. Analytical sequences are summarized as follows:

Order	Sequence	Comment
1	AR1232L3	
2	AR1221L3	
3	AR1242L1	Level 3 only for AR1232, AR1221, 1262 and 1268 are run unless this Aroclor is detected in samples.
4	AR1242L6	
5	AR1242L2	
6	AR1242L3	
7	AR1242L4	
8	AR1242L5	
9	AR1248L1	
10	AR1248L6	
11	AR1248L2	
12	AR1248L3	
13	AR1248L4	
14	AR1248L5	
15	AR1254L1	
16	AR1254L6	
17	AR1254L2	
18	AR1254L3	
19	AR1254L4	
20	AR1254L5	
21	AR1660L1	
22	AR1660L6	
23	AR1660L2	
24	AR1660L3	
25	AR1660L4	
26	AR1660L5	
27	AR1262L3	
28	AR1268L3	
29	AR1660SS	Second source ICV
30	AIBLK01	Instrument blank
31	Blank	
32	LCS	
33	Sample extracts	
34	AIBLK02	Instrument blank calibration verification
35	AR1242M01	
36	AR1248M01	
37	AR1254M01	
38	**AR1660M01**	
39	Sample extracts	
40	AIBLK03	Instrument blank calibration verification
41	AR1242M02	
42	AR1248M02	
43	AR1254M02	
44	**AR1660M02**	
45…	Sample extracts…	Sequence continues as the same as above as long as calibration verifications meet the acceptance

		criteria.

9. Data Reduction and Calculations

9.1. Sample data should be reported in units of µg/L for aqueous samples and µg/Kg dry weight for solid samples.

Results are reported to two significant figures using the USEPA guidelines in rounding up or down. For solid samples, results are reported in dry weight unless otherwise specified.

For 8082 analyses, PCB Aroclors are reported if the determined concentration is above the project reporting limits on both columns. No J flagged results are reported unless required by the project.

9.2. Soil concentrations are calculated using dry weight basis. To convert soil results to a dry weight basis, divide the sample concentration by the percent solids.

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100\%$$

where: DW = Sample weight (g) dried at 105°C overnight
WW = Sample weight (g) before drying

9.3. Recovery calculations - the recovery of a spiked analyte is calculated as follows:

$$\% \text{ Recovery (\%R)} = 100\% \times (SSR-SR)/(SA)$$

where: SSR = spiked sample result
SR = sample concentration
SA = spike added

9.4. Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

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

where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of the instrumentation and have performed a proficiency test with acceptable accuracy and precision results.

10.2 Method blanks. A method blank is an aliquot of a clean reference matrix (reagent water for water samples, or Sodium Sulfate for soil/sediment samples) that is carried through the entire analytical procedure. A Method blank is prepared and analyzed with every batch of 20 samples or less. It is used to determine the level of contamination associated with the analytical processing and analysis of the samples. The Method blank MUST be analyzed on the same instrument as the associated samples.

See **Attachment 1** for additional corrective action.

10.2.1 The recovery of the surrogates must be within the calculated acceptance limits discussed in **Section 10.6**.

10.2.2 The concentration of target compounds in the method blank must be ≤ to ½ CRDL or RL.

10.2.3 Corrective action for method blank contamination involves determining the source of the contamination and possibly re-extracting the entire batch.

10.3 Lab Control Sample (LCS) – A Lab Control Sample is a volume or weight of a clean reference matrix (organic-free water or Sodium Sulfate) that is spiked with Aroclors 1016 and 1260 and surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1 The recovery of the surrogates must be within the calculated acceptance limits discussed in **Section 10.6**.

The LCS recovery is evaluated against the established in-house limits. Any analyte/analytes not meeting the criteria requires corrective action. In-house limits may be lab-generated or from a government /method based source such as DoD QSM. Please note that the in-house limit will be verified on an annual basis. Where lab-generated limits are derived, these will be updated annually.

10.3.2 Advisory RPD limits are set at 30% when duplicate LCS are performed.

10.3.3 Any sample(s) that is/are associated with a non-compliant LCS may require re-extraction and re-analysis. See **Attachment 1** for corrective action guidelines.

10.4 Duplicate Matrix Spikes –Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix at a frequency of one set per 20 samples.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and one duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform a matrix spike and matrix spike duplicate.

10.4.1 Acceptance criteria for Duplicate Matrix Spike:

Matrix spike and matrix spike duplicate are used to assess the effect of matrix interferences on the analysis of the target analytes and the recovery should be used as advisory guidelines to answer question posed above.

10.4.2 Control limits for Method 8082 projects are the same as discussed in **Sections 10.3.2 and 10.3.3.** DoD requires 30% RPD for all matrices.

Depending on sample matrix, chromatographic based analysis such as Method 8082 is susceptible to co-eluting interferences resulted in high biased values. Thus, the control limits for the duplicate matrix spikes should be used as guidelines. The determination of outliers should be addressed in the project narrative to advise the data users of potential matrix related interferences.

10.5 Re-analysis at dilution - Any target compounds that are determined above the instrument calibration range will require reanalysis at dilution. A notation for the need to perform a dilution next to the sample run in the instrument logbook is needed to document the results. An entry such as “rerun at 4x” is acceptable. The dilution is performed by taking an aliquot of the extract and diluting it to a pre-determined volume using hexane. The analyst should not over-dilute the extract. The analytes that trigger the need for dilution should be determined in the diluted analysis at a concentration above that of the midpoint calibration standard.

10.5.1 When reporting diluted results the following guidelines should be followed. If an initial analysis is performed that meets all QC criteria with the exception of compounds exceeding the upper calibration limit, this analysis may be reported. The sample ID of the initial (less dilute) analysis is unchanged and the ID of the dilution analysis has the DL suffix appended to both the client and sample ID. Those compounds exceeding the calibration range are qualified with the “E” flag on the data sheet for the less dilute analysis, and all compounds detected in the more diluted (DL) analysis are qualified with the “D” flag.

10.5.2 If the laboratory has prior information that a sample may contain concentrations of target or non-target compounds exceeding the calibration range of the instrument, the initial analysis may be performed at dilution. If this analysis is acceptable (compounds at or above the mid-point calibration) then a less dilute analysis is not required. The sample ID is not changed by adding a DL suffix but the 'initial analysis at dilution' is noted on the data review checklist included with the data submitted for review, to allow discussion in the project narrative.

10.5.3 If the initial analysis fails QC criteria, it may be reported if specified by the project or client. If only the dilution is reported, the DL suffix is not added and the dilution is noted on the data review checklist submitted with the data for review as above.

10.6 Surrogate recoveries - The recovery of each surrogate compound in all samples, blanks, MS/MSD and LCS will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.6.1 Acceptance criteria for surrogate recovery.

For 8082 projects the percent recovery of each of the surrogate compounds in method blanks and LCS must be within the in-house acceptance criteria window. The current in-house acceptance criteria for each of the surrogate compounds in blanks, samples, duplicate matrix spikes and LCS are listed under the option SPEC for the LIMS Test code.

In-house limits may be lab generated or from a government /method based source such as DoD QSM. Please note that the in-house limit will be verified on an annual basis. Where lab generated limits are derived, these will be updated annually.

Surrogate spikes in matrix specific samples that fail to meet the in-house limits would indicate potential matrix effect. In general, high recovery of surrogates due to co-eluting interferences is acceptable. Low recovery of surrogates would indicate potential matrix related problems or one related to the extraction process. The analyst should confirm the sample volume extracted and the final extract volume to verify the calculations. If applicable, the analyst should consider additional cleanup procedures and re-analysis. If no appropriate cleanup procedure could be identified or re-analysis after additional cleanups failed to achieve the limit, this should be noted on the review checklist and also addressed in the narrative.

If surrogate recoveries do not meet recovery criteria see **Attachment 1** for corrective action

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10.7 MDL studies are conducted to establish the limit of detection applicable to this method. MDL verification at approximately 1-4 x MDL is analyzed after the study which also sets the DoD QSM Limit of Detection (LOD). MDL verification must be analyzed quarterly on each instrument used for DoD program work. Please refer to the SOP No. 80.0005 Determination of Method Detection Limits for more detail.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and samples results, is reviewed for technical accuracy and completeness by the analyst. The analyst initiates a project data review checklist and documents all comments regarding analysis there. Sample preparation logs, notebooks, and instrument logs are reviewed and signed by the laboratory supervisor. The QA Director randomly reviews 10% of the data reported by the laboratory.

11.2 All raw data is peer reviewed at the computer/Target level by another analyst or the lab supervisor prior to final form generation. Analysts generate all hard copy raw data and upload electronic data files to the LIMS for reporting. Raw Data, including all support data (such as data review checklist, run-logs, work-order sheets, SDG summaries…) are brought to the data reporting department for assembly (as hardcopy or electronic PCL files). After assembly, all data are reviewed by senior personnel (data reviewers) for quality control and completeness dependent on project specific requirements.

12. Data Management and Records Management

12.1 Electronic data generated from the analysis of PCB 8082 extracts (calibrations, QC, samples) is saved and managed per SOP 110.0029 Electronic Data Management.

12.2 All analysis information is documented in the individual Instrument Run/Injection Logbook regardless of run acceptance. No injections are deleted from the sequence.

13. Corrective Action Procedures

Corrective actions to be implemented in the event QC results are outside of the acceptance range are covered in **Sections 8, 9, and 10**. See **Attachment 1** for routine corrective action guidelines and documentation.

Discrepancy reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a discrepancy

report for the purpose of identifying the appropriate corrective action is covered in Corrective Action Procedures SOP No. 80.0007.

14. Health and Safety

The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is archived by the health and safety officer and available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Health and Safety Plan.

In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible. Spent vials and ampules are disposed of into a red metal drum in the Semivolatile Laboratory. See SOP No. 30.0024, Sample and Waste Disposal for more detail.

Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

15. Pollution Prevention; Waste Management; Definitions and Acronyms

See sections 19.0 (Waste Management) and 20.0 (Definitions, Acronyms, and Abbreviations) of the current Quality Assurance Plan.

16. References

1. U.S. Environmental Protection Agency. Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Method 8082A, SW-846 Test Methods for Evaluating Solid Wastes, Final Update IV, Feb 2007.
2. "Methods Compendium for Inorganic and Organic Methods," United States Army Corps of Engineers, Appendix I, 2001.
3. "Shell for Chemical Analytical Requirements," United States Army Corps of Engineers, Appendix H, 1997 including addendum dated 1 February 2001.
4. Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.1, April 2009 or current version.

Attachments:

1. **Table 1**: List of Target Analytes.
2. **Table 2**: List of Acronyms.
3. **Attachment 1**: Corrective Action Examples and Documentation Tables
4. **Attachment 2:** DoD QC Requirements.
5. **Attachment 3**: Aroclor Standard Chromatograms.

Table 1

List of Target Analytes for Method 8082

Aroclor	CAS Number
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

Table 2

. **List of Acronyms**

PCB	Polychlorinated Biphenyl
TCLP	Toxicity Characteristic of the Leaching Procedure
SPLP	Synthetic Precipitate Leaching Procedure
LCS	Lab control sample
MS	Matrix spike
MSD	Matrix spike duplicate
GC	Gas chromatograph
ECD	Electron capture detector
GPC	Gel Permeation chromatography
CF	Calibration factor
RSD	Relative standard deviation
RT	Retention Time
EPC	Electron pressure controller
USACE	US Army Corp. of Engineers
AFCEE	Air Force Center of Environmental Excellence
CLP	US EPA Contract Laboratory Program
DoD	Department of Defense

Attachment 1

Corrective Action Examples and Documentation Tables

OCCURRENCE	ACTION	DOCUMENTATION
1. Initial calibration does not meet QC criteria (RSD% >20% or r^2 < 0.990).	1. Check GC conditions such as temperature program, makeup gas flow rate, and carrier gas flow rate. Check if column bleeding occurs and more injection maintenance is needed. After the reasons are found, appropriate maintenance will be done and a new initial calibration will be run. If the new curve still fails, the reasons for failing the curve will be re-evaluated and another a new curve will be rerun after the reevaluation. If still not good, call GC manufacturer.	1. Note in instrument run logbook, and if necessary notation in instrument maintenance log book.
2. Initial calibration verification (ICV) check does not meet QC criteria (D% > ±20%).	2. Check the preparation of ICV standard, and if necessary, make a fresh ICV standard. If it is found that ICV standard is ok, the standards for initial calibration will be checked such as evaporation and the preparation of standards. A new ICV and a new curve will be analyzed after problems are corrected.	2. Note in instrument run logbook, and if necessary notation in instrument maintenance log book. If source determined to be bad standard solution, formal corrective action must be initiated.
3. Continuing calibration verification check does not meet QC criteria (D% > ±20%).	3. Maintenance such as replacing septum and liner for injection port will be done, and then a new set of CCVs and the samples related to the bad CCVs will be re-analyzed. If the CCVs still fail, A full maintenance including replacing septum, liner and gold seal and trimming the column will be done and a new initial calibration will be run. Then the CCV and/or QC and samples re-analyzed.	3. Note in instrument run logbook. If instrument maintenance performed, note in maintenance logbook.
4. Method blank contains target compound above reporting limit.	4. A new aliquot of the blank from prep lab will be reanalyzed to check if contaminants are carried over from previous dirty samples and/or from standards or a bad injection occurs. If the results from re-analysis of the blank are still the same, whole batch of the samples that are related to the blank will be re-extracted and re-	4. If the re-extraction/re-analysis meets the criteria, the results are reported. Document in the run log. If there is insufficient sample volume for re-extraction/re-analysis, report the initial results. Document in the run log, and include a comment on the package checklist for the data reviewer for inclusion in the narrative.

	analyzed.	5. Same as documentation #5.
5. Surrogates in the method blank are outside of acceptable range.	5. A new aliquot of the blank from prep lab will be reanalyzed. If the results from re-analysis are the same, the samples that are related to this blank will be re-extracted and re-analyzed.	6. Flag all compounds out of range on Form 3 of data report, if samples are re-analyzed within holding times, note in instrument run logbook. If samples are beyond holding time and both sets of data are to be reported, note in instrument run logbook, comment in data review checklist to be included in project narrative. If re-analysis cannot be performed due to insufficient sample, comment in data review checklist to be included in project narrative, and document as above.
6. Compound out of acceptance range in laboratory control sample.	6. Investigate source of problem. If LCS recovery is above upper QC limit, and if analyte is not detected in associated samples, data may be flagged and reported. If LCS is not acceptable per method/SOP/project requirements, this LCS will be reanalyzed. If the results of reanalysis are the same, the samples that are related to the LCS will be re-extracted and re-analyzed.	
7. Surrogates in samples are out of control limits.	7. 7.1 No actions are needed for the following situations: • One of the surrogates (TCX and DCB) is in control limits, and the other is above the upper limit due to coelution with contaminants. • TCX is in the control limits, and DCB is below the lower limit. This is possibly due to matrix effect. 7.2 Further actions are needed for the following cases: • Both of the surrogates (TCX and DCB) do not meet criteria. • TCX is lower than the lower limits and DCB is in the limits. This is most possibly due to evaporation of TCX in the extracts. Actions for 7.2: The samples will be reanalyzed. If the results of re-analysis are the same, the samples will be re-extracted	7. If only re-analysis is reported, note in instrument run log. If both sets of data are to be reported, note in instrument run log, re-extraction request form, preparation logbooks, and comment on data review checklist to be included in project narrative, flagging all non-compliant values on Form 2 of data report.

	and re-analyzed.	8. Note in instrument run log, comment on data review checklist to be included in project narrative.
8. Compound in sample exceeds upper calibration standard concentration.	8. Re-analyze sample at dilution. If calibration limit exceedence is the only QC problem, report both initial and dilution analyses. If initial analysis has multiple QC problems, evaluate further to determine if initial run is to be reported (often this cannot be determined until the results of the dilution are evaluated). Instrument must be shown to be free of carryover contamination prior to acceptable analysis of next sample. If running an instrument using auto-sampler, evaluate following sample. If following sample contains less than reporting limit of compound, the analysis is valid, and no instrument blank is required. If following sample(s) contain compound (typically in decreasing concentration—carryover typically occurs at 1% of concentration of high sample in following analysis, with effect more pronounced for later-eluting compounds. Effected samples must be re-analyzed if sufficient volume exists.	9. If only re-analysis is reported, note in instrument run log. If both sets of data are to be reported, note in instrument run log, re-extraction request form, preparation logbooks, and comment on data review checklist to be included in project narrative.
9. Instrument blank (GC) contains contamination above QC criteria.	9. Investigate source of problem, decontaminate purge and trap instrument, re-analyze blank and all effected samples.	10. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.
10. Matrix spike recovery out of QC range.	10. Evaluate problem. If duplicate spike (MSD) shows same effect, it is generally matrix interference. If concentration of spike analyte is significantly (approx. 4 times) greater in un-spiked sample, this is matrix interference masking quantitation of spike concentration. If source cannot be determined, re-analyze spike sample.	11. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion

11. Duplicate (or MSD) relative percent difference exceeds QC limit. 12. Retention Time shift.	11. Evaluate problem. If concentration of analyte is close to reporting limit, variation of analysis is acceptable. If sample is soil or other heterogeneous matrix, high RPD is typical. If sample is a typically homogeneous matrix, re-analyze duplicate sample. 12. A. No further action is required if the retention times of the CCV and samples shift after regular maintenance such as replacing the septum, liner, gold seal and trimming of the columns. The retention times will be established. B. If the retention times of the CCV shift during analysis, GC maintenance will be performed and the CCV and associated samples and QC will be re-analyzed. C. If the retention times of the CCV do not shift, but the retention times of surrogates in some of the samples do shift, the samples will be re-analyzed. If the retention times still do not meet the criteria, matrix affect is assumed and the results will be reported. This situation will be documented.	in report narrative. Flag RPD on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative. 12. Document in the run log. If CCV, QC, and samples were re-analyzed, document in the run log. If the retention times shift with respect to re-analysis, document in the run log, in the checklist for the case narrative.

Attachment 2
Department of Defense QC Requirements

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Breakdown check (Endrin / DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 15% for both DDT and Endrin.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 15% for both DDT and Endrin.

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL; HPLC methods: All project analytes within ± 15% of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL; HPLC methods: All project analytes within ± 15% of expected value from the ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (with the exception of Method 8015).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column (see Box D-16).
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Attachment 3
Aroclor Standard Chromatograms

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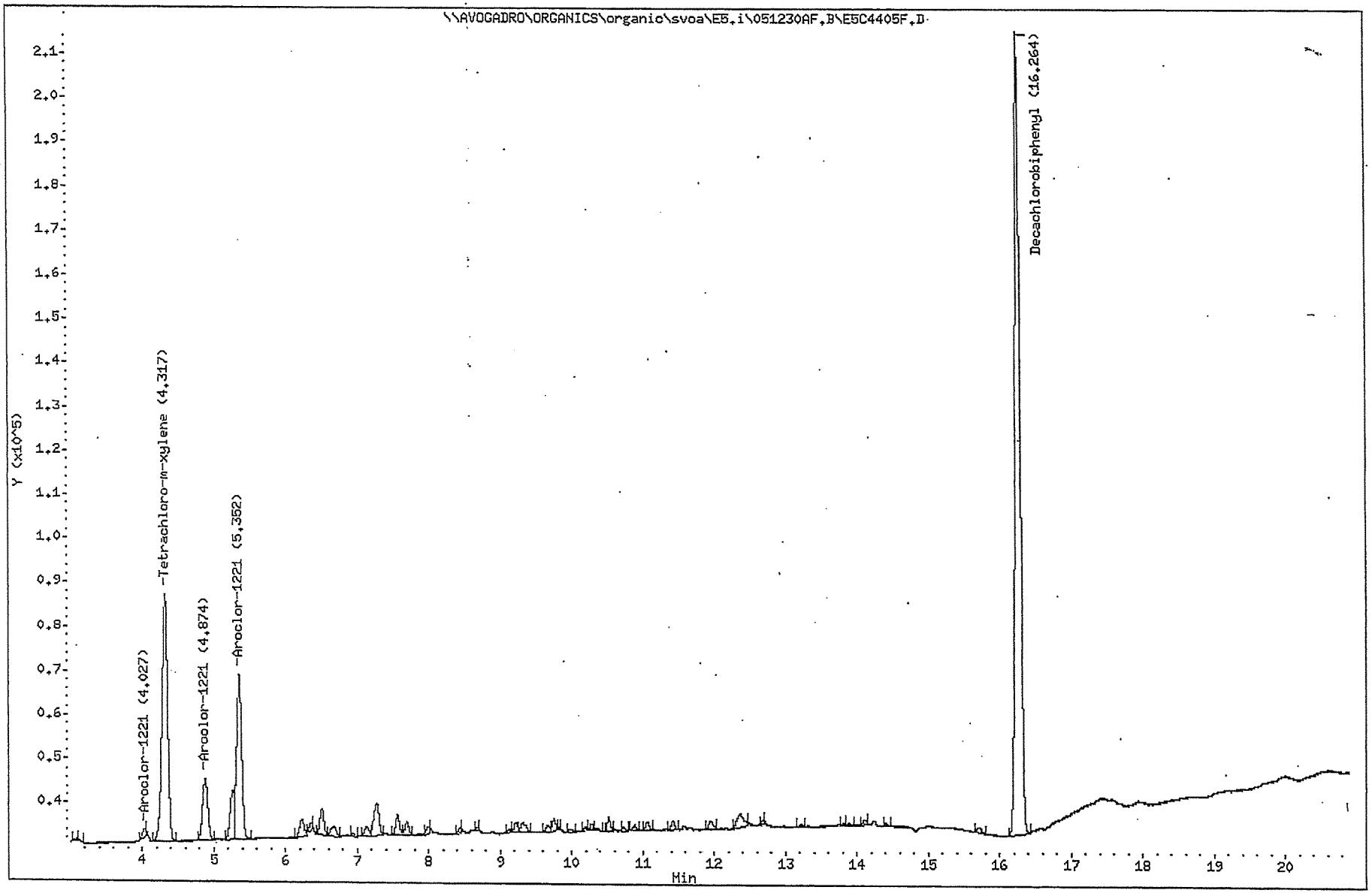
Column phase: RtxCLPPest

Instrument: E5.i
Operator: SRC:
Column diameter: 0.53



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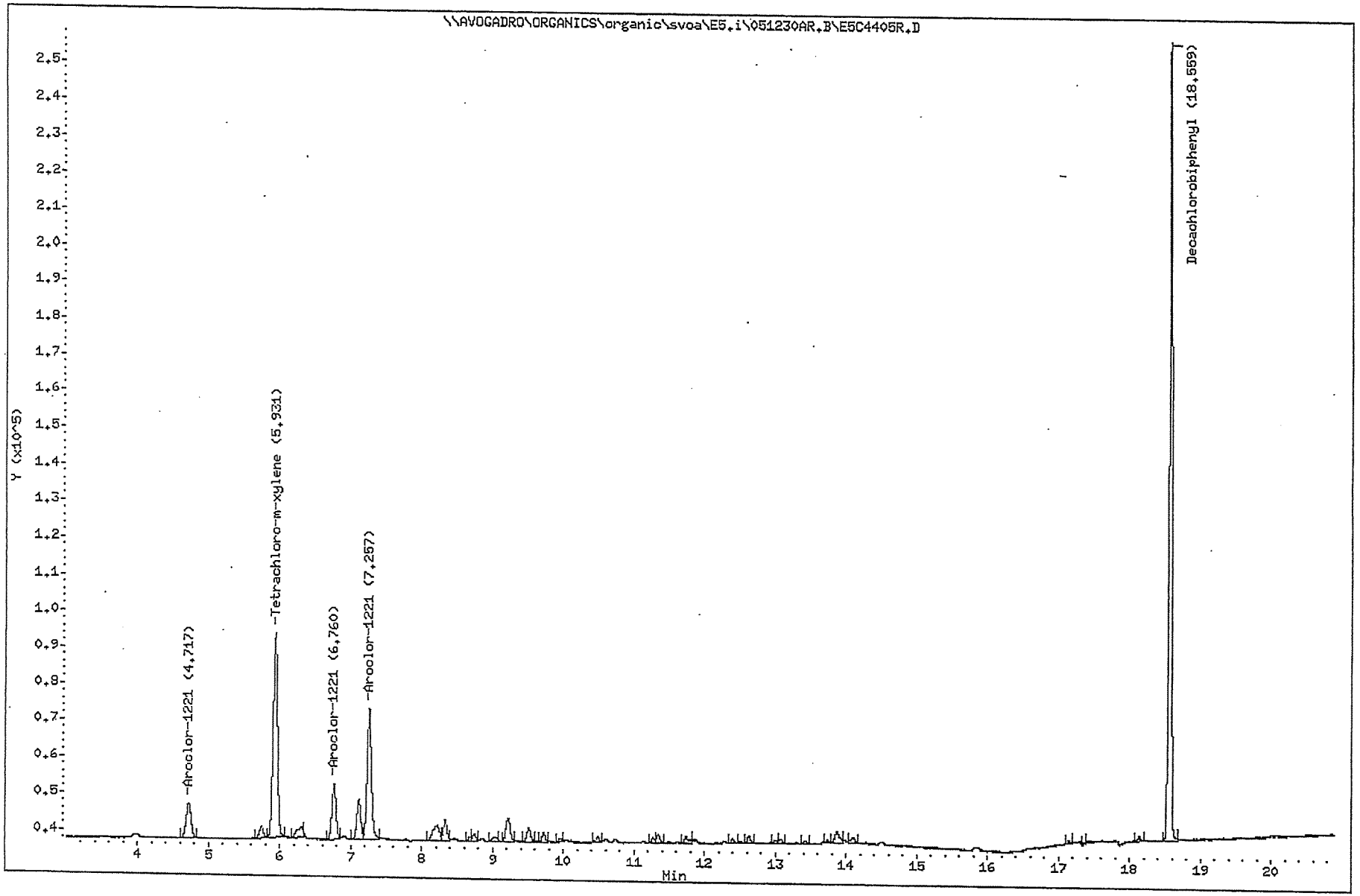
Instrument: E5.i

Operator: SRC:
Column diameter: 0.53



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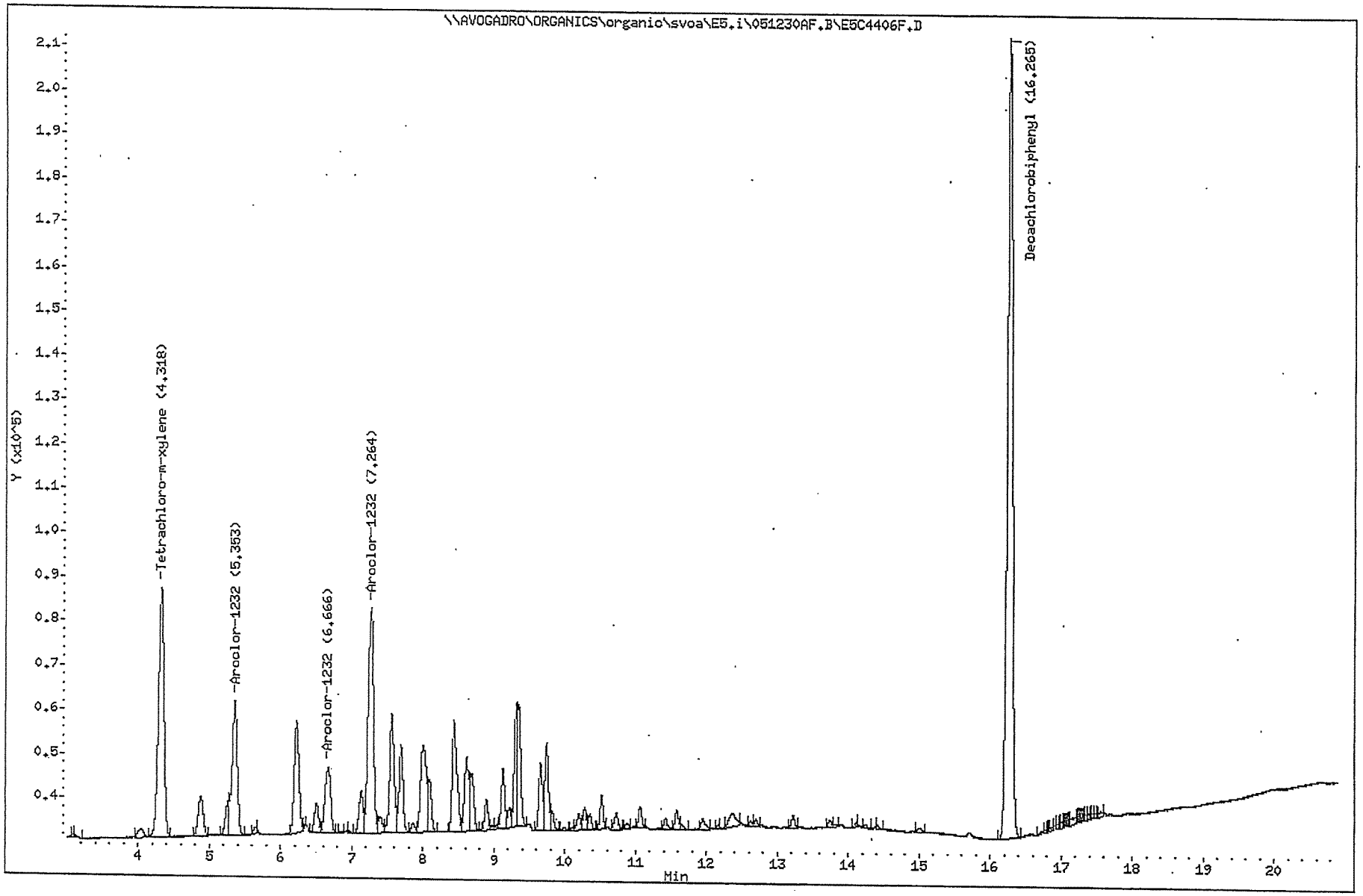

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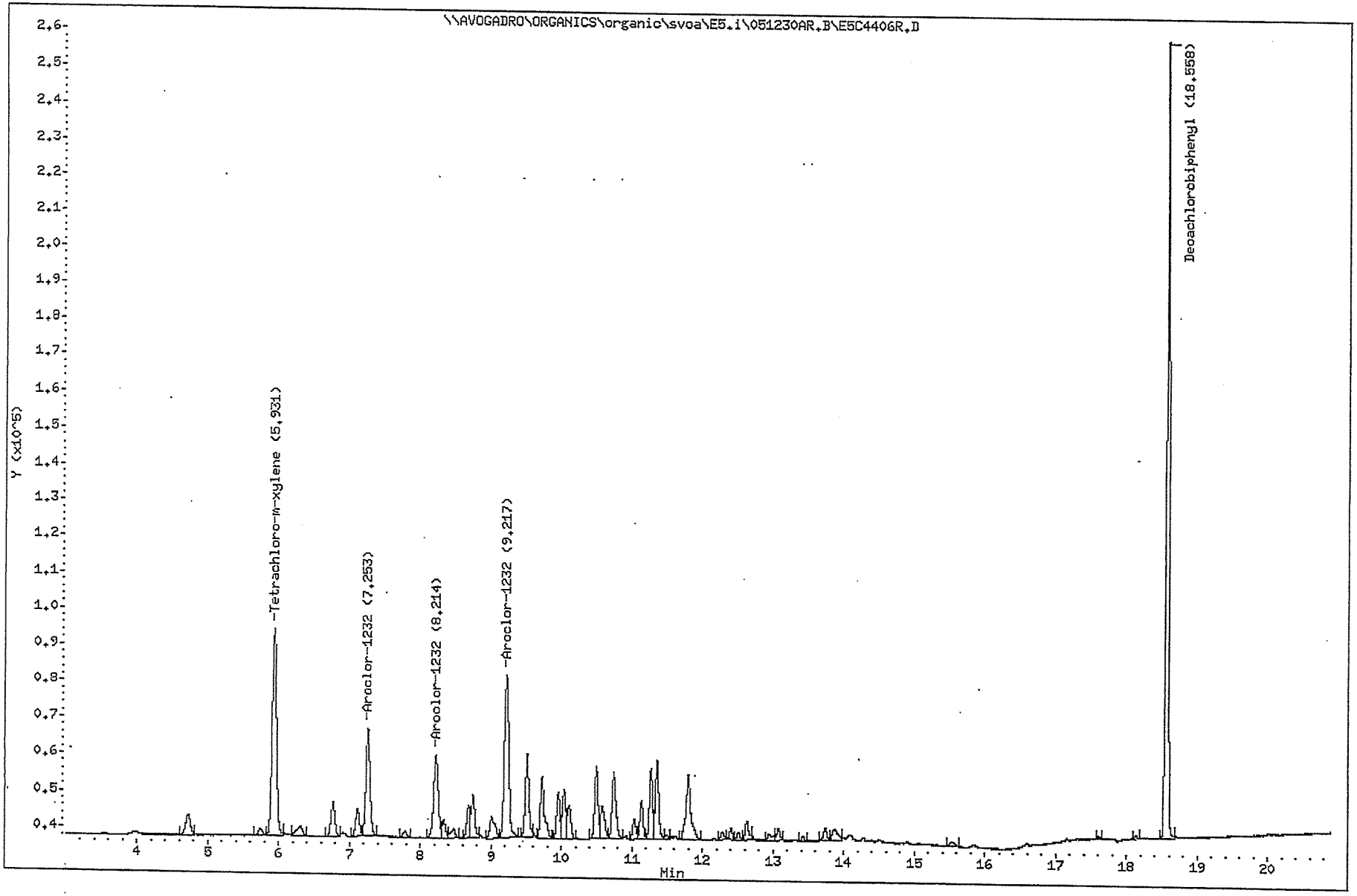
Instrument: E5.i
Operator: SRC:
Column diameter: 0.53



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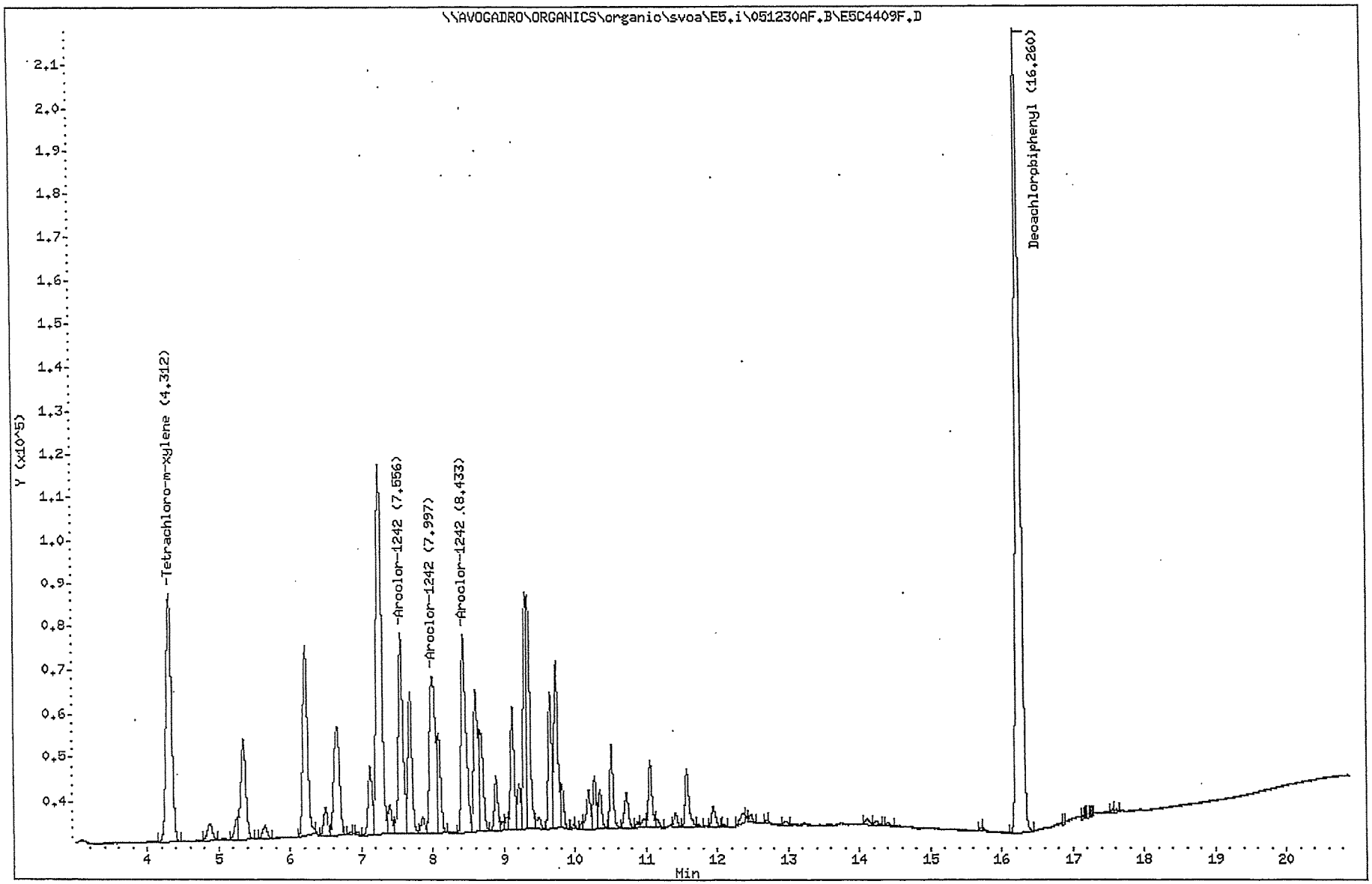

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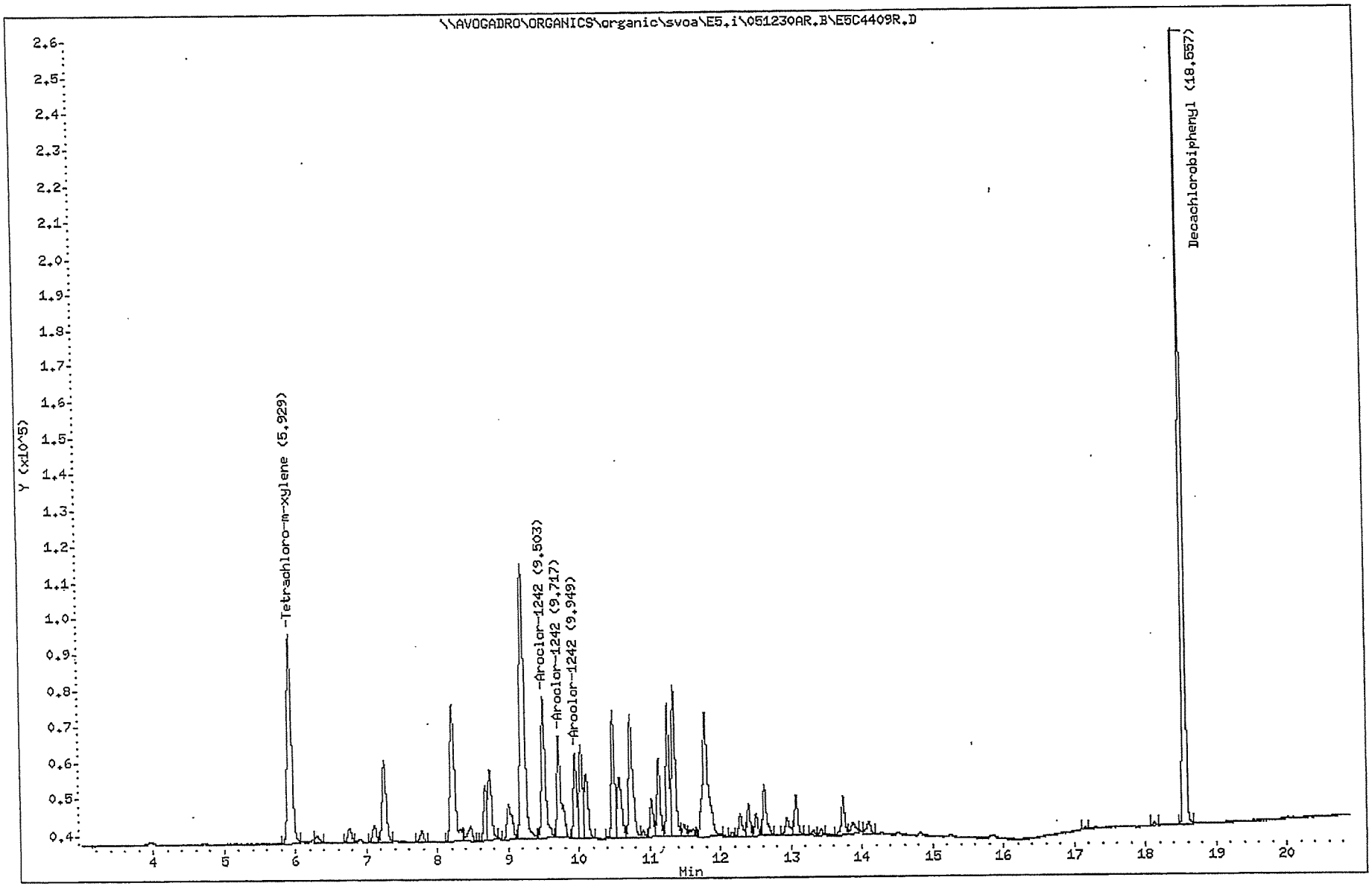

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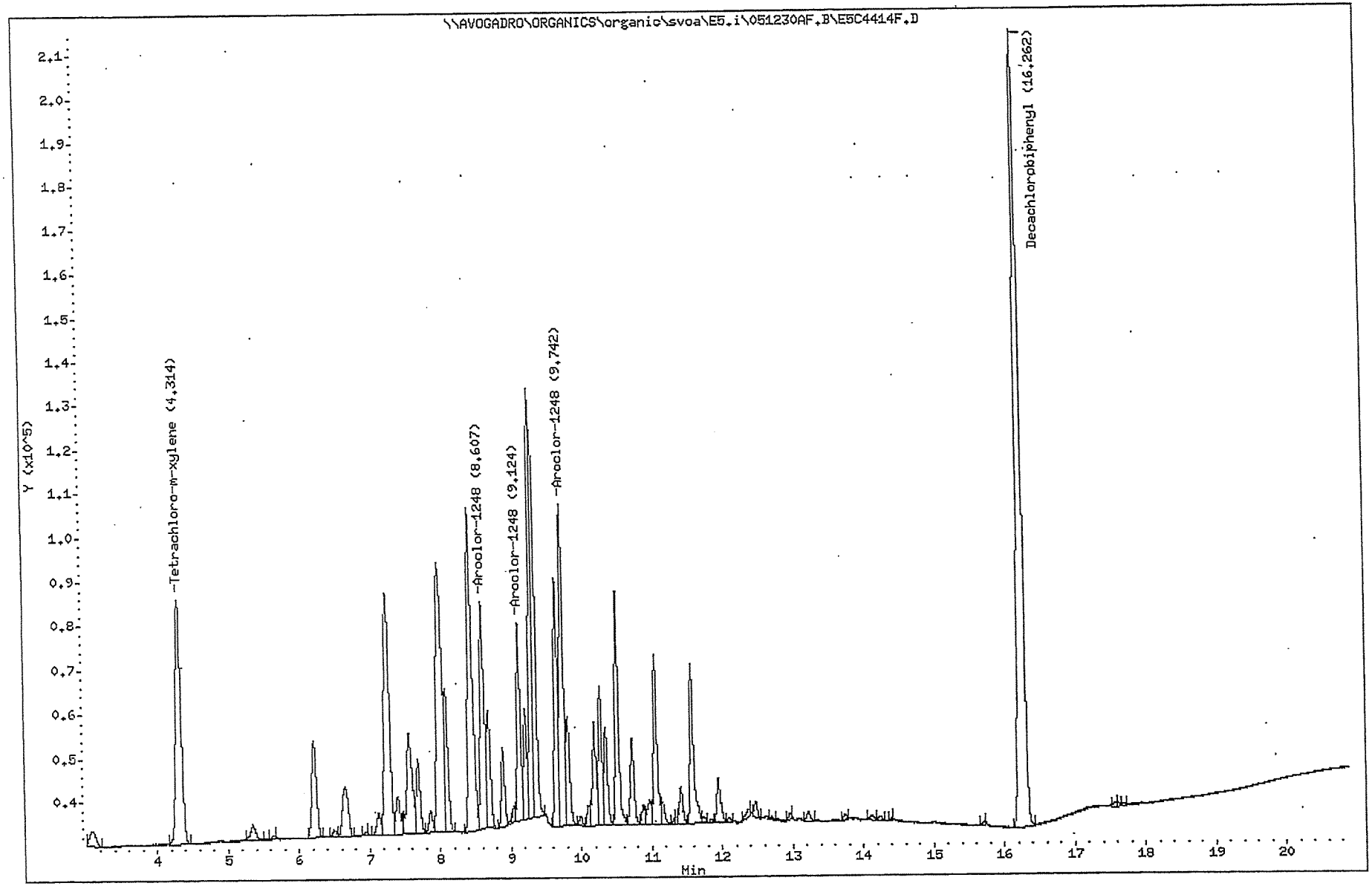

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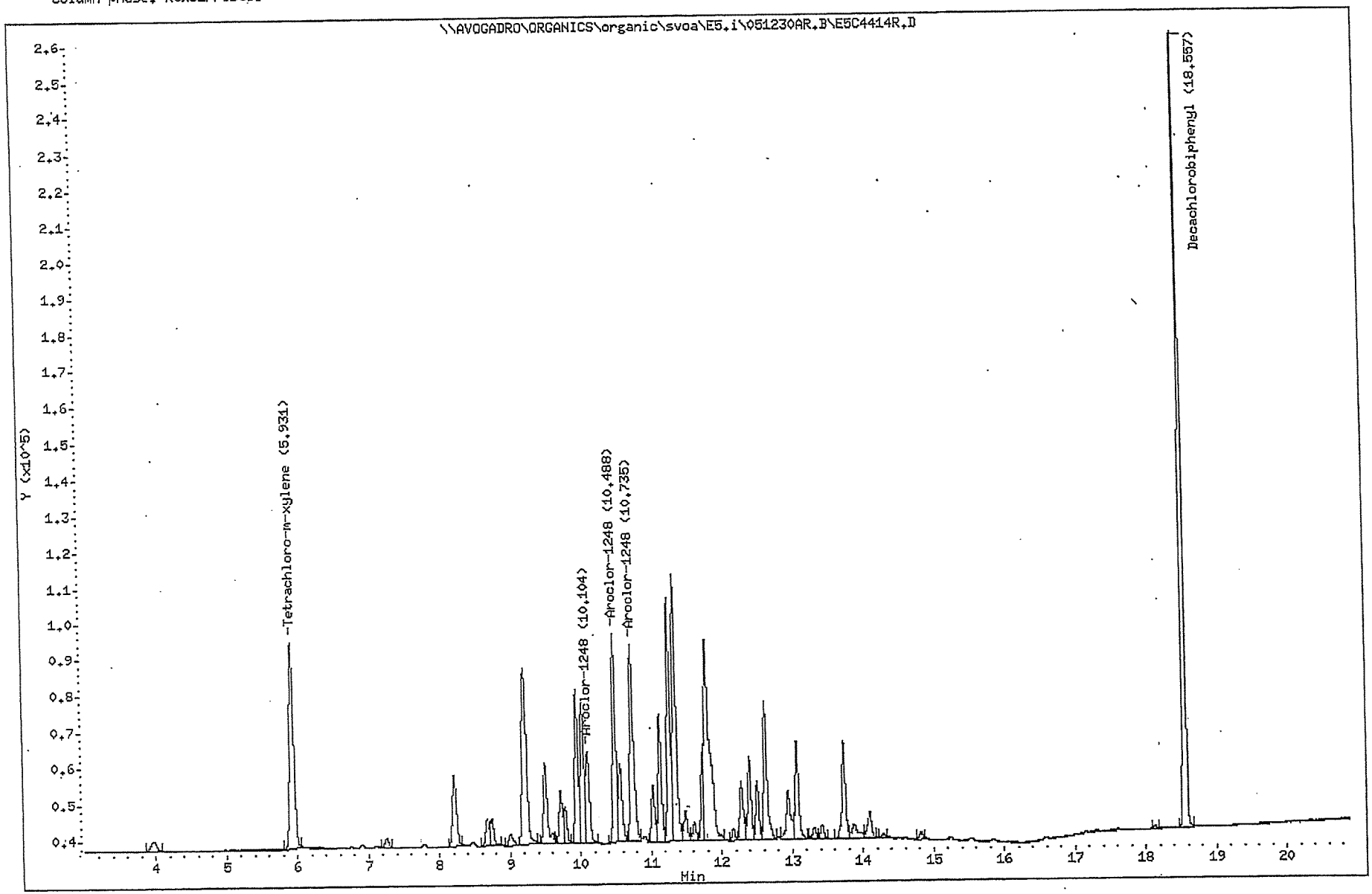

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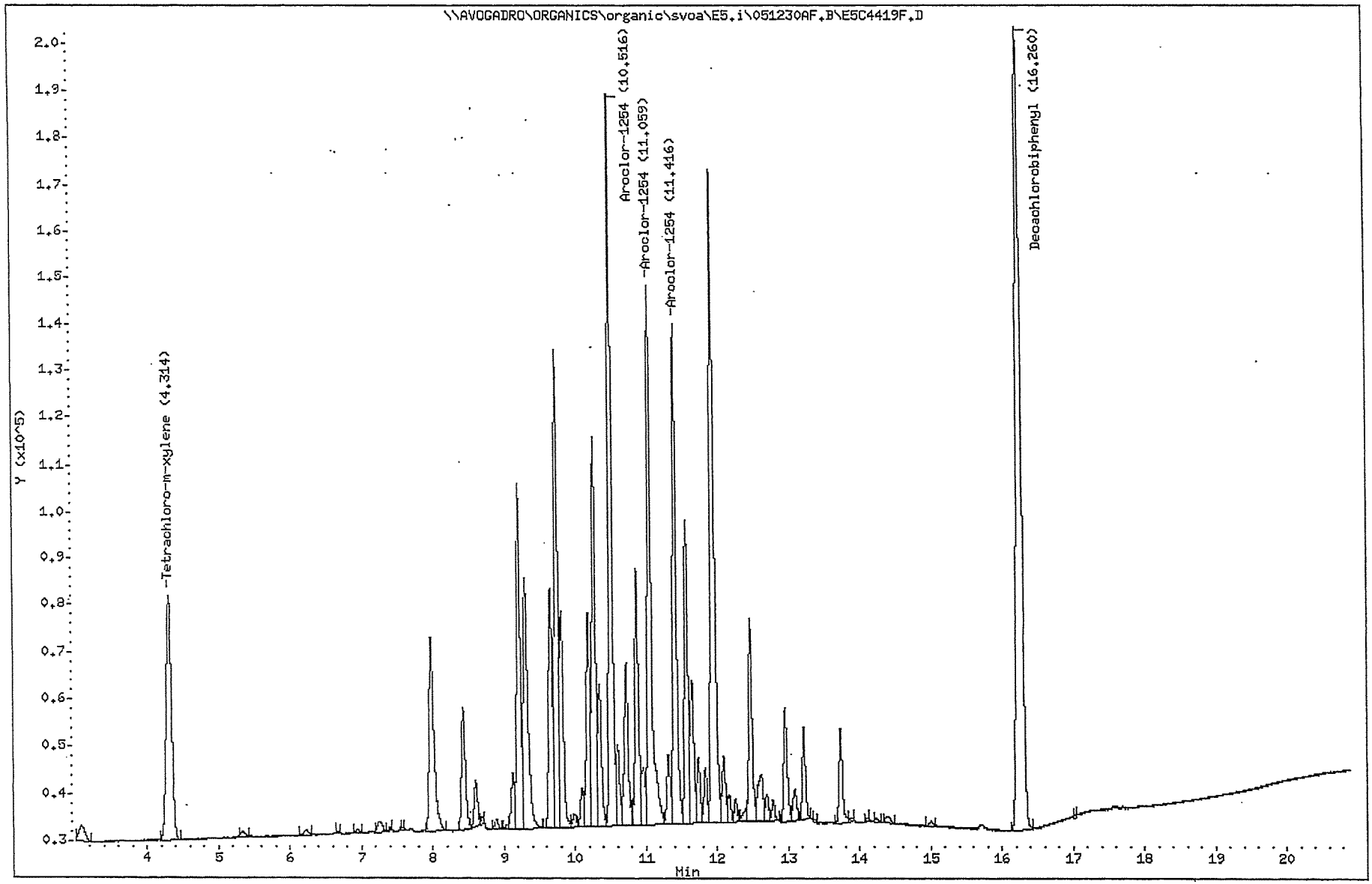

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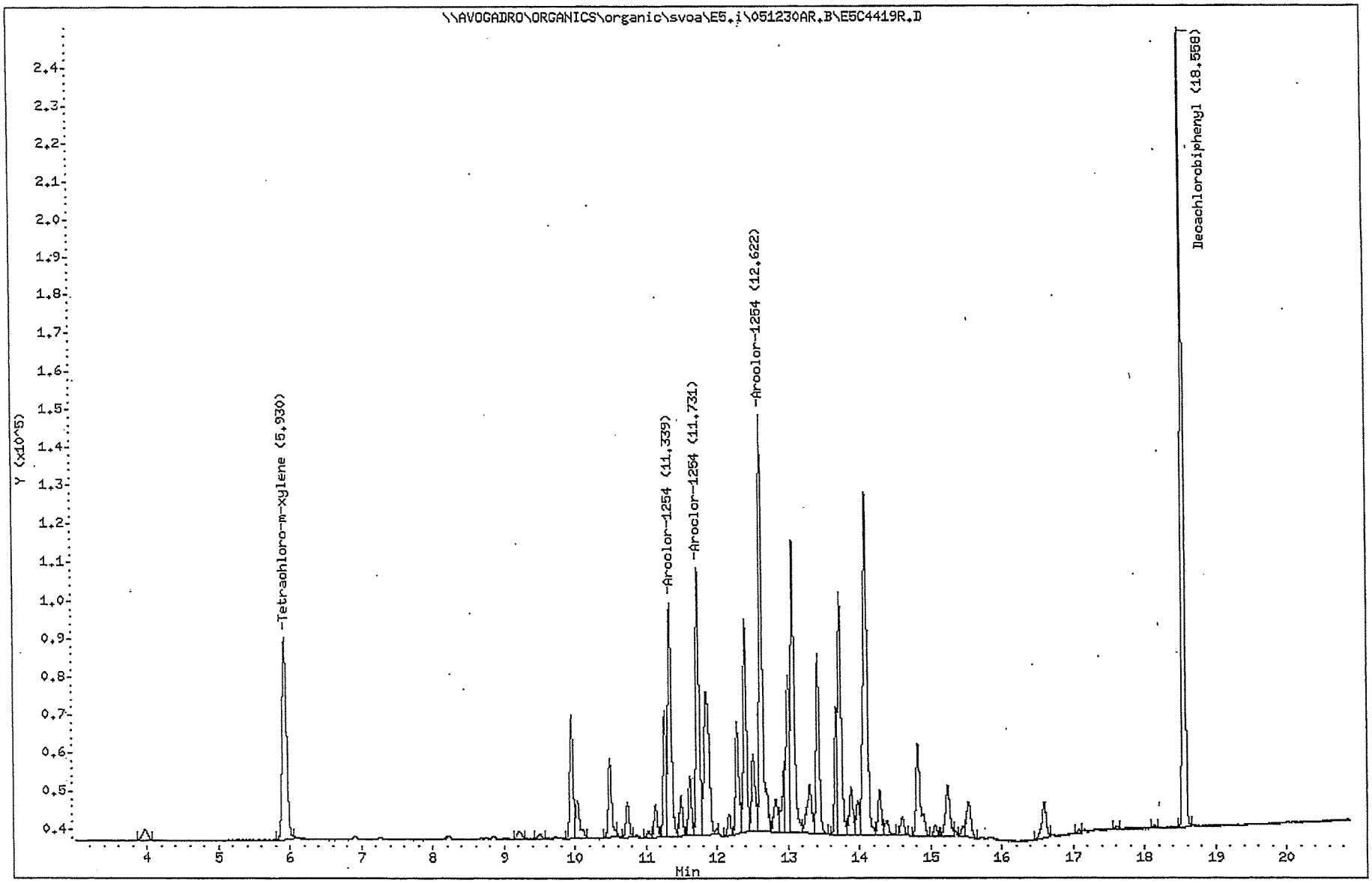

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Column phase: RtxCLPPest

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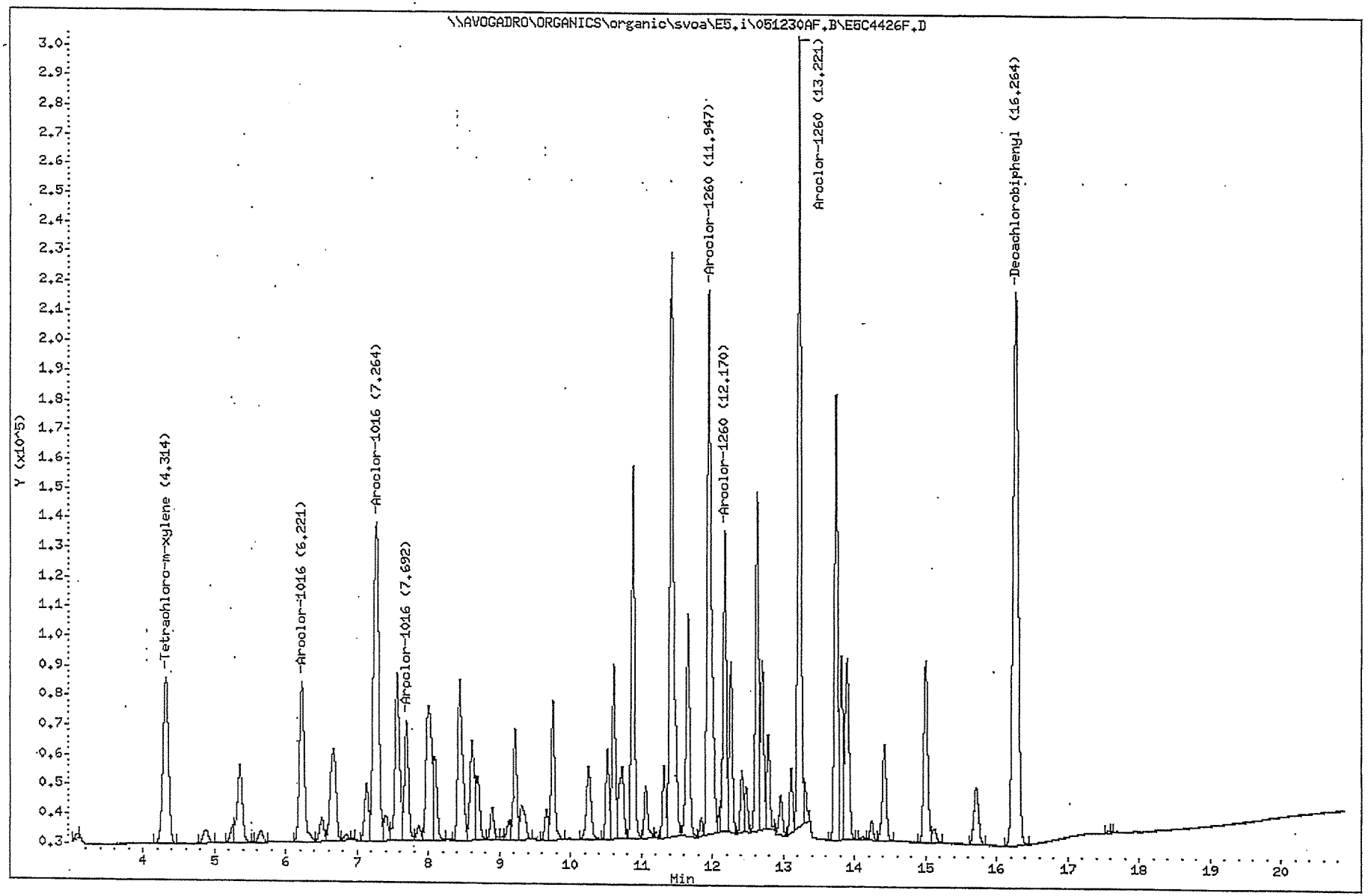
Column diameter: 0.53



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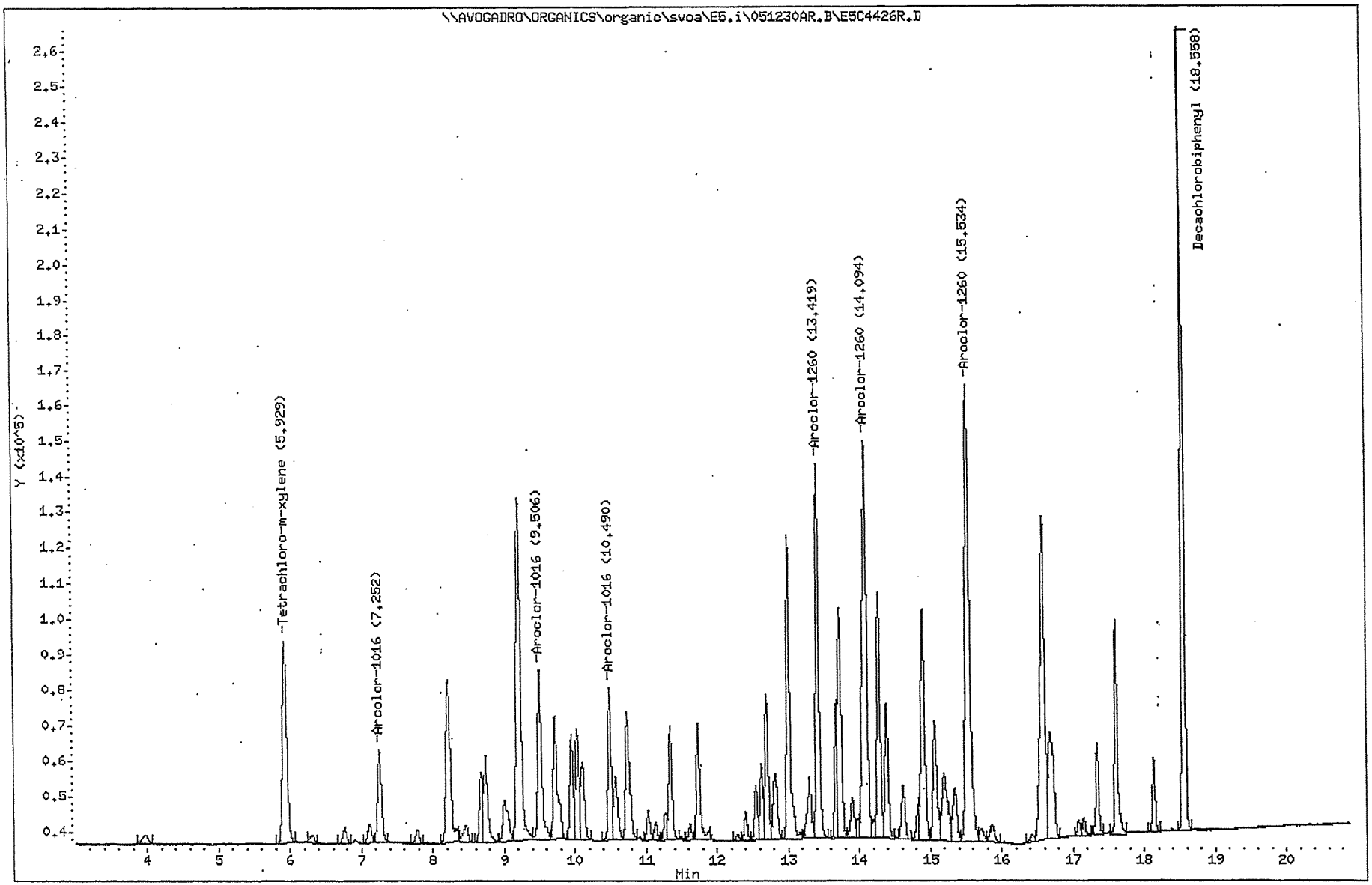

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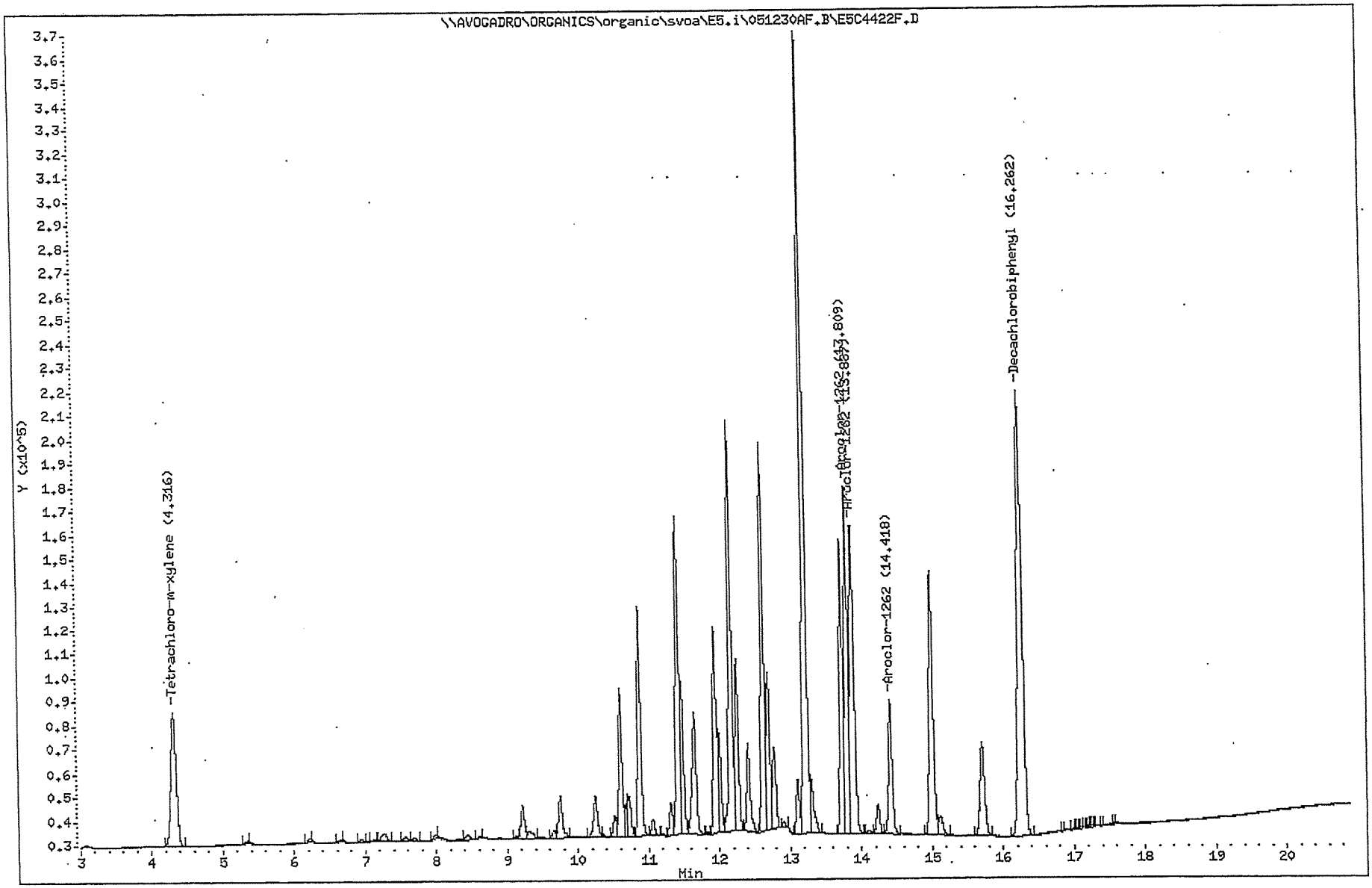

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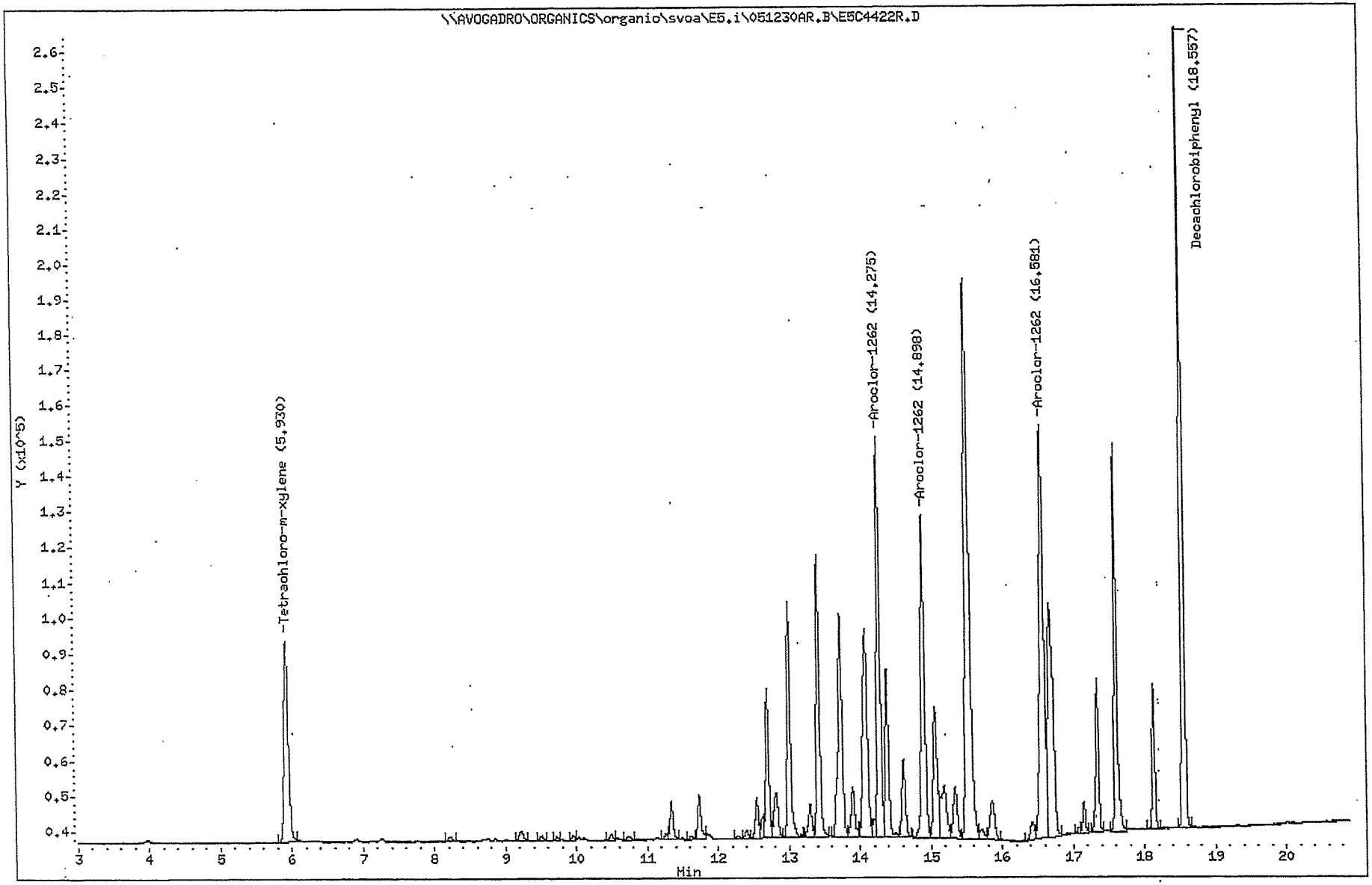
Column diameter: 0.53



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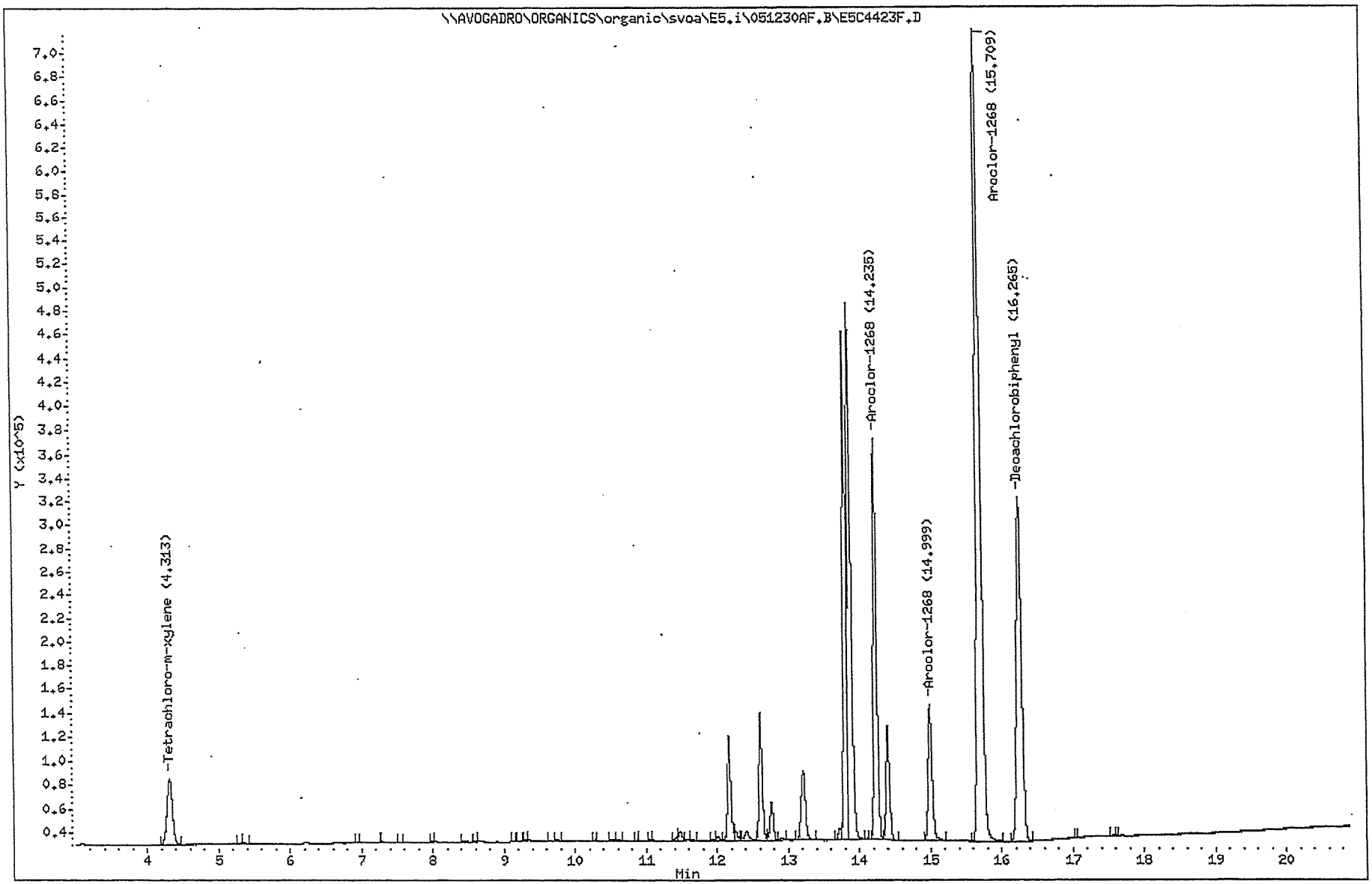
Operator: SRC:

Column phase: RtxCLPPest

Column diameter: 0.53



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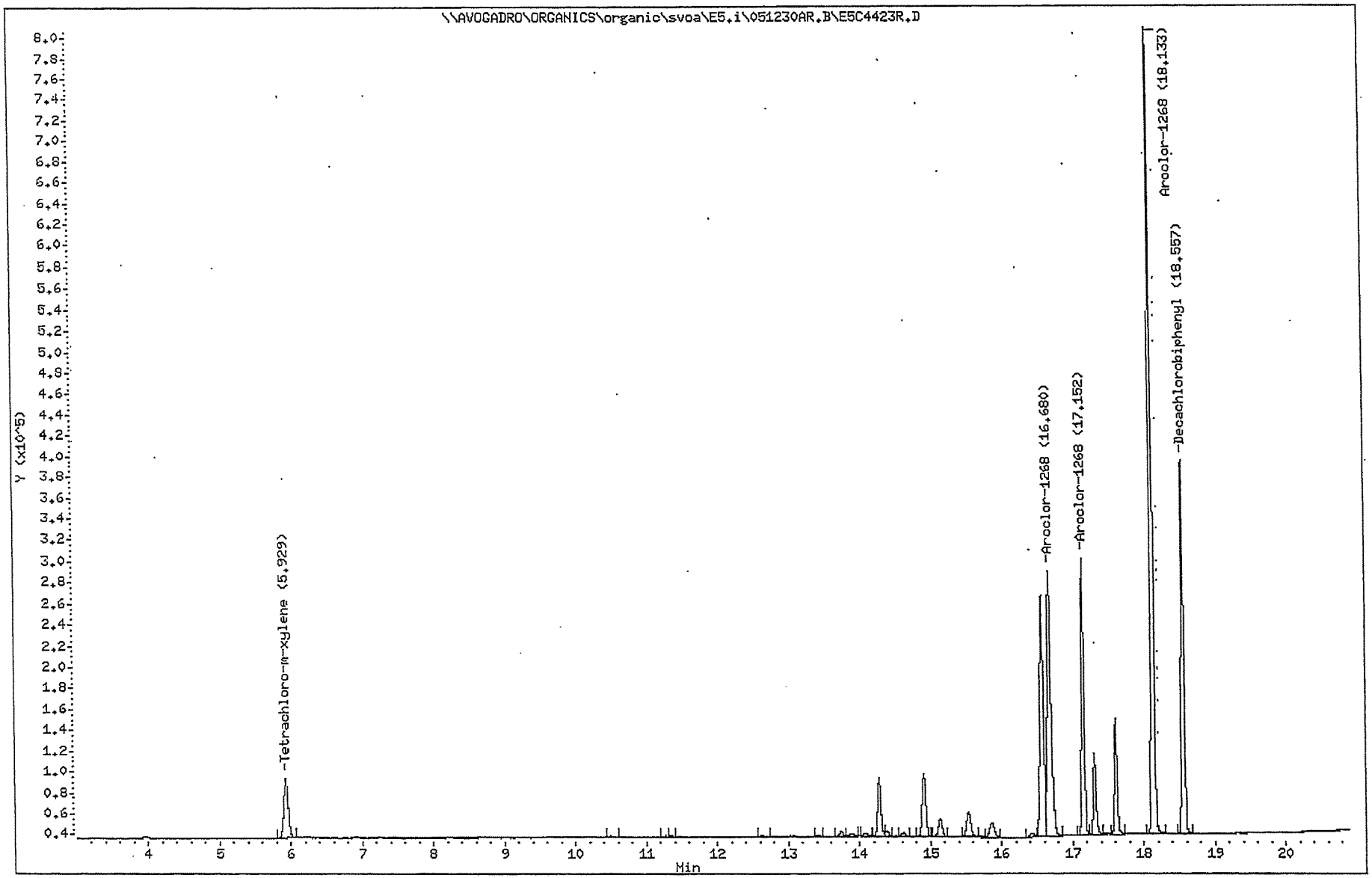
Operator: SRC:
Column phase: RtxCLPPestII
Column diameter: 0.53



Determination of Pesticides by Gas Chromatography/Electron Capture Detector Analysis by SW846 Method 8081B

Contents SOP NO. 60.0006

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		4/12/11
Quality Assurance Director	Shannon Bowler	4/11/11
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
Derek Turner	analyst	4.6.12			

Revision Record

Revision Date	Revision Description	Comments	Initials
12/10/02	Added control page and renamed SOP	Was SOP #O 06A	
4/17/08	Lab name change, update to 8081B	Additional DoD edit per audit 2008	SBL
8/22/08	Edited RPD calculation to match EPA SOM (definitive, conservative formula)	Consistency on report forms	SBL
1/5/09	%RSD calculation for Form X edit	Using SOM %D, new LIMS form	SBL
5/7/09	Add Mirex to SOP		SBL
11/24/09	QSM 4.1 ADDED	Attachment 2 Revised	SBL
4/8/11	Updated RT window info, LIMS std, added ASE. Removed QSM V3	full	SBL
7/15/11	Added new section 12	Removed E1	SBL
4/6/12	Analytical sequence order edit	Minor	DL
03/29/13	Added E6	Minor	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Determination of Pesticides by Gas Chromatography/Electron Capture Detector

Analysis by SW846 Method 8081B

Rev. 10

	Signature	Date
QA Director:		4/11/11
Lab Director:		4/12/11
Effective Date:	4/19/11	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Determination of Pesticides by Gas Chromatography/Electron Capture Detector

Analysis by SW846 Method 8081B

Rev. 10

1. Scope and Application

This SOP describes the procedures applicable to the analysis of the compounds listed in **Figure 1**, using a gas chromatograph equipped with an electron capture detector. This SOP gives specific information to perform the analysis according to protocols discussed in USEPA SW846 Test Methods for Evaluating Solid Waste Method 8081B, and Department of Defense Quality System Manual for Environmental Laboratories, Final Version 4.1. All matrices including ground water, aqueous samples, TCLP and SPLP extracts, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require extraction and/or clean-up prior to analysis. **Section 8.1** provides the SOP references for sample extraction and clean-up procedures to be used with this analytical procedure. A list of target analytes for TCLP analysis provided in **Figure 2.** A list of acronyms used in this SOP is included in **Figure 3**.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that this SOP is accurate and up-to-date, and that it is implemented appropriately. **Supervisors or Peer Analysts** review the logbooks and data generated from this procedure. The Data Reviewer evaluates all laboratory reports for reasonableness of the results and signs the reports. The **QA Director** reviews all quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure

3.1 A sample extract is analyzed by injecting a 1-2µL aliquot into a gas chromatograph (GC) using an auto-sampler. Concentrations of various organochlorine pesticides are determined by separation of the analytes using a GC equipped with fused silica, open-tubular, megabore columns and electron capture detectors (ECD). Hewlett Packard's EnviroQuant software is used to handle data acquisition. Target software from ThruPut is used for data reduction and forms generation.

3.2 Organochlorine pesticides are determined using dual-column systems with dissimilar phases. The method allows for the option of dual columns joined to a single injection port and individually connected to two ECDs

4. Sample and Sample Extracts Preservation, Containers, Handling and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. In some instances, clients will provide their own containers**.** Water samples are collected in 1liter amber glass bottles with no preservation added to the sample. Solid samples are collected in 8-ounce amber glass containers with no preservation. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may be required for the analysis of laboratory QC samples.

4.2 All sample extracts are stored at 4°C ± 2°C until analyzed.

4.3 Sample extract holding time for pesticides analysis is 40 days from date of extraction to date of analysis. The holding time for sample extraction is covered in the corresponding extraction SOPs.

4.4 The sample extracts are transferred from Organic Prep Laboratory with all appropriate sample prep information in 2ml auto-sampler vials with Teflon lined crimp cap. All vials should have a meniscus drawn marking the level of the extracts.

4.5 Samples, sample extracts and standards must be stored separately.

5. Interferences and Potential Problems

5.1 Sources of interference in this method can be grouped into three broad categories: (1) contaminated solvents, reagents, or sample processing hardware; (2) contaminated GC carrier gas, parts, column surfaces or detector surfaces; and (3) the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Interferences co-extracted

from the samples will vary considerably from waste to waste. While general clean-up techniques are referenced or provided as part of this method, unique samples may require additional clean-up approaches to achieve the desired degree of discrimination and quantitation.

5.2 Interference by phthalate esters introduced during sample preparation can pose a major problem in pesticide determination. These materials may be removed prior to analysis using Gel Permeation Clean-up (Method 3640). Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with plastic materials and batch analyzing the solvents and reagents for phthalate contamination. Exhaustive clean up of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

5.3 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination may be expected with sediment samples. Method 3660 is suggested for removal of sulfur.

5.4 Waxes, lipids, and other high molecular weight materials may interfere with the analysis of organochlorine pesticides and can be removed by Gel-Permeation Clean-up (GPC) (Method 3640). Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Certain co-eluting organophosphorous pesticides are eliminated by GPC. Co-eluting chlorophenols are eliminated by florisil clean-up (Method 3620).

5.5 The following target analytes may co-elute depending on the GC temperature program:

RTX-Pesticides	4,4'-DDE/Endosulfan I
	4,4'-DDD/Endosulfan II
RTX-Pesticides II	Heptachlor/delta-BHC
	4,4'-DDD/Endosulfan II

6. Equipment and Apparatus

6.1 Gas Chromatograph/Electron Capture Detector System.

6.1.1 There are 4 GC/ECD in the laboratory. Three are GC/µ-ECD are labeled as E4, E5 and E6. GC E2 is a Hewlett Packard (HP) Model 5890 Series II with electron Pressure Control. GC E4, E5 and E6 are HP Model 6890 Series II with u-Electron Pressure Control. All GC's are temperature programmable instruments with single injection port and dual electron capture detectors, and are equipped with HP Model 7673A Auto-sampler. A single gooseneck splitless injection liner is used in the injection port and connected to a piece (up to a 5m long) of uncoated megabore column (0.53mm id) which serves as a guard column. A guard column is connected to the tee split. Each of two columns is connected to the split tee also. HP 3365 Chemstation software is used in conjunction with EnviroQuant software to handle data acquisition and processing. All GC's are interfaced to the network workstation. LIMS software is used to generate data reports.

6.1.2 HP Vectra PC.

6.1.3 HP 3365, EnviroQuant and Target 4.14 software.

6.1.4 HP Model 7673A auto-sampler.

6.1.5 Chromatographic columns used in the laboratory:

RTX- CLPesticides (Restek or equivalent) 30 m x 0.53 mm id (0.5 um film thickness) and
RTX-CLPesticides II (Restek or equivalent) 30 m x 0.53 mm id (0.42 um film thickness).

6.1.6 Gooseneck splitless injection liner Cat #20799 from Restek or equivalent.

6.1.7 Uncoated megabore guard column (0.53 mm id, up to 5 m long) Cat #10028 from Restek or equivalent.

6.1.8 Universal "Y" Press-tight tee split Cat #20406 from Restek or equivalent.

6.2 Glassware:

6.2.1 Class A volumetric flasks:
5ml, 10ml, 25 ml, 50 ml, 100 ml, and 250 ml.

6.2.2 Syringes:

10 µl, 25 µl, 50 µl, 100 µl, 500 µl, 1 ml, 2.5 ml (accuracy to ± 1% per vendor's specification).

7. Reagents and Standards.

7.1 n-Hexane: pesticide quality or equivalent.

7.2 Reagent Water (ASTM Type II water) - prepared by passing tap water through a mixed bed of cation and anion exchange resin and activated carbon.

7.3 Acetone: pesticide quality or equivalent.

7.4 The standards used in the method are discussed below. Please note that standards from other vendors could be used as long as the standards are of high purity (>96%) and traceable to reference materials.

7.4.1 The list of primary (ampulated) standards:

- Performance evaluation mixture (PEM) at 1-25ug/mL (Restek Cat. # 32074).
- Toxaphene Mix at 1000ug/mL (Restek Cat. # 32005)
- Technical chlordane at 1000ug/mL (Restek Cat. # 32021)
- Organochlorine Pesticide Mix AB#2 (Restek Cat #32292)
- Tetrachloro-m-xylene (TCX) (Restek Cat.# 32027) at 200 ug/mL.
- Decachlorobiphenyl (DCB), (Restek Cat.# 32029) at 200 ug/mL
- Mirex (UltraScientific Cat. # PST-720M100A01) at 100 ug/mL.

7.4.2 The list of second source standards:

- Pesticide standard Mix A-1 at 0.5-5ug/mL (Supelco Cat. # 4-7977)
- Pesticide standard Mix B-1 at 0.5-1ug/mL (Supelco Cat. # 4-7978)
- Toxaphene at 1000ug/mL (Supelco Cat. # 4-8103)
- Technical chlordane at 1000ug/mL (Supelco Cat. # 4-8065)
- Mirex (Supelco Cat.# 861428-U) at 1000 ug/mL.

7.4.3 All of the standard information is recorded in the LIMS Standard Logbook upon receiving. See **Figure 4** for an example. All vials containing primary standards must be labeled according to the current version of SOP No. 80.0001 Standard Preparation, Equivalency and Traceability.

7.4.4 The expiration date for ampulated standards shall not exceed the manufacturer's expiration date. All primary standards are stored according to manufacturer's recommendation.

7.4.5 All Standards made from a Primary Standard must not exceed the Primary Standard's expiration date.

7.5 Standard Preparation:

Working standards are labeled as **PW*yymmdd*X**

where: PW = Pesticide Working standard
yymmdd = date the standard is prepared
X = the order the standard is prepared on that date, in alphabetical order.

An intermediate standard is labeled as **PIyymmddX**, as above, where **PI**=Pesticides Intermediate standard.

Be sure the vial label is not worn or difficult to read. Any container whose label becomes worn or difficult to read should be re-labeled.

Please note that the following preparation procedures pertain to the use of the primary stock standards listed in **Sections 7.4.1** and **7.4.2**. Different preparation schemes are needed if different stocks are used. All standards' preparation is documented in the LIMS Standard Logbook.

7.5.1 Surrogate Mix: An intermediate stock standard is made by diluting 0.5mL of stock TCX and 1.0 mL of stock DCB and diluting to 50 mL with hexane. Intermediate Pesticide/PCB Surrogate Mix contains TCX at 2 ug/mL and DCB at 4ug/mL.

7.5.2 Performance Evaluation Mixture (PEM): the Working solution is prepared by diluting 1mL of the stock to 100mL using n-hexane. The working PEM solution contains:

Compound	Concentration (ng/mL)
gamma-BHC	10
alpha-BHC	10
4,4'-DDT	100
beta-BHC	10
Endrin	50
Methoxychlor	250
Tetrachloro-m-xylene	20
Decachlorobiphenyl	20

7.5.3 Pesticide Standard Mix C (INDC): The Working Standard Mix C is prepared at 5 levels as follows:

Level 5: 1mL of the primary stock Mix AB and 4mL of the intermediate surrogate standard mix are diluted to 100mL in hexane. If Mirex is being analyzed, then 0.8mL of a 1.0ug/mL Mirex intermediate standard* is added to Level 5.

Level 4: 25mL of Level 5 diluted to 50mL in hexane.

Level 3: 25mL of Level 5 diluted to 100mL in hexane.
Level 2: 5mL of Level 4 diluted to 20mL in hexane.
Level 1: 2.5mL of Level 4 diluted to 20mL in hexane.

* dilute 1.0mL of Mirex primary standard to 10mL in hexane to prepare the intermediate standard at 1.0ug/mL.

The five level Working solutions contain:

	Concentration (ng/mL)				
Compound	Level 5	Level 4	Level 3	Level 2	Level 1
alpha-BHC	80	40	20	10	5
Heptachlor	80	40	20	10	5
gamma-BHC	80	40	20	10	5
Endosulfan I	80	40	20	10	5
Mirex	80	40	20	10	5
Dieldrin	160	80	40	20	10
Endrin	160	80	40	20	10
4,4'-DDD	160	80	40	20	10
4,4'-DDT	160	80	40	20	10
Methoxychlor	800	400	200	100	50
beta-BHC	80	40	20	10	5
delta-BHC	80	40	20	10	5
Aldrin	80	40	20	10	5
Heptachlor epoxide	80	40	20	10	5
alpha-Chlordane	80	40	20	10	5
beta-Chlordane	80	40	20	10	5
4,4'-DDE	160	80	40	20	10
Endosulfan sulfate	160	80	40	20	10
Endrin aldehyde	160	80	40	20	10
Endrin ketone	160	80	40	20	10
Endosulfan II	160	80	40	20	10
Tetrachloro-m-xylene	80	40	20	10	5
Decachlorobiphenyl	160	80	40	20	10

7.5.4 Toxaphene (TOX):

The working standard of toxaphene at 8.0ug/mL (level 5) is prepared by diluting 0.2mL of the primary stock Toxaphene standard, 1.0mL of the intermediate surrogate standard prepared in **Section 7.5.1** to 25mL using hexane. The other 4 level toxaphene standards are prepared as follows:

Level 1: 5.0mL of L2 diluted to 10mL of hexane.
Level 2: 5.0mL of L3 diluted to 10mL of hexane.

Level 3: 2.5mL of L5 diluted to 10mL of hexane.
Level 4: 5.0mL of L5 diluted to 10mL of hexane.

The concentrations of toxaphene in the standards are as follows (concentration in ug/mL):

	Toxaphene	TCMX	DCB
Level 1:	0.5	0.005	0.01
Level 2:	1	0.01	0.02
Level 3:	2	0.02	0.04
Level 4:	4	0.04	0.08
Level 5:	8	0.08	0.16

7.5.5 Technical Chlordane (TC):

Per project requirement, technical chlordane might be analyzed either in lieu of or in addition to the alpha- and gamma- isomers. If needed, technical chlordane standards are prepared as follows:

Level 5: 0.25mL of the primary Chlordane standard, 2.5mL of intermediate surrogate standard diluted to 50mL of hexane.
Level 4: 25mL of the above diluted to 50mL of hexane.
Level 3: 10mL of Level 5 diluted to 50mL of hexane.
Level 2: 5mL of Level 5 diluted to 50mL of hexane.
Level 1: 2.5mL of Level 5 diluted to 50mL of hexane.

The concentrations of technical chlordane in the standards are as follows (concentration in ug/mL):

	Technical Chlordane	TCMX	DCB
Level 1:	0.25	0.005	0.010
Level 2:	0.5	0.01	0.02
Level 3:	1.0	0.02	0.04
Level 4:	2.5	0.05	0.1
Level 5:	5.0	0.1	0.2

7.5.6 Second source standards:

The second source INDC mix is prepared by diluting 200uL of the second source stock pesticide standard Mix A, 200uL of the second source stock pesticide standard Mix B to 5mL using hexane. Add 100 uL second source intermediate Mirex standard when necessary. The constituents and concentration of the second source are the same as that of the mid-level primary calibration standard.

The second source toxaphene standard is prepared by diluting 25uL of the second source stock to 10mL using hexane. The second source toxaphene is at 2.5ug/mL.

The second source technical chlordane is prepared by diluting 100uL of the second source stock to 100mL using hexane. The second source technical chlordane is at 1.0ug/mL.

7.5.7 With the use of stock standards listed above, preparation volumes used could be adjusted to make smaller or larger volume of standards as long as the final working concentrations are the same.

The standards are stored in amber containers under refrigeration at 4°C ± 2°C. All standards are stored in a separate location from the samples and/or extracts to minimize cross contamination.

The expiration dates for the working standards are six months from the date of preparation.

8. Procedure

8.1 Extraction and Cleanup:

8.1.1 Preparation – The methods in USEPA SW-846 for sample extraction are as follows:

Method 3510 (SOP# 50.0051) extracts aqueous samples for water-insoluble and slight water-soluble organics. The samples are serially extracted with methylene chloride using a separatory funnel.

Method 3520 (SOP# 50.0050) extracts aqueous samples for water-insoluble and slightly water-soluble organics. The samples are placed in continuous liquid-liquid extractor and extracted with methylene chloride for a minimum of 18 hours.

Method 3540 (SOP# 50.0053) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate to form a free-flowing mixture, then extracted in 1:1 v/v methylene chloride/acetone in a soxhlet extraction.

Method 3550 (SOP# 50.0052) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate to form a free-flowing mixture, then extracted in 1:1 v/v methylene chloride/acetone using ultrasonic extraction.

Method 3570 (SOP# 50.0100) is the extraction of soil, sediment, tissues, biota and any sample considered solid. A 2-20gram sample is solvent extracted first with acetone, and then with hexane by either a manual shake approach or via rotation or spinning of the sample.

Method 3545 (SOP# 50.0101) is the extraction of soil or sediment. A 15gram sample is mixed with diatomaceous earth to form a free-flowing mixture. This is added to the sample extraction cell. The cell is loaded on the PFE extractor to perform the extraction. The resulting extract is then filtered through anhydrous sodium sulfate, and concentrated to the appropriate final volume.

In addition, clean-up procedures are employed to remove co-eluting interferences. The lab uses florisil clean-up (Method 3620), GPC clean-up (Method 3640) and sulfur clean-up (Method 3660).

8.2 Instrument conditions:

8.2.1 Optimize the GC operating conditions for analytes separation and sensitivity. Once optimized the same conditions must be used for analysis of all standards, samples, blanks and QC samples.

The GC operating conditions are as follows:

Carrier Gas	Helium (99.999%)
Column Flow	5-10 mL/min., independent of temperature
Make-up Gas	5% methane/95% argon (P-5)
Make-up Gas Flow	80-100 mL/min.
Injector	Splitless
Injector Temperature	200°C-210°C
Initial GC Temperature	110°C-150°C
Initial GC Hold	1 min.
Temperature Ramp	3-9°C/min
Final Temperature	270-300°C
Final GC Hold	3-15 min.
Detector Temperature	300°C

In the event that these conditions are changed, Enviroquant Data Acquisition methods containing the actual GC operating conditions are copied and sent to the network along with all GC/ECD raw data files. They are located in a folder in the sequence batch called “Zacq”.

8.2.2 Preventative Maintenance

The injector septum is replaced every time the instrument is set up to perform a sequence of analyses, when a leak develops, or when initial and/or continuing calibrations fail to meet the method requirements.

The injection liner is replaced every time the instrument is set up to perform a sequence of analyses, when a leak develops, when the degradation criteria (**section 8.2.7**) fail, or when initial and/or continuing calibrations fail to meet the method requirements. The gold seal will be replaced and the columns will be trimmed every time before a new calibration is run.

The column will be replaced if standard chromatograms show excessive peak tailing or initial and/or continuing calibrations repeatedly fail to meet the method requirements.

Major instrument maintenance must be documented in the LIMS Maintenance Logbook. Routine (daily) maintenance can be documented in Instrument Run Logbook. Also refer to SOP 110.0040 Instrument Maintenance and Documentation for additional information.

8.2.3 Troubleshooting:

Please refer to HP reference manual and operation manual on HP 5890 Series II GC.

8.2.4 The auto-sampler makes 1-2µl injections for the analysis of all standards, QC, and sample extracts.

8.2.5 All analytical runs need to be documented in the appropriate Instrument Run Log. In addition to listing the data file, the associated working standard ID and standard name should be noted.

8.2.6 After replacing column and/or performing major chromatographic maintenance, the column should be primed (deactivated) by injecting a mid-level or high-level standard prior to the analysis of the initial calibration. An instrument blank (hexane) will need to be analyzed after priming the column to minimize any carryover. In some instances two instrument blanks may be needed.

8.2.7 After the instrument blank(s) have been successfully analyzed and the system is ready, a PEM standard is analyzed to ensure the chromatographic system is inert and that breakdown of labile analytes is minimal.

PEM is analyzed at the start of the initial calibration sequence and is part of the continuing calibration process.

Breakdown is evaluated as follows:

$$\% \text{ DDT Breakdown} = \frac{\text{Total DDT degradation areas (DDE + DDD)}}{\text{peak area (DDT + DDE + DDD)}}$$

$$\% \text{ Endrin Breakdown} = \frac{\text{Total Endrin degradation (aldehyde + ketone)}}{\text{peak area (Endrin + aldehyde + ketone)}}$$

The % breakdown for each of the two compounds should not be greater than 15%.

If the breakdown exceeds the above criteria, perform and document inlet maintenance including replacing the gold seal at the base of the injection liner.

8.3 Initial calibration

Prior to the analysis of the samples each GC /ECD must be initially calibrated. The initial calibration is performed by analyzing 5 levels of IND C and Mid level only of multi-component (Toxaphene and Chlordane) standards in the following sequence:

Order	Sequence	Comment
1	PEM01	Degradation check for DDT and Endrin
2	TOXL3	Mid level Toxaphene
3	TCL3	Mid level Technical chlordane
4	INDCML1	
5	INDCML2	
6	INDCML3	
7	INDCML4	
8	INDCML5	
9	PIBLK	
10	PEM02	Degradation check for DDT and Endrin
11	INDCMss	

ss = Second Source

Based upon the positive identification of either multi- component pesticide (Toxaphene or Chlordane) in the samples, a new initial calibration will be performed with 5 level standards and all affected samples will be reanalyzed for accurate quantitation.

The chromatograms of the above standards are presented in **Figures 6** through **9** for CLP PEST columns.

When using the CLPPEST1 and CLPPEST2 columns, it is important to make certain that the analytes Endosulfan II and 4, 4' DDD are resolved on at least one column. If the criterion is not met, positive identification of either analyte can not be made.

From the initial calibration, the following parameters are determined:

8.3.1 Retention time and retention time window:

It is our experience that the GC retention times operating under EPC conditions are very tight. Using the 72 hour approach to determine retention time windows at times had resulted in extremely narrow retention time windows. This may result in false negatives due to slight retention time shifts, especially when sample extracts are subjected to co-eluting interferences. Therefore, the procedures for establishing analyte RT and their windows have been taken from the USEPA SOM Statement of Work.

For the single component pesticides, the retention time is measured in each of the initial calibration standards. For the multi-component standards including toxaphene and technical chlordane four major peaks are chosen for toxaphene and three for technical chlordane. The mean RT is calculated as the average value for each of these peaks.

The retention time windows used by the laboratory are listed in **Table 1.** These windows are applied to the mean RT of the target compounds as determined from the initial calibration. Technical Chlordane uses ±0.07minutes. It has been the laboratory's experience that the use of these windows are optimal in that they are narrow enough to minimize the detection of spurious peaks as false positives but also wide enough to detect true positives.

8.3.2 Calibration factor. Mitkem chooses to use external quantitation for instrument calibration and sample quantitation. Internal Standards calibration is allowed per method 8081.

The following equation is used to calculate the calibration factor (CF):

$$CF = \frac{\text{Peak area (or height) of standard}}{\text{Mass injected (ng)}}$$

The above equation is used to determine the calibration factor for both the single component and multi-component standards. Please note that either peak area or peak height can be used for the calculation .

Mitkem chooses to use peak area for single component analytes. For multi-component analytes such as toxaphene, the laboratory uses peak height. At present, area percent reports can be generated for clients who request them. Area percent reports list the height for each peak in the run. Due to the multi-component nature of Toxaphene (chlorinated camphenes), it contains both resolved and unresolved peaks. The unresolved "hump" area is an important part of the total response of Toxaphene and should be included in the calibration calculation. In all Toxaphene standards, the baseline should be constructed between the retention times of the first and last eluting peaks. The heights of the four major peaks chosen for calibration should originate from this constructed baseline.

8.3.3 Linearity - linearity is used to evaluate the dynamic range of the calibration factor for each of the single component and multi-component standards. Linearity, as measured by the % Relative Standard Deviation (RSD) is calculated using:

$$\% \text{ RSD} = \frac{SD_{CF}}{CF_{av}} \times 100$$

where: $SD_{CF} = \sqrt{\sum (CF_i - CF_{av})^2 / (n-1)}$
CF_{av} = average calibration factor
CF_i = calibration factor
n = total number of values = 5

8.3.3.1 % RSD Acceptance criteria for linearity:

Initial Calibration is deemed acceptable if % RSD for each target analytes is less than or equal to 20%. If this condition is met, the instrument is determined to be linear within the calibration range.

Given the large number of target analytes, it is likely that some analytes may exceed the acceptance limit. In those instances, the linearity check is deemed acceptable if the following conditions are met (in order of preference):

8.3.3.2 Acceptance criteria for linearity using a least squares regression calibration:

In this case, the analyst may employ a regression equation for the analyte(s) that do (es) not pass using the earlier approach. The regression will produce the slope and intercept terms for a linear equation in the following form:

$$y = mx + b$$

Where y = instrument response (peak area or height)
m = slope of the line
x = concentration of the calibration standard
b = intercept

It is important that the origin (0,0) is **not** included as the sixth calibration point and that the above equation is not forced through the origin.

The linear regression is deemed acceptable if the correlation coefficient $r \geq 0.995 (r^2 \geq 0.990)$

8.3.3.3 Acceptance criteria for linearity using second order quadratic fit:

If the above still fails, a final approach in evaluating the linearity is performed using a second order quadratic fit. This, however, requires the analysis of an additional standard level. If this approach is taken, the additional level will be prepared at concentration that is the average of the 4^{th} and 5^{th} standard.

The second order quadratic fit will have the following equation:

$$y = ax^2 + bx + c$$

Where y = instrument response (peak area or height)
a and b = slope of the curve
x = concentration of the calibration standard
c = intercept

In performing second order quadratic fit, the analyst should not force the curve to pass through the origin (0,0). In addition, the origin should not be used as an additional calibration point.

From the quadratic fit, the "goodness of fit" is evaluated by calculating the coefficient of determination (COD). In order to be acceptable, the COD of the polynomial must be greater than or equal to 0.99.

Please note that the above discussion applies to both the single component analytes as well as the multi-component analytes including toxaphene and technical chlordane. For these multi-component analytes where three or four peaks are used for qualitative and quantitative determination, the linearity is evaluated using these individual peaks.

A second column confirmation is not required for the analysis of toxaphene and chlordane.

8.3.4 Second source calibration verification – a second source calibration verification (CCV) or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. This calibration is performed by analyzing the standards prepared in **section 7.5.8**. The calculated value of the analytes in the ICV should be **80 – 120%** of the expected value. If the ICV does not meet the criteria, see corrective action guidelines in **Attachment 2** of this SOP.

Results of this evaluation should be documented in the Instrument run log, and any outliers noted.

If the above criteria are not met for the ICV, the analyst must evaluate the integrity of the primary and confirmation standards. If needed, preparation and re-analysis of the initial calibration is required.

8.3.5 Initial calibration acceptance criteria must be met before any sample, blanks or LCS is to be analyzed.

8.3.6 Corrective Action for Initial Calibration – see **Attachment 2** for corrective action guidelines and documentation.

8.3.7 Initial calibration data must be archived in the Mitkem organics analysis calibration (OCAL) database. See Mitkem SOP #10.0009 (Application Xtender) for scanned archive information.

8.4 Calibration Verification and Sample Analysis:

Sample analysis may start immediately upon successful completion of the initial calibration. Continuing calibration verification (CCV) standards are then analyzed every ten field samples to make sure that the system performance is maintained. The analytical sequence can continue as long as the CCV meets the criteria. The sequence is concluded by analysis of the CCV standards. All standard analyses are to be documented in the appropriate Instrument Run Log (**Figure 5**) with associated working standard IDs.

Continuing calibration verification standards (CCV) containing all single target analytes and surrogate compounds are performed every time samples are to be analyzed to ensure that the GC/ECD system continues to meet instrument sensitivity and linearity requirements.

8.4.1 Frequency of Continuing Calibration- A CCV standard must be injected at intervals of not less than once every twenty field samples (the method recommends the frequency to be once every ten samples, to minimize

reanalyses due to unacceptable calibration verifications) and at the end of the analysis sequence.

DoD QSM requires CCV standards must be injected at intervals of not less than once every ten field samples.

8.4.2 Procedure for Performing Continuing Calibration Verification - The CCV is performed using the PEM, midpoint standard for INDC and low-point standard of Toxaphene and Technical chlordane (when needed).

- Calculate the percent breakdown of DDT and Endrin based on area, in the PEM standard. Refer to **section 8.2.5**. Results should be documented in the appropriate instrument logbook.

- Calculate the % difference between the continuing calibration CF and those from the most recent initial calibration. The % difference is determined as follows:

$$\% \text{ Difference} = \frac{CF_c - CF_i}{CF_i} \times 100$$

where:

CF_c = calibration factor from continuing calibration
CF_i = mean calibration factor from the most recent initial calibration which meets acceptance criteria

- Use % drift when using linear regression or second order quadratic fit calibration.

$$\% \text{ Drift} = \frac{Conc_c - Conc_t}{Conc_t} \times 100$$

where: $Conc_c$ = concentration obtained from continuing calibration
$Conc_t$ = theoretical concentration of standard

8.4.3 Evaluating the CCV

%D (% Drift) between the continuing calibration factor and that of the initial calibration for INDC should be no greater than ± 20% at least on one column. If this criterion is met then the calibration has been verified and sample analysis can proceed. The analyst should mark whether the CCV passed on one or both columns in the instrument logbook. Checkmarks or the term "OK" are acceptable entries.

There are no QC limits for multi-component (Toxaphene and Technical Chlordane) low - level standards at this time. Analysts must note in the runlog whether the standard was "detected/undetected".

Please note that the above calibration verification discussion applies to both the single component analytes as well as the multi-component analytes including toxaphene and technical chlordane.

8.4.4 Comparison of the retention time window: the retention time of each analyte and surrogate in the calibration standards should fall within the retention time windows established from the initial calibration. Due to the need to perform routine column maintenance (clipping off a small length of the guard and/or analytical columns), the retention times might be shortened. The retention time windows can then be updated using the first continuing calibration verification's absolute RTs to set the midpoint of the window, or a new curve must be analyzed. All subsequent closing and opening CCV analyses must fall within the new retention time windows to be valid.

8.4.5 Corrective action for the CCV:

See **Attachment 2** for corrective action guidelines and documentation.

The results for the bracketing calibration verification standards must meet the criteria discussed above. When a calibration verification standard fails to meet the QC criteria on both columns, all samples that were injected after the last compliant standard must be evaluated to ensure the data is valid.

Data associated with unacceptable calibration verification may be useable (with narrative notation) under the following special conditions:

1) When the acceptance criteria for the bracketing continuing calibration verifications are exceeded high, (>20%D=high bias), and the associated samples are non-detects, then those non-detects may be reported.

2) When the acceptance criteria for the bracketing continuing calibration verifications are exceeded high on one column only,(>20%D=high bias), and the associated samples are detects, the reported values must come from the compliant column, even if this is the higher result.

3) When the acceptance criteria for the bracketing continuing calibration verifications are exceeded high on both columns, (>20%D=high bias), and the associated samples are detects, then these samples will require reanalysis.

4) When the acceptance criteria for bracketing continuing calibration verifications are exceeded low (>-20%D=low bias), those sample results

may be used for screening purposes. These samples will require reanalysis for reportable results.

8.5 Identification of Analytes:

For single component target pesticides, analytes are identified when peaks are observed within the retention time windows for the analyte on both GC columns.

For multi-component target pesticides including toxaphene and technical chlordane, analytes are identified when peaks are observed in the retention time windows for the three peaks that were selected for calibration. In addition to the retention time window, the GC pattern of the residue should be comparable to the standards, and may take precedence over retention time.

Samples that contained weathered chlordane and/or toxaphene present special analytical challenges. Weathering could alter the pattern to the extent that different peaks have to be selected for quantitation. In these instances, the expertise of the analyst performing the evaluation is required. Analysts should bring samples of this nature to the attention of their supervisor for further assistance.

The peaks meeting the above retention time criteria will be quantitated using the appropriate calibration factor. The concentration of the target compounds and surrogates are calculated separately for both columns using the following equation:

$$\text{For aqueous samples, concentration } ug/L = \frac{(Ax)\,(Vt)\,(DF)}{(CF)\,(Vo)\,(Vi)}$$

$$\text{For soil samples, concentration } ug/kg = \frac{(Ax)\,(Vt)\,(DF)}{(CF)\,(Ws)\,(Vi)\,(S)}$$

where: Ax = area/height of the peak for the compound to be determined
CF = calibration factor
Vo = volume of water extracted, in mL
Ws = weight of soil sample, in gram
Vi = volume of extract injected, in µL
Vt = volume of extract, in µL
DF = dilution factor
S = solid content, expressed in decimal

For multi-component pesticides including toxaphene and technical chlordane, the reported target concentration will be determined by taking an average of the three-four peaks selected for the quantitation.

The sample concentration is determined for both columns. The lower of the two values is reported unless special circumstances exist such as co-elution or state/government programs require otherwise.

In instances where the % D, as discussed in **Section 8.4.5,** pass for only one of the two columns in the closing CCV, the analytical value is reported from the compliant column.

The difference between the two determined values is calculated using the equation listed below. The analyst should evaluate the chromatogram to see if the discrepancy might be due to co-eluting interferences. If the difference is greater than 40% between the columns, both analytical values will be reported and the lower value flagged with a 'P' qualifier on the sample data report form (Form I) to notify the data user that one of the concentrations might be biased due to co-eluting interferences. Results from both GC columns are reported on the Form X.

$$\%D = \frac{C_H - C_L}{(C_L)} \times 100\%$$

Where C_H = Higher Concentration
C_L= Lower Concentration

Please note that this formula is taken from the determinative EPA SOM methods and not SW-846 Method 8000. Method 8000 suggests using the average of the two concentrations as the denominator. The above calculation provides a more conservative approach to the RPD calculation. An additional data report sheet is available through LIMS comparing the two GC column results using the SW-846 calculation if needed.

The concentration of the surrogate analytes are calculated and reported. The acceptance limits for the recoveries are discussed in **Section 10.5**.

The analyte recoveries of the LCS and matrix spike compounds are determined and reported.

8.6 Re-analysis at dilution - Any target compounds that are determined above the instrument calibration range will require reanalysis at dilution. A notation for the need to perform a dilution next to the sample run in the instrument logbook is needed to document the results. An entry such as "rerun at 4x" is acceptable.

The dilution is performed by taking an aliquot of the extract and diluting it to a pre-determined volume using hexane. The analyst should not over-dilute the extract. The analytes that trigger the need for dilution should be determined in the diluted analysis at a concentration above that of the midpoint calibration standard

8.6.1 When reporting diluted results the following guidelines should be followed: If an initial analysis is performed that meets all QC criteria with the exception of compounds exceeding the upper calibration limit, this analysis may be reported. The sample ID of the initial (less dilute) analysis is unchanged and the ID of the dilution analysis has the DL suffix appended to both the client and sample ID. Those compounds exceeding the calibration range are qualified with the "E" flag on the data sheet for the less dilute analysis, and all compounds detected in the more diluted (DL) analysis are qualified with the "D" flag.

8.6.2 If the laboratory has prior information that a sample may contain concentrations of target or non-target compounds exceeding the calibration range of the instrument, the initial analysis may be performed at dilution. If this analysis is acceptable (compounds at or above the mid-point calibration) then a less dilute analysis is not required. The sample ID is not changed by adding a DL suffix but the 'initial analysis at dilution' is noted on the data review checklist included with the data submitted for review, to allow discussion in the project narrative.

8.6.3 If the initial analysis fails QC criteria, it may be reported if specified by the project or client. If only the dilution is reported, the DL suffix is not added and the dilution is noted on the data review checklist submitted with the data for review as above.

8.7 Analytical sequences are summarized as follows:

Order	Sequence	Comment
1	PIBLK01	Instrument blank
2	PEMAA	Degradation check for DDT and Endrin
3	INDCAA	INDC mid-level
4	TOXAA	low-level Toxaphene
5	TCAA (if needed)	low-level Tech. Chlordane
6	Blank	Method blank
7	LCS	Method LCS
8-17	(≤) 10 extracts	

18	PIBLK02	
19	PEMAB	
20	INDCAB	CCV
21	TOXAB	CCV
22	TCAB (if needed)	CCV
23	PIBLK02	

9. Data Reduction and Calculations

9.1 Sample data should be reported in units of µg/L for aqueous samples and µg/Kg dry weight for solid samples.

Results are reported to two significant figures using the USEPA guidelines in rounding up or down. For 8081B analysis, analytes are detected if the determined concentration is above the reporting limit on both columns. No J flagged values are reported on sample data report form Is.

9.2 Soil concentrations are calculated using dry weight basis. To convert soil results to a dry weight basis, divide the sample concentration by the percent solids.

The % solids in solid samples is calculated as follows:

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100\%$$

where: DW = Sample weight (g) dried at 105°C overnight
WW = Sample weight (g) before drying

9.3 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

$$\% \text{ Recovery (\%R)} = 100\% \times (SSR-SR)/(SA)$$

where: SSR = spiked sample result
SR = sample concentration
SA = spike added

9.4 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

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

where: RPD = relative percent difference
D1 = first sample value

D2 = second sample value

9.5 Manual integration will be performed if needed and documented according to the current revision of SOP Number 110.0008. Manual integration is appropriate when sample-specific chromatographic conditions prevent the automatic integration routines from properly assigning baseline, resulting in improper quantitation. Manual integration is prohibited from use to achieve any specific numerical QC criteria; such as to reduce surrogate peak area in order to be within recovery limits. The use of manual integration to purposefully modify non-compliant data for this reason is prohibited, and will subject the analyst to immediate disciplinary action. Any questions should be referred to the QA Director or Technical Director. The analyst will further initial and date the manual integration on the quantitation report with the proper reason code per SOP No.110.0008.

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of the instrumentation and have performed a proficiency test with acceptable accuracy and precision results.

10.2 Method blanks. A method blank is an aliquot or volume of a clean reference matrix (reagent water for water samples, or Ottawa sand for soil/sediment samples) that is carried through the entire analytical procedure. A Method blank is prepared and analyzed with every batch of 20 samples or less. It is used to determine the level of contamination associated with the analytical processing and analysis of the samples.

10.2.1 Frequency of Method Blank: the Method Blank must be analyzed on the same instrument as its associated samples and QC samples.

10.2.2 The recovery of the surrogates must be within the calculated acceptance limits discussed in **Section 10.5**.

10.2.3 Acceptance Criteria for Method Blanks:

The concentration of target compounds in the method blank must be ≤1/2 CRDL or RL.

10.2.4 Corrective action for method blank contamination involves determining the source of the contamination and re-extracting the batch. In the event that the contaminant is not present in the all associated samples, only a sub group of samples will need re-extracting. See **Attachment 2** for corrective action.

10.3 Lab Control Sample (LCS) – A Lab Control Sample is a volume or weight of a clean reference matrix (organic-free water or Ottawa Sand) that is spiked with all single component target analytes and surrogates and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1 Acceptance criteria for LCS:

For most projects, Mitkem's in-house QC limits are used. There may be instances where specific project LCS limits may be used. This information is usually relayed through the project manager to the laboratory verbally or on the Mitkem workorder. To view the current in-house limits, use the SPEC option for the testcode in the LIMS.

The recovery of the surrogates must be within the calculated acceptance limits discussed in **section 10.5**.

The recovery is evaluated against the established in-house limits. For any analyte/analytes not meeting the criteria, corrective action must be taken. See **Attachment 2**. Also see DoD QC Requirements in **Attachment 1** for specific criteria for DoD projects.

For projects that require only a sub-set of target analytes reported, the numbers of allowance for marginal failure will be reduced as shown below. Outliers must be noted in the narrative.

Number of Analytes	Allowance of SMF
<11	0
11 – 30	1
31 – 50	2
51 - 70	3
71 – 90	4
>90	5

10.4 Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicate are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix at a frequency of one set per 20 samples.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and one duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform a matrix spike and matrix spike duplicate.

10.4.1 Acceptance criteria for Duplicate Matrix Spikes:

Matrix spike and matrix spike duplicates are used to assess the effect of matrix interferences on the analysis of the target analytes. Recoveries should be used as advisory guidelines to answer the question posed above.

Depending on sample matrix, chromatographic based analysis such as Method 8081B is susceptible to co-eluting interferences resulting in high biased values. Thus, the control limits for the duplicate matrix spikes should be used as guidelines. No re-extraction is required for matrix spike samples bases on the spike recoveries. However, corrective action is required. See **Attachment 2**.

RPD criteria are set at 30% for all matrices.

10.5 Surrogate recoveries - The recovery of each surrogate compound in all samples, blanks and LCS will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.5.1 Acceptance criteria - The current in-house acceptance criteria for each of the surrogate compounds in blanks, samples, duplicate matrix spikes and LCS are listed under the option SPEC for the LIMS Testcode.

In-house limits may be lab-generated or from a government /method based source such as DoD QSM. Please note that the in-house limit will be verified on an annual basis. Where lab-generated limits are derived, these will be updated annually.

Surrogate recoveries should meet recovery criteria for all analyses. See **Attachment 2** for corrective action.

Surrogate spikes in matrix specific samples that fail to meet the in-house limits would indicate potential matrix effect. In general, high recovery of surrogates due to co-eluting interference is acceptable. Low recovery of surrogates would indicate potential matrix related problems or one related to the extraction process. The analyst should confirm the sample volume extracted and the final extract volume to verify the calculations. If applicable, the analyst should consider additional cleanup procedures and re-analysis. If no appropriate cleanup procedure could be identified or re-analysis after additional cleanups failed to achieve the limit, this should be noted on the review checklist and also addressed in the narrative.

10.6 MDL studies are conducted to establish the limit of detection applicable to this method. MDL verification at approximately 1-4 x MDL is analyzed after the study which also sets the DoD QSM Limit of Detection (LOD). MDL verification (LOD) must be analyzed quarterly on each instrument used for DoD program work. Please refer to the SOP No. 80.0005 Determination of Method Detection Limits for more detail.

11. Data Validation and Reporting

All raw data, including calibrations, QC results, and samples results, is reviewed for technical accuracy and completeness by the analyst. The analyst initiates a project data review checklist and documents all comments regarding analysis there. Sample preparation logs, notebooks, and instrument logs are reviewed and signed by the laboratory supervisor. The QA Director randomly reviews 10% of the data reported by the laboratory.

All raw data is peer reviewed at the computer/Target level by another analyst or the lab supervisor prior to final form generation. Analysts generate all raw data and upload electronic data files to the LIMS for reporting. Raw data, including all support data (such as data review checklist, run logs, work order sheets, SDG summaries…) are brought to the data reporting department for assembly (as hardcopy or electronic PCL files). After assembly, all data is reviewed by senior personnel (data reviewers) for quality control and completeness dependent on project specific requirements.

12. Data Management and Records Management

12.1 Electronic data generated from the analysis of Pesticide 8081 extracts (calibrations, QC, samples) is saved and managed per SOP 110.0029 Electronic Data Management.

12.2 All analysis information is documented in the individual Instrument Run/Injection Logbook regardless of run acceptance. No injections are deleted from the sequence.

13. Corrective Action Procedures

Corrective Actions to be implemented in the event QC results are outside of the acceptance range are covered in **Sections 8, 9, and 10**. See **Attachment 2** for routine corrective action guidelines and documentation.

Discrepancy reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a discrepancy report for the purpose of identifying the appropriate corrective action is covered in Corrective Action Procedures SOP 80.0007. Enter the information directly into the LIMS corrective action log.

14. Health and Safety

The toxicity or carcinogenicity of each reagent used in the method has not been fully established. Each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is archived by the health and safety officer and is available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Health and Safety Plan.

In general, use gloves, a lab coat, and safety glasses when handling these reagents and work in a hood whenever possible. Spent vials and ampules are disposed of into a red metal drum in the Semivolatile Laboratory. See SOP No. 30.0024, Sample and Waste Disposal for more detail.

Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

15. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 (Waste Management) and 20.0 (Definitions, Acronyms, and Abbreviations) of the current Quality Assurance Plan.

16. References

1. U.S. Environmental Protection Agency. Organochlorine Pesticides by Gas Chromatography, Method 8081B, SW-846 Test Methods for Evaluating Solid Wastes, Final Update IV, Feb 2007.

2. "Methods Compendium for Inorganic and Organic Methods," United States Army Corps of Engineers, Appendix I, 2001.

3. "Shell for Chemical Analytical Requirements," United States Army Corps of Engineers, Appendix H, 2001 including addendum dated February 1, 2001.

4. "Quality Assurance Project Plan," Version 3.0, Air Force Center for Environmental Excellence, March 1998.

5. Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.1, April 2009 or current version.

6. HP reference manual and operation manual on HP 5890 Series II GC

Attachments:

1. **Figure 1**: List of Target 8081 Analytes.
2. **Figure 2**: List of Target TCLP Analytes.
3. **Figure 3**: List of Acronyms.

4. **Figure 4**: LIMS Standard Logbook
5. **Figure 5**: GC/ECD Instrument Run Logbook
6. **Figure 6**: INDC Standard Chromatogram and Quantitation Report.
7. **Figure 7**: Toxaphene Standard Chromatogram and Quantitation Report.
9. **Figure 8**: Technical Chlordane Standard Chromatogram and Quantitation Report
10. **Table 1**: RT window limits
11. **Attachment 1**: DoD QC Requirements
12. **Attachment 2**: Corrective Action Examples and Documentation Tables.

Figure 1 – List of Target Analytes for 8081

Pesticide	CAS Number	Reporting limits	
		Water (ug/L)	Soil(ug/Kg)
α-BHC	319-84-6	0.05	1.7
β-BHC	319-85-7	0.05	1.7
δ-BHC	319-86-8	0.05	1.7
γ-BHC (Lindane)	58-89-9	0.05	1.7
α-Chlordane	5103-71-9	0.05	1.7
γ-Chlordane	5103-74-2	0.05	1.7
4,4'-DDD	72-54-8	0.1	3.3
4,4'-DDE	72-55-9	0.1	3.3
4,4'-DDT	50-29-3	0.1	3.3
Aldrin	309-00-2	0.05	1.7
Dieldrin	60-57-1	0.1	3.3
Endosulfan I	959-98-8	0.05	1.7
Endosulfan II	33213-65-9	0.1	3.3
Endosulfan sulfate	1031-07-8	0.1	3.3
Endrin	72-20-8	0.1	3.3
Endrin aldehyde	7421-93-4	0.1	3.3
Heptachlor	74-44-8	0.05	1.7
Heptachlor epoxide	1024-57-3	0.05	1.7
Mirex	2385-85-5	0.05	1.7
Methoxychlor	72-43-5	0.5	17
Toxaphene	8001-35-2	5.0	170
Endrin ketone	53494-70-5	0.1	3.3
Technical chlordane *	12789-03-6	2.5	85

*Chlordane may be reported as a multi-component mixture in lieu of the alpha and gamma isomers.

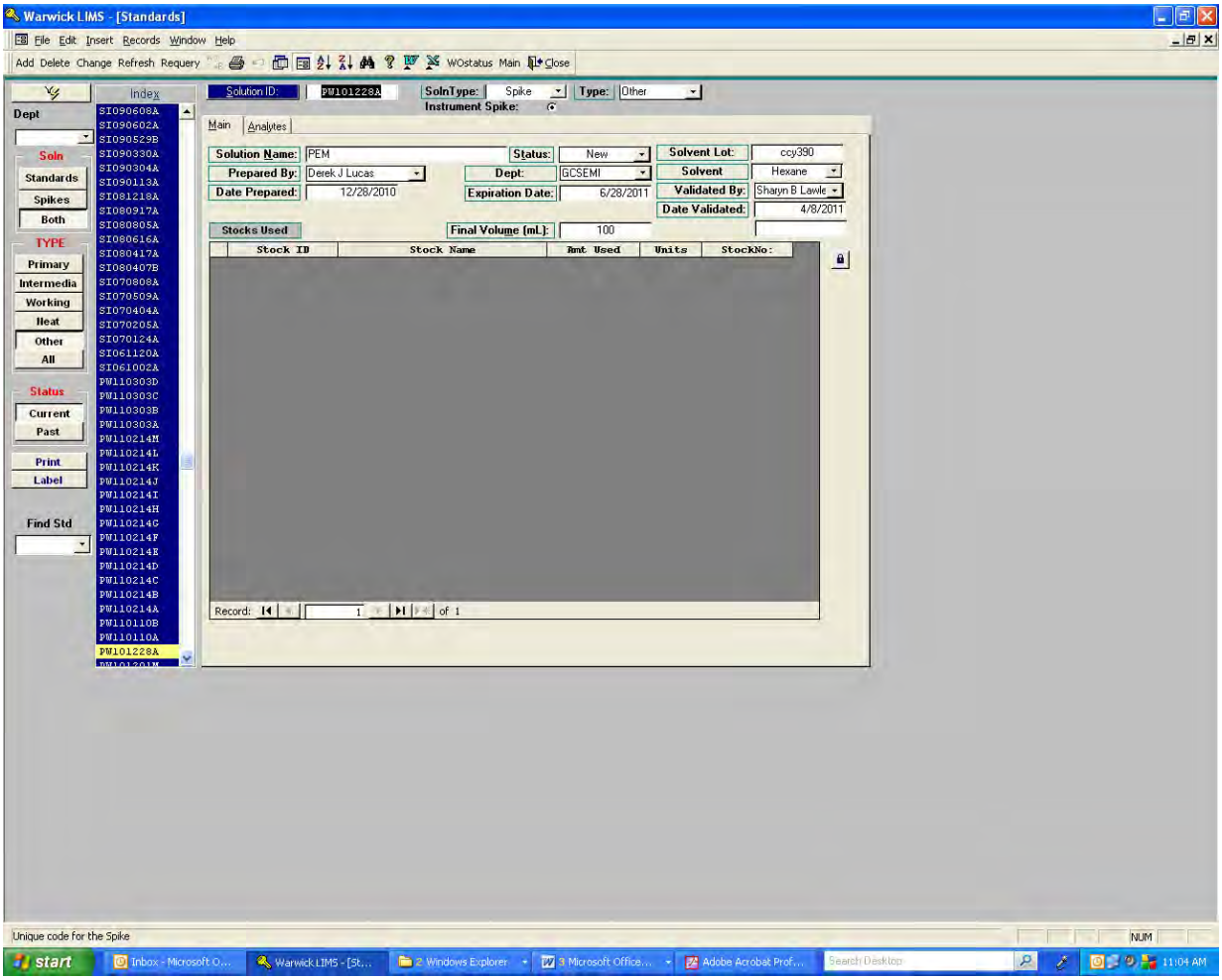
Figure 2 - TCLP Target analytes

Pesticide	CAS Number
γ-BHC (Lindane)	58-89-9
Endrin	72-20-8
Heptachlor	74-44-8
Heptachlor epoxide	1024-57-3
Methoxychlor	72-43-5
Toxaphene	8001-35-2
Technical chlordane	12789-03-6

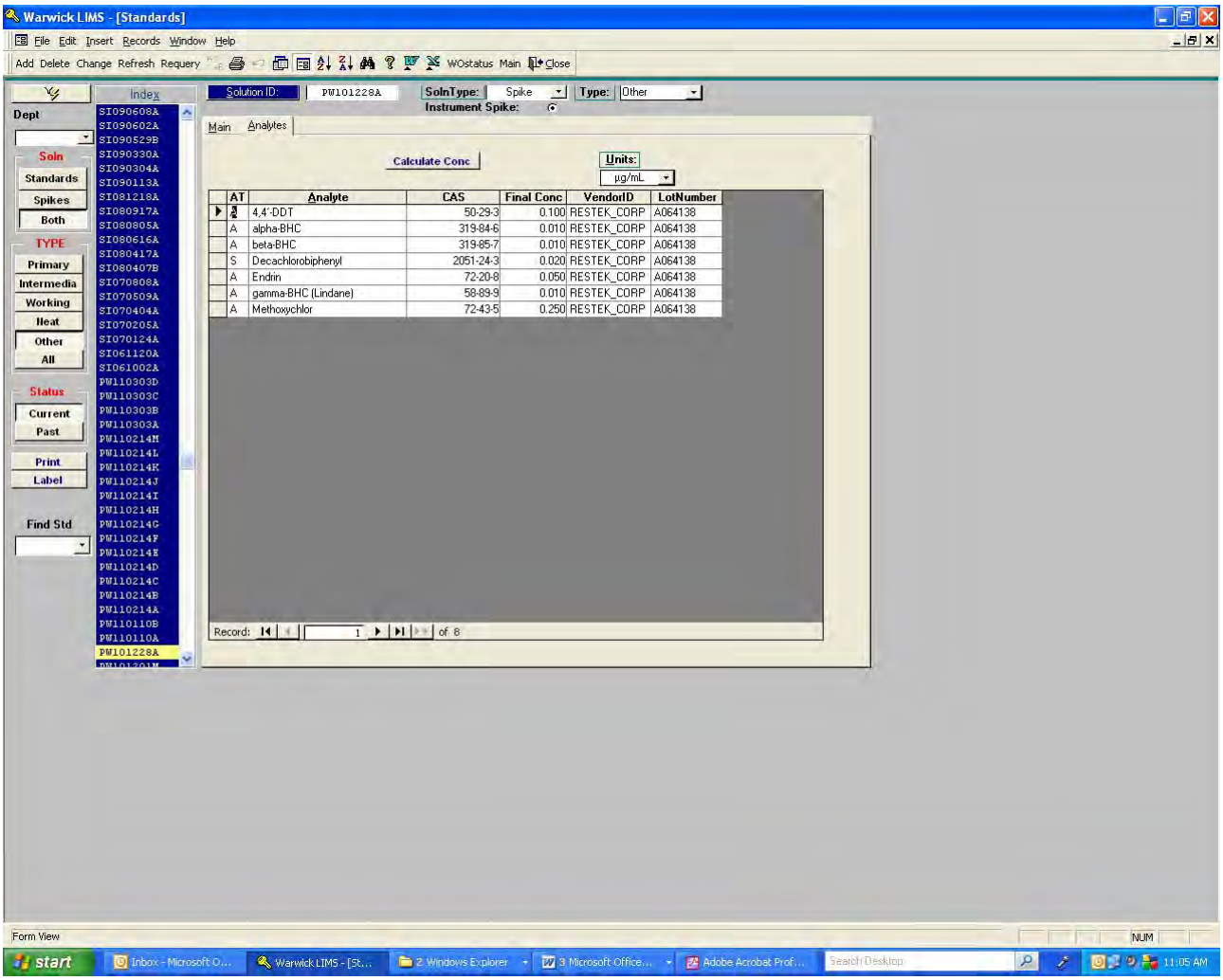
Figure 3 – List of Acronyms

RL	Reporting Limit
MDL	Method detection limit
MQL	Method quantitation limit
LCS	Lab control sample
MS	Matrix spike
MSD	Matrix spike duplicate
TCLP	Toxicity Characteristics of Leaching Procedure
SPLP	Synthetic Precipitate Leaching Procedure
GC	Gas chromatograph
ECD	Electron capture detector
EPC	Electron Pressure Controller
USACE	US Army Corps. Of Engineers
CLP	US EPA Contract Laboratory Program
AFCEE	Air Force Center for Environmental Excellence
USEPA	US Environmental Protection Agency
GPC	Gel Permeation Chromatography
PFE	Pressurized Fluid Extraction
ASE	Automated Sample Extraction
CF	Calibration factor
RSD	Relative Standard Deviation
CCV	Continuing Calibration Verification
ICV	Initial Calibration Verification
PEM	Performance Evaluation Verification
TOX	Toxaphene
TC	Technical Chlordane
DoD	Department of Defense
QSM	Quality Systems Manual

Figure 4
LIMS Standard Logbook



Example of Standard Logbook in LIMS: Main page



Example of Standard Logbook in LIMS: Analytes page

Figure 5
GC/ECD Instrument Run Logbook

MITKEM LABORATORIES - PEST/PCB RUN LOGBOOK: INSTRUMENT E5

Sequence _____ Method _____ ICAL Date _____

Date	Sample ID		Client ID	File Name		Dilution	F	R	Comments	Analyst

Standard ID's

_____ _____

_____ _____

_____ _____

_____ _____

Comments

_____

_____

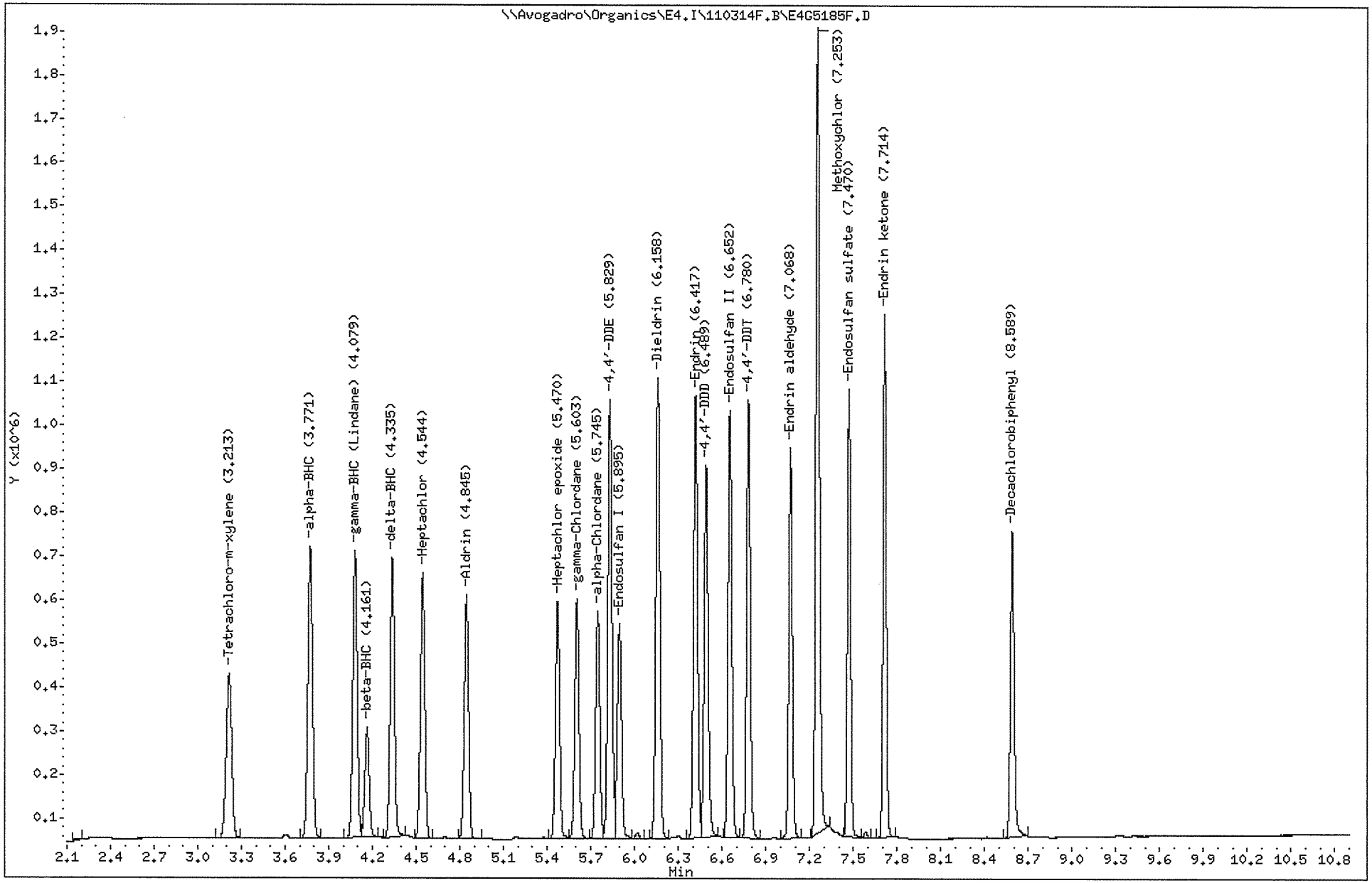
Logbook ID 60.0225-02/10 **Reviewed**_____

Figure 6

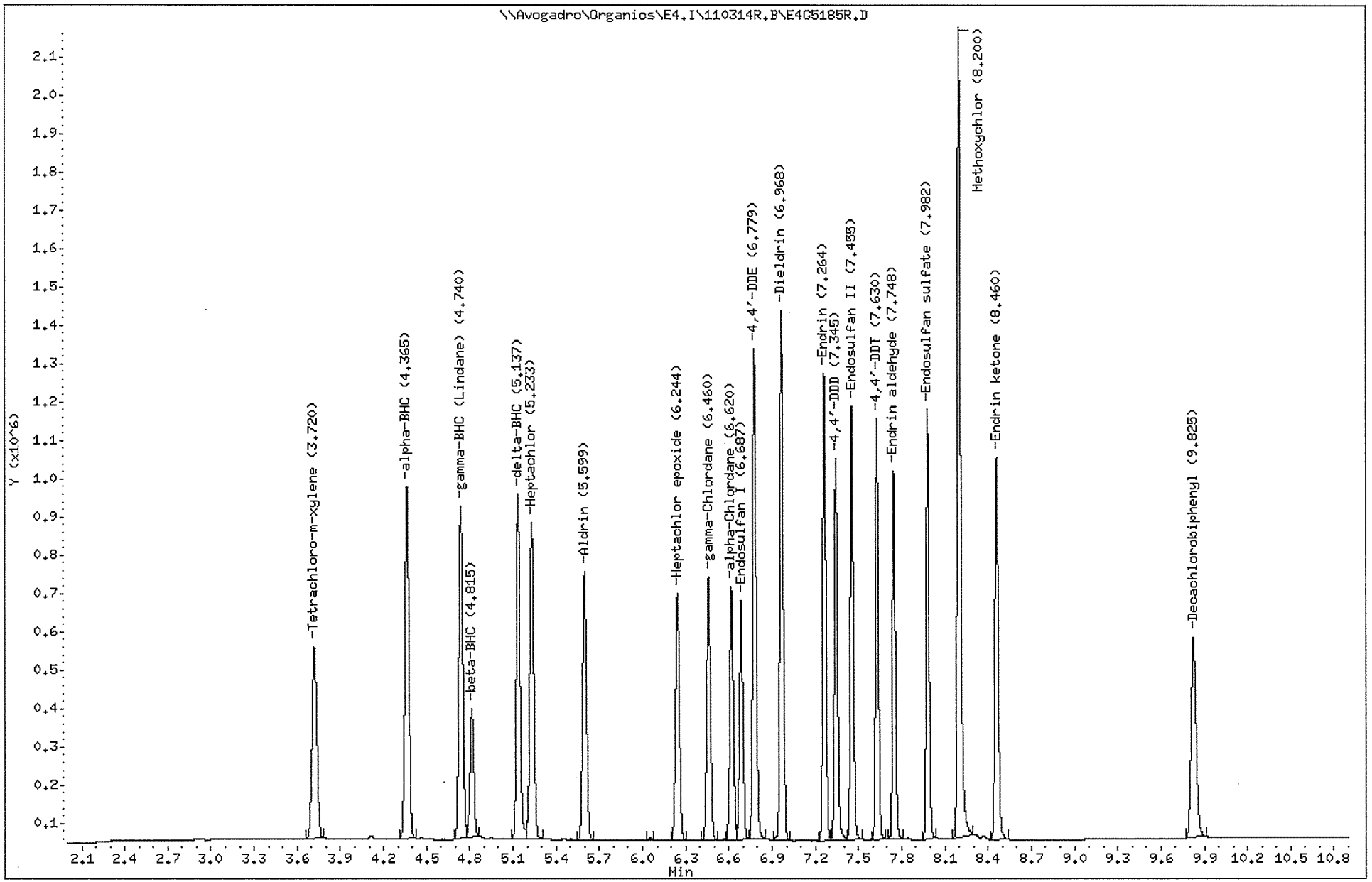
INDC Standard Chromatogram and Quantitation Report

Data File: \\Avogadro\Organics\E4.I\110314F.B\E4G5185F.D
Date : 14-MAR-2011 09:51
Client ID: INDC3VA
Sample Info: INDC3VA,INDC3VA,,indA.sub,,
Volume Injected (uL): 1.0
Column phase: CLPPest

Instrument: E4.i
Operator: DL SRC: DL
Column diameter: 0.53



Data File: \\Avogadro\Organics\E4.I\110314R.B\E4G5185R.D
Date : 14-MAR-2011 09:51
Client ID: INDC3VA
Instrument: E4.i
Sample Info: INDC3VA,INDC3VA,,indA.sub,,
Volume Injected (uL): 1.0
Operator: DL SRC: DL
Column phase: CLPPestII
Column diameter: 0.53



Data File: \\Avogadro\Organics\E4.I\110314F.B\E4G5185F.D
Report Date: 15-Mar-2011 13:54

Mitkem Laboratories

NYASP Pesticide Quantitation Report

Data file : \\Avogadro\Organics\E4.I\110314F.B\E4G5185F.D
Lab Smp Id: INDC3VA Client Smp ID: INDC3VA
Inj Date : 14-MAR-2011 09:51
Operator : DL SRC: DL Inst ID: E4.i
Smp Info : INDC3VA,INDC3VA,,indA.sub,,
Misc Info : 2,,,1
Comment :
Method : \\Avogadro\Organics\E4.I\110314F.B\E48081f.m
Meth Date : 15-Mar-2011 08:23 dlucas Quant Type: ESTD
Cal Date : 17-JAN-2011 21:28 Cal File: E4G4169F.D
Als bottle: 2 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: indA.sub
Target Version: 4.14 Sample Matrix: WATER
Processing Host: TARGET112

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi) * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (mL)
Vi	1.000	Volume injected (uL)
Cpnd Variable		Local Compound Variable

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 50-29-3			
3.212	3.212	0.000	1007296	0.02000	0.020			(a)
6 alpha-BHC					CAS #: 319-84-6			
3.771	3.769	0.002	664807	0.02000	0.021			(a)
7 gamma-BHC (Lindane)					CAS #: 58-89-9			
4.078	4.075	0.003	1382623	0.02000	0.021			(a)
10 beta-BHC					CAS #: 319-85-7			
4.161	4.157	0.004	525688	0.02000	0.019			(a)
11 delta-BHC					CAS #: 319-86-8			
4.334	4.331	0.003	1313082	0.02000	0.020			(a)
8 Heptachlor					CAS #: 76-44-8			
4.543	4.540	0.003	1259352	0.02000	0.021			(a)
9 Aldrin					CAS #: 309-00-2			
4.844	4.841	0.003	1149510	0.02000	0.021			(a)

Data File: \\Avogadro\Organics\E4.I\110314F.B\E4G5185F.D
Report Date: 15-Mar-2011 13:54

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	ON-COL (ng)	TARGET RANGE	RATIO	
14 Heptachlor epoxide						CAS #: 1024-57-3		
5.469	5.466	0.003	1099050	0.02000	0.021			(a)
16 gamma-Chlordane						CAS #: 5103-74-2		
5.602	5.598	0.004	549056	0.02000	0.020			(a)
17 alpha-Chlordane						CAS #: 5103-71-9		
5.745	5.742	0.003	1039187	0.02000	0.021			(a)
18 4,4'-DDE						CAS #: 72-55-9		
5.828	5.824	0.004	1005963	0.04000	0.041			(a)
15 Endosulfan I						CAS #: 959-98-8		
5.894	5.891	0.003	490869	0.02000	0.021			(a)
19 Dieldrin						CAS #: 60-57-1		
6.157	6.154	0.003	2071205	0.04000	0.042			(a)
20 Endrin						CAS #: 72-20-8		
6.417	6.412	0.005	1013589	0.04000	0.041			(a)
21 4,4'-DDD						CAS #: 72-54-8		
6.488	6.484	0.004	1614357	0.04000	0.041			(a)
22 Endosulfan II						CAS #: 33213-65-9		
6.652	6.648	0.004	1770665	0.04000	0.042			(a)
23 4,4'-DDT						CAS #: 50-29-3		
6.779	6.777	0.002	1771043	0.04000	0.045			(a)
24 Endrin aldehyde						CAS #: 7421-93-4		
7.067	7.065	0.002	894757	0.04000	0.043			(a)
26 Methoxychlor						CAS #: 72-43-5		
7.252	7.249	0.003	3293670	0.20000	0.20			(a)
25 Endosulfan sulfate						CAS #: 1031-07-8		
7.469	7.467	0.002	1574462	0.04000	0.043			(a)
27 Endrin ketone						CAS #: 53494-70-5		
7.713	7.712	0.001	1197441	0.04000	0.043			(a)
$ 2 Decachlorobiphenyl						CAS #: 2051-24-3		
8.588	8.586	0.002	1269741	0.04000	0.046			(a)

QC Flag Legend

a - Target compound detected but, quantitated amount Below Limit Of Quantitation(BLOQ).

Data File: \\Avogadro\Organics\E4.I\110314R.B\E4G5185R.D
Report Date: 15-Mar-2011 13:54

Mitkem Laboratories

NYASP Pesticide Quantitation Report

Data file : \\Avogadro\Organics\E4.I\110314R.B\E4G5185R.D
Lab Smp Id: INDC3VA Client Smp ID: INDC3VA
Inj Date : 14-MAR-2011 09:51
Operator : DL SRC: DL Inst ID: E4.i
Smp Info : INDC3VA,INDC3VA,,indA.sub,,
Misc Info : 2,,,1
Comment :
Method : \\Avogadro\Organics\E4.I\110314R.B\E48081r.m
Meth Date : 15-Mar-2011 08:27 dlucas Quant Type: ESTD
Cal Date : 17-JAN-2011 21:28 Cal File: E4G4169R.D
Als bottle: 2 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: indA.sub
Target Version: 4.14 Sample Matrix: WATER
Processing Host: TARGET112

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi) * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (mL)
Vi	1.000	Volume injected (uL)
Cpnd Variable		Local Compound Variable

				AMOUNTS			
				CAL-AMT	ON-COL		
RT	EXP RT	DLT RT	RESPONSE	(ng)	(ng)	TARGET RANGE	RATIO
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8		
3.720	3.725	-0.005	498142	0.02000	0.021		(a)
6 alpha-BHC					CAS #: 319-84-6		
4.364	4.369	-0.005	1758162	0.02000	0.022		(a)
7 gamma-BHC (Lindane)					CAS #: 58-89-9		
4.739	4.744	-0.005	1637787	0.02000	0.022		(a)
10 beta-BHC					CAS #: 319-85-7		
4.815	4.821	-0.006	610256	0.02000	0.021		(a)
11 delta-BHC					CAS #: 319-86-8		
5.137	5.144	-0.007	1572100	0.02000	0.022		(a)
8 Heptachlor					CAS #: 76-44-8		
5.232	5.239	-0.007	1566997	0.02000	0.022		(a)
9 Aldrin					CAS #: 309-00-2		
5.598	5.605	-0.007	1313076	0.02000	0.022		(a)

Data File: \\Avogadro\Organics\E4.I\110314R.B\E4G5185R.D
Report Date: 15-Mar-2011 13:54

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	ON-COL (ng)	TARGET RANGE	RATIO	
14 Heptachlor epoxide						CAS #: 1024-57-3		
6.243	6.252	-0.009	1196317	0.02000	0.022			(a)
16 gamma-Chlordane						CAS #: 5103-74-2		
6.460	6.468	-0.008	1210195	0.02000	0.022			(a)
17 alpha-Chlordane						CAS #: 5103-71-9		
6.619	6.628	-0.009	1108000	0.02000	0.022			(a)
18 4,4'-DDE						CAS #: 72-55-9		
6.778	6.786	-0.008	2069758	0.04000	0.045			(a)
15 Endosulfan I						CAS #: 959-98-8		
6.687	6.694	-0.007	621231	0.02000	0.022			(a)
19 Dieldrin						CAS #: 60-57-1		
6.967	6.975	-0.008	2148086	0.04000	0.045			(a)
20 Endrin						CAS #: 72-20-8		
7.263	7.273	-0.010	1219716	0.04000	0.042			(a)
21 4,4'-DDD						CAS #: 72-54-8		
7.344	7.351	-0.007	1513561	0.04000	0.043			(a)
22 Endosulfan II						CAS #: 33213-65-9		
7.455	7.463	-0.008	1647255	0.04000	0.045			(a)
23 4,4'-DDT						CAS #: 50-29-3		
7.629	7.637	-0.008	1518926	0.04000	0.046			(a)
24 Endrin aldehyde						CAS #: 7421-93-4		
7.747	7.755	-0.008	961563	0.04000	0.045			(a)
26 Methoxychlor						CAS #: 72-43-5		
8.199	8.205	-0.006	3385674	0.20000	0.22			(a)
25 Endosulfan sulfate						CAS #: 1031-07-8		
7.982	7.989	-0.007	1516632	0.04000	0.045			(a)
27 Endrin ketone						CAS #: 53494-70-5		
8.459	8.469	-0.010	999357	0.04000	0.042			(a)
$ 2 Decachlorobipheny1						CAS #: 2051-24-3		
9.825	9.839	-0.014	1249209	0.04000	0.044			(a)

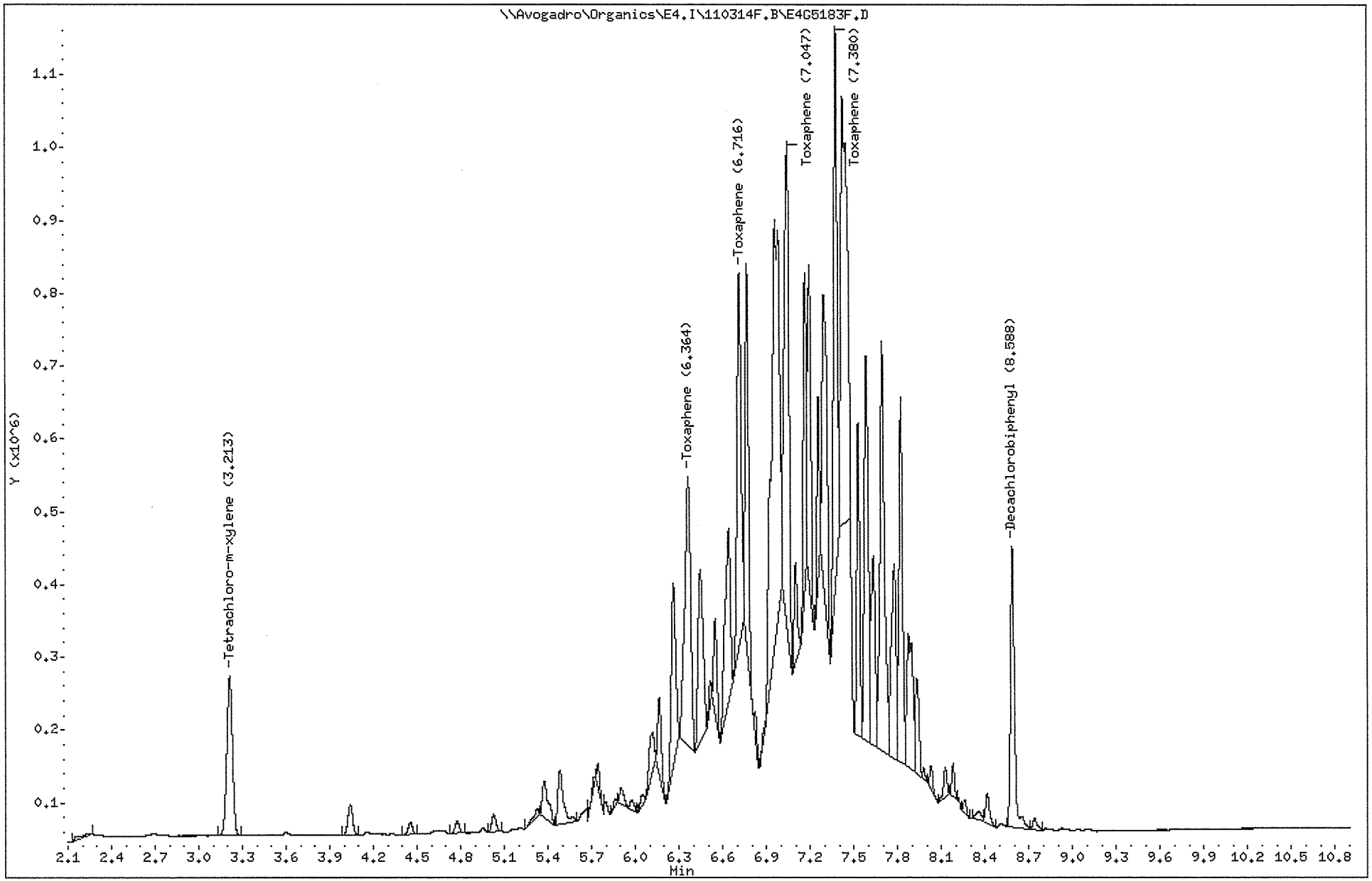
QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

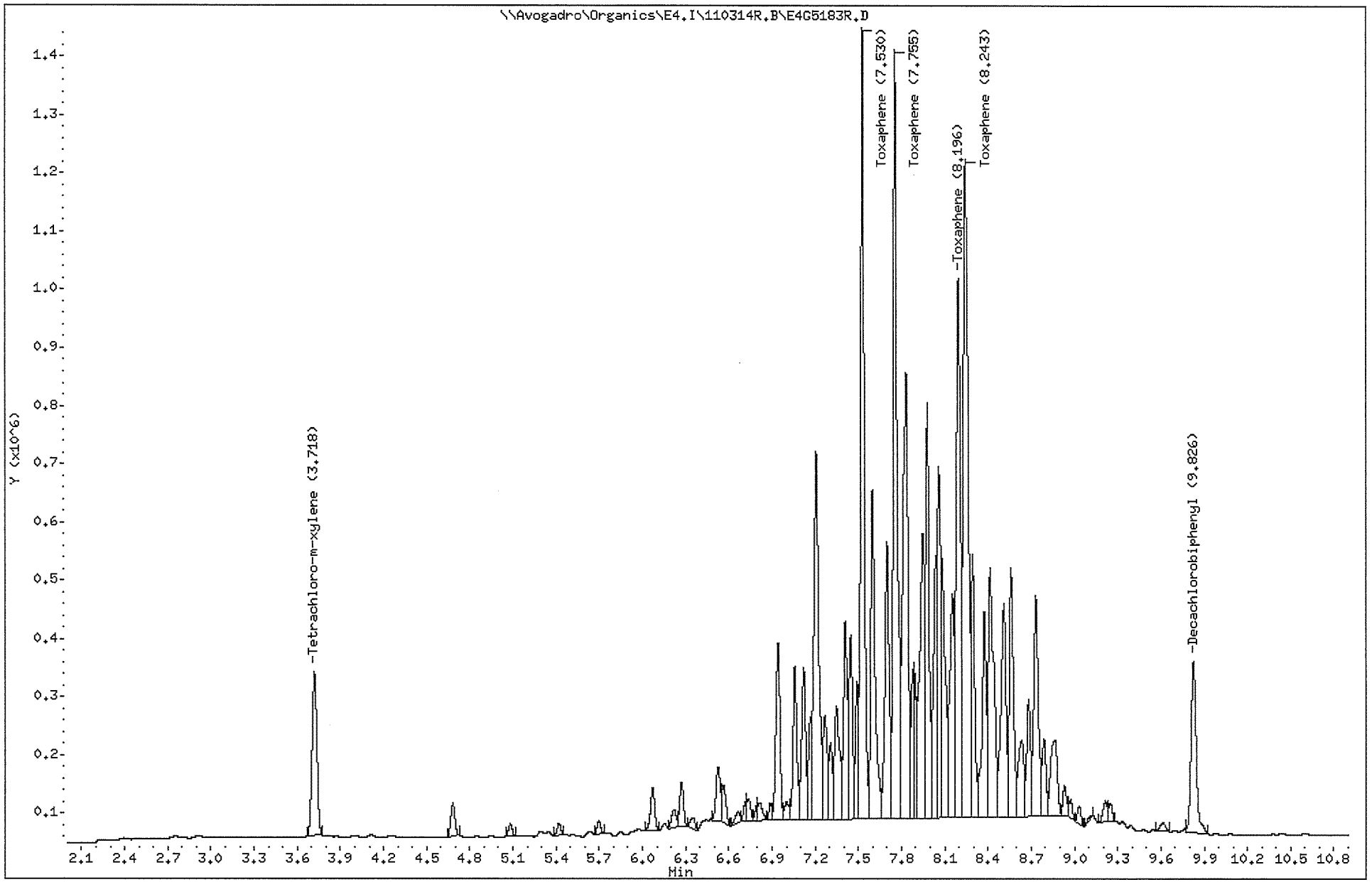
Figure 7

Toxaphene Standard Chromatogram and Quantitation Report

Data File: \\Avogadro\Organics\E4.I\110314F.B\E4C5183F.D
Date : 14-MAR-2011 09:22
Client ID: TOXAPH3VA
Instrument: E4.i
Sample Info: TOXAPH3VA,TOXAPH3VA,,SOMTOX.sub,,
Volume Injected (uL): 1.0
Operator: DL SRC: DL
Column phase: CLPPest
Column diameter: 0.53



Data File: \\Avogadro\Organics\E4.I\110314R.B\E4G5183R.D
Date : 14-MAR-2011 09:22
Client ID: TOXAPH3VA
Instrument: E4.i
Sample Info: TOXAPH3VA,TOXAPH3VA,,SOMTOX.sub,,
Volume Injected (uL): 1.0
Operator: DL SRC: DL
Column phase: CLPPestII
Column diameter: 0.53



Data File: \\Avogadro\Organics\E4.I\110314F.B\E4G5183F.D
Report Date: 15-Mar-2011 13:54

Mitkem Laboratories

NYASP Pesticide Quantitation Report

Data file : \\Avogadro\Organics\E4.I\110314F.B\E4G5183F.D
Lab Smp Id: TOXAPH3VA Client Smp ID: TOXAPH3VA
Inj Date : 14-MAR-2011 09:22
Operator : DL SRC: DL Inst ID: E4.i
Smp Info : TOXAPH3VA,TOXAPH3VA,,SOMTOX.sub,,
Misc Info :
Comment :
Method : \\Avogadro\Organics\E4.I\110314F.B\E48081f.m
Meth Date : 15-Mar-2011 08:23 dlucas Quant Type: ESTD
Cal Date : 17-JAN-2011 21:28 Cal File: E4G4169F.D
Als bottle: 98 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: SOMTOX.sub
Target Version: 4.14 Sample Matrix: WATER
Processing Host: TARGET112

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi) * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (mL)
Vi	1.000	Volume injected (uL)
Cpnd Variable		Local Compound Variable

				AMOUNTS				
				CAL-AMT	ON-COL			
RT	EXP RT	DLT RT	RESPONSE	(ng)	(ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 50-29-3			
3.213	3.212	0.001	584911	0.02000	0.012			(a)
$ 2 Decachlorobiphenyl					CAS #: 2051-24-3			
8.588	8.586	0.002	740068	0.04000	0.027			(a)
28 Toxaphene					CAS #: 8001-35-2			
6.364	6.362	0.002	370069	2.00000	0.98	80.00- 120.00	100.00	(a)
6.715	6.712	0.003	508480	2.00000	0.78	134.65- 174.65	137.40	
7.047	7.044	0.003	674109	2.00000	0.99	176.53- 216.53	182.16	
7.379	7.377	0.002	759060	2.00000	0.96	208.38- 248.38	205.11	
		Average of Peak Amounts =			0.92750			

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: \\Avogadro\Organics\E4.I\110314R.B\E4G5183R.D
Report Date: 15-Mar-2011 13:54

Mitkem Laboratories

NYASP Pesticide Quantitation Report

Data file : \\Avogadro\Organics\E4.I\110314R.B\E4G5183R.D
Lab Smp Id: TOXAPH3VA Client Smp ID: TOXAPH3VA
Inj Date : 14-MAR-2011 09:22
Operator : DL SRC: DL Inst ID: E4.i
Smp Info : TOXAPH3VA,TOXAPH3VA,,SOMTOX.sub,,
Misc Info :
Comment :
Method : \\Avogadro\Organics\E4.I\110314R.B\E48081r.m
Meth Date : 15-Mar-2011 08:27 dlucas Quant Type: ESTD
Cal Date : 17-JAN-2011 21:28 Cal File: E4G4169R.D
Als bottle: 98 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: SOMTOX.sub
Target Version: 4.14 Sample Matrix: WATER
Processing Host: TARGET112

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi) * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (mL)
Vi	1.000	Volume injected (uL)
Cpnd Variable		Local Compound Variable

			AMOUNTS				
				CAL-AMT	ON-COL		
RT	EXP RT	DLT RT	RESPONSE	(ng)	(ng)	TARGET RANGE	RATIO
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8		
3.718	3.725	-0.007	281608	0.02000	0.012		(a)
$ 2 Decachlorobiphenyl					CAS #: 2051-24-3		
9.825	9.839	-0.014	744707	0.04000	0.026		(a)
28 Toxaphene					CAS #: 8001-35-2		
7.529	7.538	-0.009	1358974	2.00000	1.3	80.00- 120.00	100.00(a)
7.754	7.764	-0.010	1319404	2.00000	1.3	75.78- 115.78	97.09
8.195	8.204	-0.009	927029	2.00000	1.3	43.55- 83.55	68.22
8.243	8.252	-0.009	1129599	2.00000	1.4	59.02- 99.02	83.12
		Average of Peak Amounts =			1.32500		

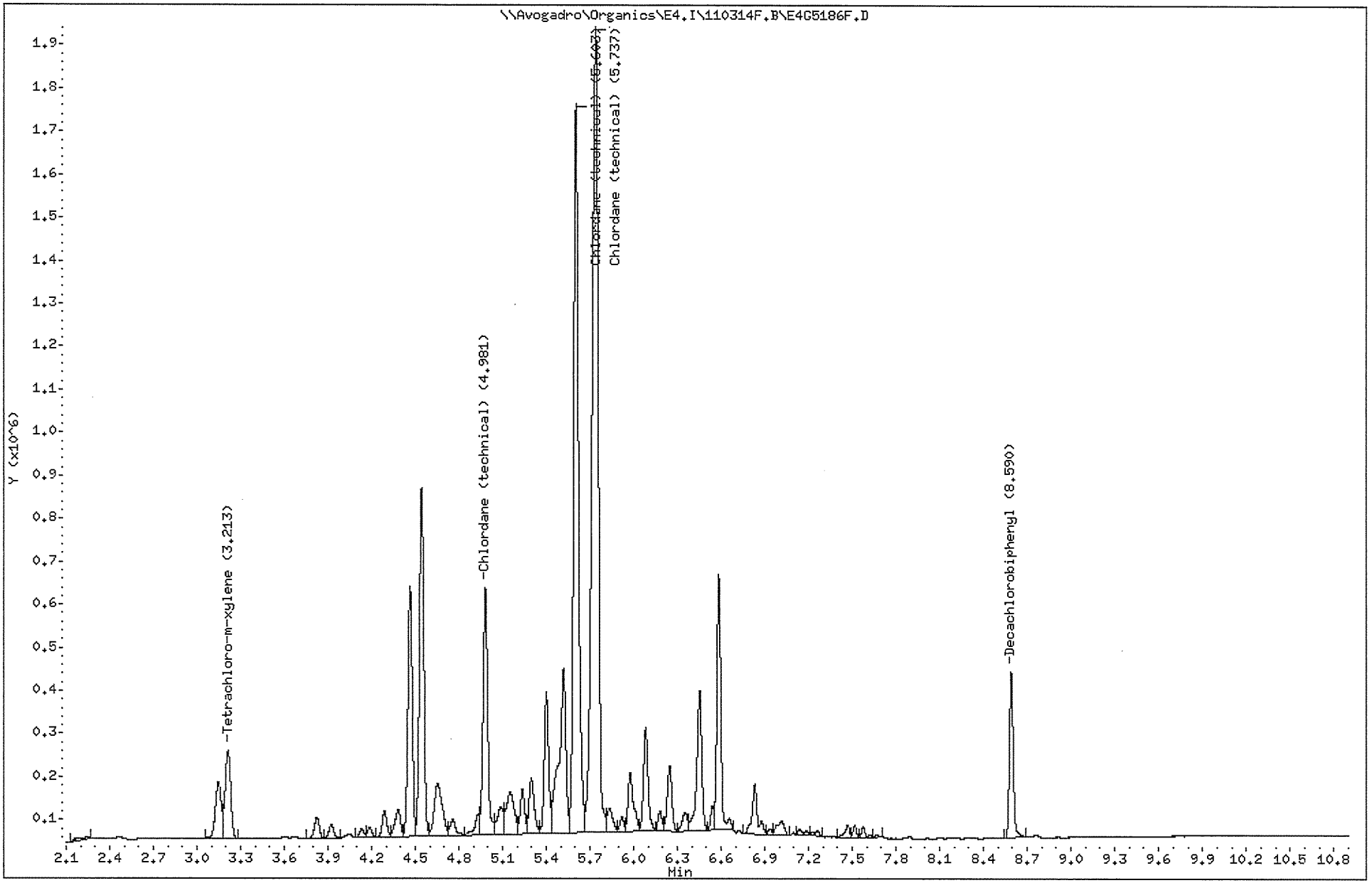
QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

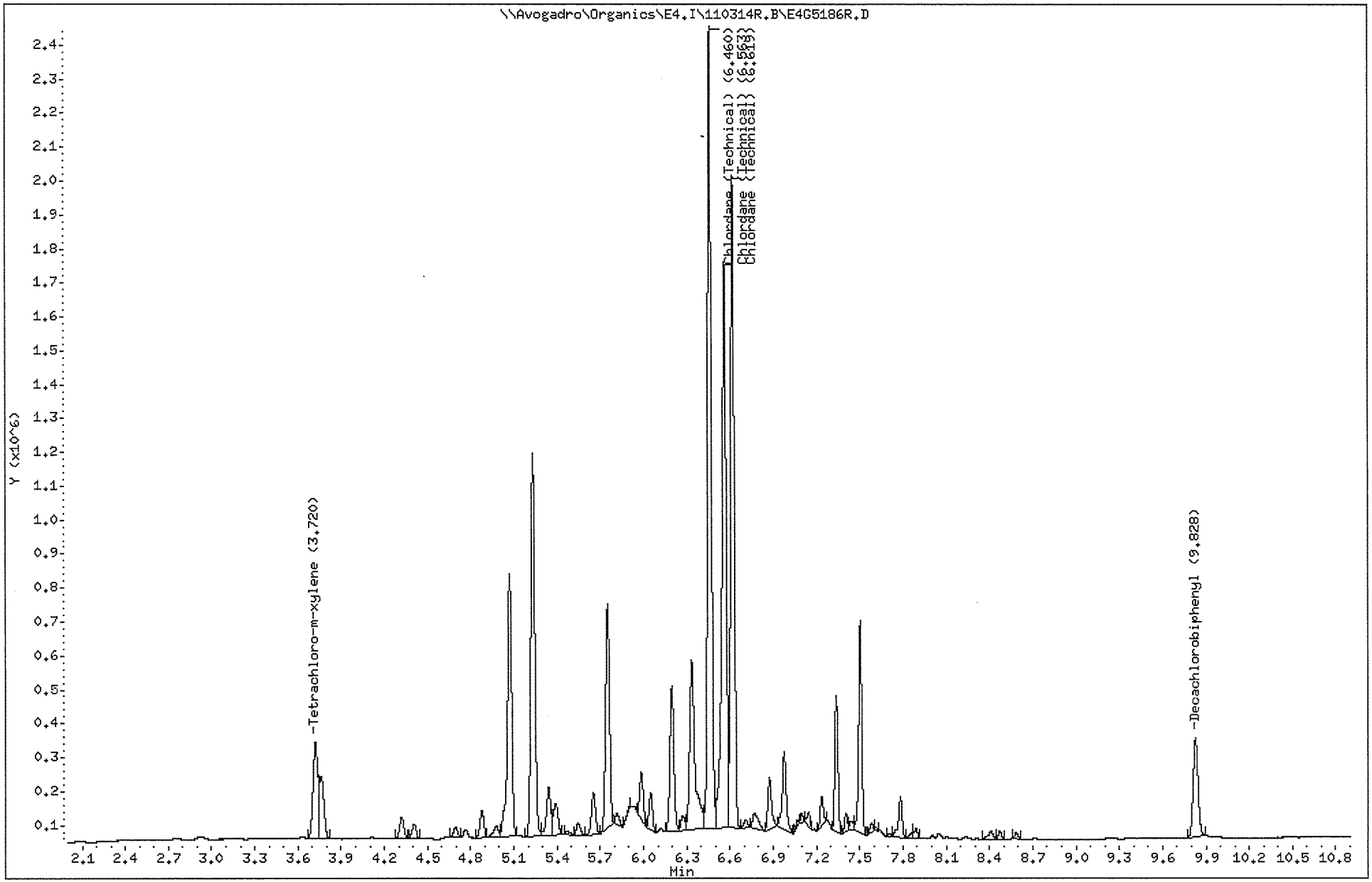
Figure 8

Technical Chlordane Standard Chromatogram and Quantitation Report

Data File: \\Avogadro\Organics\E4.I\110314F.B\E4G5186F.D
Date : 14-MAR-2011 10:06
Client ID: TC3VA
Instrument: E4.i
Sample Info: TC3VA,TC3VA,,CHLORDANE.sub,,
Volume Injected (uL): 1.0
Operator: DL SRC: DL
Column phase: CLPPest
Column diameter: 0.53



Data File: \\Avogadro\Organics\E4.I\110314R.B\E4G5186R.D
Date : 14-MAR-2011 10:06
Client ID: TC3VA
Instrument: E4.i
Sample Info: TC3VA,TC3VA,,CHLORDANE.sub,,
Volume Injected (uL): 1.0
Operator: DL SRC: DL
Column phase: CLPPestII
Column diameter: 0.53



Data File: \\Avogadro\Organics\E4.I\110314F.B\E4G5186F.D
Report Date: 15-Mar-2011 13:54

Mitkem Laboratories

NYASP Pesticide Quantitation Report

Data file : \\Avogadro\Organics\E4.I\110314F.B\E4G5186F.D
Lab Smp Id: TC3VA Client Smp ID: TC3VA
Inj Date : 14-MAR-2011 10:06
Operator : DL SRC: DL Inst ID: E4.i
Smp Info : TC3VA,TC3VA,,CHLORDANE.sub,,
Misc Info :
Comment :
Method : \\Avogadro\Organics\E4.I\110314F.B\E48081f.m
Meth Date : 15-Mar-2011 08:23 dlucas Quant Type: ESTD
Cal Date : 17-JAN-2011 21:28 Cal File: E4G4169F.D
Als bottle: 97 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: CHLORDANE.sub
Target Version: 4.14 Sample Matrix: WATER
Processing Host: TARGET112

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi) * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (mL)
Vi	1.000	Volume injected (uL)
Cpnd Variable		Local Compound Variable

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 50-29-3			
3.212	3.212	0.000	551241	0.02000	0.011			(a)
$ 2 Decachlorobiphenyl					CAS #: 2051-24-3			
8.589	8.586	0.003	703117	0.04000	0.026			(a)
29 Chlordane (technical)					CAS #: 12789-03-6			
4.981	4.977	0.004	1285952	1.00000	0.64	80.00- 120.00	100.00	
5.602	5.598	0.004	3908877	1.00000	0.65	282.82- 322.82	303.97	
5.737	5.732	0.005	5567167	1.00000	0.61	409.02- 449.02	432.92	
		Average of Peak Amounts =			0.63333			

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: \\Avogadro\Organics\E4.I\110314R.B\E4G5186R.D
Report Date: 15-Mar-2011 13:54

Mitkem Laboratories

NYASP Pesticide Quantitation Report

Data file : \\Avogadro\Organics\E4.I\110314R.B\E4G5186R.D
Lab Smp Id: TC3VA Client Smp ID: TC3VA
Inj Date : 14-MAR-2011 10:06
Operator : DL SRC: DL Inst ID: E4.i
Smp Info : TC3VA,TC3VA,,CHLORDANE.sub,,
Misc Info : 2,,,1
Comment :
Method : \\Avogadro\Organics\E4.I\110314R.B\E48081r.m
Meth Date : 15-Mar-2011 08:27 dlucas Quant Type: ESTD
Cal Date : 17-JAN-2011 21:28 Cal File: E4G4169R.D
Als bottle: 97 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: CHLORDANE.sub
Target Version: 4.14 Sample Matrix: WATER
Processing Host: TARGET112

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi) * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (mL)
Vi	1.000	Volume injected (uL)
Cpnd Variable		Local Compound Variable

			AMOUNTS				
				CAL-AMT	ON-COL		
RT	EXP RT	DLT RT	RESPONSE	(ng)	(ng)	TARGET RANGE	RATIO
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8		
3.720	3.725	-0.005	282129	0.02000	0.012		(a)
$ 2 Decachlorobiphenyl					CAS #: 2051-24-3		
9.827	9.839	-0.012	684670	0.04000	0.024		(a)
29 Chlordane (technical)					CAS #: 12789-03-6		
6.460	6.468	-0.008	2365987	1.00000	0.70	80.00- 120.00	100.00
6.562	6.570	-0.008	1666593	1.00000	0.69	51.84- 91.84	70.44
6.618	6.626	-0.008	1921385	1.00000	0.67	65.15- 105.15	81.21
		Average of Peak Amounts =			0.68667		

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Table 1:
SOM RT window limits

Retention Time Windows for Single Component Analytes, Toxaphene, and Surrogates

Compound	RT Window (minutes)
alpha-BHC	± 0.05
beta-BHC	± 0.05
gamma-BHC (Lindane)	± 0.05
delta-BHC	± 0.05
Heptachlor	± 0.05
Aldrin	± 0.05
alpha-Chlordane	± 0.07
gamma-Chlordane	± 0.07
Heptachlor epoxide	± 0.07
Dieldrin	± 0.07
Endrin	± 0.07
Endrin aldehyde	± 0.07
Endrin ketone	± 0.07
4,4'-DDD	± 0.07
4,4'-DDE	± 0.07
4,4'-DDT	± 0.07
Endosulfan I	± 0.07
Endosulfan II	± 0.07
Endosulfan sulfate	± 0.07
Methoxychlor	± 0.07
Toxaphene	± 0.07
Tetrachloro-m-xylene	± 0.05
Decachlorobiphenyl	± 0.10

Attachment 1
DoD QC Requirements

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Breakdown check (Endrin / DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 15% for both DDT and Endrin.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 15% for both DDT and Endrin.

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL; HPLC methods: All project analytes within ± 15% of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL; HPLC methods: All project analytes within ± 15% of expected value from the ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (with the exception of Method 8015).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column (see Box D-16).
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Attachment 2
Corrective Action and Documentation Examples

OCCURRENCE	ACTION	DOCUMENTATION
1. Initial calibration does not meet QC criteria (RSD% >20% or $r^2 < 0.99$).	1. Check GC conditions such as temperature program, makeup gas flow rate, and carrier gas flow rate. Check if column bleeding occurs and more injection maintenance is needed. After the reasons are found, appropriate maintenance will be done and a new initial calibration will be run. If the new curve still fails, the reasons for failing the curve will be re-evaluated and another a new curve will be rerun after the reevaluation. If still not good, call GC manufacturer.	1. Note in instrument run logbook, and if necessary notation in instrument maintenance log book. . If source determined to be bad standard solution, formal corrective action form must be initiated
2. Initial calibration verification (ICV) check does not meet QC criteria (D% > 20%).	2. Check the preparation of ICV standard, and if necessary, make a fresh ICV standard. If it is found that ICV standard is ok, the standards for initial calibration will be checked such as evaporation and the preparation of standards. A new ICV and a new curve will be analyzed after problems are corrected.	2. Note in instrument run logbook, and if necessary notation in instrument maintenance log book.
3. Continuing calibration verification check does not meet QC criteria (D% > 20%).	3. Maintenance such as replacing septum and liner for injection port will be done, and then a new set of CCVs and the samples related to the bad CCVs will be re-analyzed. If the CCVs still fail, A full maintenance including replacing septum, liner and gold seal and trimming the column will be done and a new initial calibration will be run. Then the CCV and/or QC and samples re-analyzed.	3. Note in instrument run logbook. If instrument maintenance performed, note in maintenance logbook.
4. Endrin and DDT breakdowns in PEM do not meet criteria	4. The septum and liner will be replaced, and new PEMs will be rerun. If PEM breakdowns still are out, the column will be trimmed. If PEM breakdowns still fail, new columns will be installed.	4. Notation in instrument run log book. If instrument maintenance performed, note in maintenance logbook.
5. Method blank contains target compound above ½ reporting limit.	5. A new aliquot of the blank from prep lab will be reanalyzed to check if contaminants are carried over from previous dirty samples and/or from standards or a bad injection occurs. If the results from re-analysis of the blank are still the same, whole batch of the samples that are related to the blank will be re-extracted and re-analyzed.	5. If the re-extraction/re-analysis meets the criteria, the results are reported. Document in the run log. If there is insufficient sample volume for re-extraction/re-analysis, report the initial results. Document in the run log, include a comment on the package checklist for the data reviewer and for inclusion in the narrative.

6. Surrogates in the method blank are outside of acceptable range.	6. A new aliquot of the blank from prep lab will be reanalyzed. If the results from re-analysis are the same, the samples that are related to this blank will be re-extracted and re-analyzed.	6. Same as documentation #5.
7. Compound out of acceptance range in laboratory control sample.	7. Investigate source of problem. If LCS recovery is above upper QC limit, and if analyte is not detected in associated samples, data may be flagged and reported. If LCS is not acceptable per method/SOP/project requirements, this LCS will be reanalyzed. If the results of reanalysis are the same, the samples that are related to the LCS will be re-extracted and re-analyzed.	7. Flag all compounds out of range on Form 3 of data report, if samples are re-analyzed within holding times, note in instrument run logbook. If samples are beyond holding time and both sets of data are to be reported, note in instrument run logbook, comment in data review checklist to be included in project narrative, and document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer. If re-analysis cannot be performed due to insufficient sample, comment in data review checklist to be included in project narrative, and document as above.
8. Surrogates in samples are out of control limits.	8. 8.1 No actions are needed for the following situations: • One of the surrogates (TCX and DCB) is in control limits, and the other is above the upper limit due to coelution with contaminants. • TCX is in the control limits, and DCB is below the lower limit. This is possibly due to matrix effect. 8.2 Further actions are needed for the following cases: • Both of the surrogates (TCX and DCB) do not meet criteria. • TCX is lower than the lower limits and DCB is in the limits. This is most possibly due to evaporation of TCX in the extracts. Actions for 8.2: The samples will be reanalyzed. If the results of re-analysis are the same, the samples will be re-extracted and re-analyzed.	8. If only re-analysis is reported, note in instrument run log. If both sets of data are to be reported, note in instrument run log, re-extraction request form, preparation logbooks, and comment on data review checklist to be included in project narrative, flagging all non-compliant values on Form 2 of data report.
9. Compound in sample exceeds upper calibration standard concentration.	9. Re-analyze sample at dilution. If calibration limit exceedence is the only QC problem, report both initial and dilution analyses. If initial analysis has multiple QC problems, evaluate further to determine if initial run is to be reported (often this cannot be determined until the results of the dilution are evaluated). Instrument must be shown to be free of carryover contamination prior to acceptable analysis of next sample. If running an instrument using auto-sampler, evaluate following sample. If following sample contains less than reporting limit of compound, the analysis is valid, and no instrument blank is required. If following sample(s) contain compound (typically in decreasing concentration—carryover typically occurs at 1% of concentration of high sample in following analysis, with effect more pronounced for later-eluting compounds. Effected samples must be re-analyzed if sufficient volume exists.	9. Note in instrument run log, comment on data review checklist to be included in project narrative. 10. If only re-analysis is reported, note in instrument run log. If both sets of data are to be reported, note in instrument run log, re-extraction request

10. Instrument blank (GC) contains contamination above QC criteria.	10. Investigate source of problem, decontaminate purge and trap instrument, re-analyze blank and all effected samples.	form, preparation logbooks, and comment on data review checklist to be included in project narrative.
11. Matrix spike recovery out of QC range.	11. Evaluate problem. If duplicate spike (MSD) shows same effect, it is generally matrix interference. If concentration of spike analyte is significantly (approx. 4 times) greater in un-spiked sample, this is matrix interference masking quantitation of spike concentration. If source cannot be determined, re-analyze spike sample.	11. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.
12. Duplicate (or MSD) relative percent difference exceeds QC limit.	12. Evaluate problem. If concentration of analyte is close to reporting limit, variation of analysis is acceptable. If sample is soil or other heterogeneous matrix, high RPD is typical. If sample is a typically homogeneous matrix, re-analyze duplicate sample.	12. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative. Flag RPD on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.
13. Retention Time shift.	13. A. No further action is required if the retention times of the CCV and samples shift after regular maintenance such as replacing the septum, liner, gold seal and trimming of the columns. The retention times will be established. B. If the retention times of the CCV shift during analysis, GC maintenance will be performed and the CCV and associated samples and QC will be re-analyzed. C. If the retention times of the CCV do not shift, but the retention times of surrogates in some of the samples do shift, the samples will be re-analyzed. If the retention times still do not meet the criteria, matrix affect is assumed and the results will be reported. This situation will be documented.	13. Document in the run log. If CCV, QC, and samples were re-analyzed, document in the run log. If the retention times shift with respect to re-analysis, document in the run log, in the checklist for the case narrative.

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8270D

Contents SOP NO. 70.0011

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		7/21/11
Quality Assurance Director		7/20/11
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	Analyst	7/7/12			

Revision Record

Revision Date	Revision Description	Comments	Initials
5/29/07	Reference to section 10.5 changed to 10.6 on page 26		SBL
6/27/07	Added requirement of 10 % surrogate recovery	Not valid for phenol-d5 in water	SBL
3/13/08	Added “x 100” to breakdown calc, lab name change	Per Navy data audit 2008.	SBL
4/28/08	Expanded state program disclaimer to include DoD.	Per Navy data audit 2008. Unable to use 8270D calibration criteria	SBL
4/28/08	Removed letter suffix of prep methods so updates will not require multiple SOP		SBL
11/7/08	Revised LIMS table and clarification of Table 3=Method Table 5	Per NJ Audit	SBL
5/18/09	Include Add-on cmpds in standard prep, 12month stock/working std exp date.	Removed COA, figure 1	SBL
11/24/09	QSM4.1 ADDED		
12/23/09	ICV 80-120 per QSM4.1 now		SBL
10/20/10	Reworded section		SBL
7/1/11-7/18/11	Added S6, revised some language, surrogate info,added ASE. Lower calibration range and MI documentation	Lab name change. Full revision	SBL
8/31/11	Added soil mdl/rl print out	Minor, per NJDEP	SBL
6/7/12	Removed S2 from instrument list	Minor	SBL

Procedure Superseded By _____ Date: _____

Procedure Discontinued By _____ Date: _____

Procedure Archived By _____Date: _____

Spectrum Analytical, Inc
Featuring Hanibal Technology
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8270D

Rev. 11

	Signature	Date
QA Director:		7/20/11
Lab Director:		7/20/11
Effective Date:	7/27/11	

Spectrum Analytical, Inc
Featuring Hanibal Technology
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8270D

Rev. 11

1. Scope and Application

This SOP describes the analysis of semivolatile organic compounds in solvent extracts of aqueous and soil samples using gas chromatography/mass spectrometry (GC/MS). The SOP covers the analyses according to protocols discussed in USEPA SW-846 Update IV, Method 8270D.

This SOP meets all of the requirements specified in the method. Where applicable, additional requirements for the US Department of Defense (DoD) are also included.

To further familiarize with the procedures, the analyst is encouraged to consult the following instrument manuals and SOP:

- Department of Defense Quality Systems Manual for Environmental Laboratories.
- Hewlett Packard HP 5972A MSD Hardware Manual.
- Spectrum RI Organic Prep Lab SOPs for Semivolatile sample extraction.

All sample matrices must be extracted and concentrated prior to analysis.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors** or Peer analysts review the logbooks and data generated from this procedure and approve all reported results. The **Laboratory Director** or a member of senior management evaluates all laboratory reports for reasonableness of the results and signs the reports. The **QA Director** reviews all quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure/Instrumentation

3.1 The samples are extracted using appropriate sample extraction methods (see separate SOPs for sample extraction) and, if necessary, sample clean-up procedures.

3.2 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 connected to the gas chromatograph.

3.3 Analytes eluted from the capillary column are introduced into the Mass Spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron-impact- spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a minimum of a five-point calibration curve.

3.4 A list of acronyms used in this SOP is included in **Table 1**.

3.5 The list of compounds to be analyzed and reported may vary from project to project. The SW-846 method contains several different lists of analytes, so there is no "official" EPA list of Method 8270D compounds. Spectrum RI typically analyzes samples for and reports a fairly extensive list of target analytes. Certain projects may also have additional compounds not on the normal list. Alternatively, certain projects may have a shorter list of compounds than the normal list. These project-specific lists of analytes are specified by the client through discussion with the Spectrum RI Project Manager who discusses the list with the Laboratory Supervisor. The lists are handled in the laboratory by the use of "sublists" in the Target data reduction and reporting software. In addition, when utilizing the LIMS system, the sublist can be viewed using the SEL list option. SEL refers to the select list of target analytes requested by the client. It is used when this list differs from the "routine" analyte list. The list of Method 8270D compounds routinely analyzed by this method is presented in **Table 2**. Refer to the LIMS Test Information category/Test option/ limits of the test code, for the most current MDL values. Those listed in **Table 2** may not be the most up to date.

3.5.1 Several options exist for the reporting extra compounds that are not on the routine list. The ideal approach includes purchasing a primary calibration standard and a second source check standard. This is followed by the determination of method detection limits and inclusion of the extra compounds to the initial calibration standards as well as calibration verification standards and laboratory control spiking standards. Depending on the clients' needs, alternate approaches may be appropriate. This may include single point calibration or searching for the compound as a "Tentatively Identified Compound" using Target software's library search routines. The approach taken must be discussed with the client prior to analyses, and if needed, sufficient documentation is included in the analysis report to enable validating the data. The analyst will be instructed by the lab supervisor as to what documentation is needed and what is required to be sent to the data reporting area for inclusion in the final report.

3.5.2 The Quality Control requirements contained in this SOP apply to the specific list of analytes being reported. QC criteria are to be evaluated for all project target analytes. While QC issues with non-routine analytes should be investigated, they are not critical if the compound is not reported. Spectrum RI's calibration standards and LCS/MS spiking solutions may contain additional compounds that are not reported for a particular project.

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. In some instances, clients will provide their own containers. For semivolatile organic compound analysis by Method 8270D, water samples are collected in 1-liter amber glass bottles. Solid samples are collected in 8-ounce amber glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 Sample extracts are transferred to the semivolatile organic analysis lab with appropriate sample preparation information. Extracts are stored at 4° ± 2°C, protected from light, in sealed crimp cap vials equipped with unpierced PTFE-lined septa and stored in a separate location from the analytical standards.

4.3 Extract hold-time for semivolatile organic compound analysis by Method 8270D is 40 days from date of sample extraction. The sample preparation holding times are covered in the corresponding extraction procedures SOPs.

5. Interferences and Potential Problems

5.1 Evaluate the raw GC/MS data to verify that interferences were not introduced during the extraction and/or clean up of the samples. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed with solvent between sample injections. Whenever a highly concentrated (compounds detected at 200x MRL) sample is encountered, the subsequent sample must be evaluated for possible contamination. Presence of similar analytes in the subsequent sample(s) will require reanalysis of these samples to establish that the analytes were not the result of contamination.

5.2 Method 8270D is not appropriate for multi-component analytes (Aroclors, toxaphene, chlordane, etc.). Refer instead to Methods 8081 and 8082.

5.3 The following compounds may require special treatment when being determined by this method:

5.3.1 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the GC, chemical reaction in acetone solution, and photochemical decomposition.

5.3.2 N-nitroso-di-methylamine and pyridine are difficult to separate from the solvent under the chromatographic conditions described; the filament should be turned on early enough to detect these compounds.

5.3.3 N-nitroso-di-phenylamine decomposes in the GC inlet and cannot be separated from diphenylamine.

5.3.4 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 injection port is not properly maintained, or if reactive samples are analyzed previously in the sequence.

5.3.5 In the presence of samples containing residual chlorine, phenol-d6 has been known to react to form chlorinated phenolic compounds that are not detected as the original spiked surrogate. Sample preservation precautions outlined in Chapter Four should be used when residual chlorine is known to be present in order to minimize degradation of deuterated phenols or any other susceptible target analyte.

6. Equipment and Apparatus

6.1 Equipment:

6.1.1 There are four GC/MS in the semivolatile organic analysis lab. The instruments used in the laboratory include two HP Model 6890 GCs interfaced to HP Model 5973 MSs, one Hewlett Packard (HP) Model 7890A GC interfaced to a HP Model 5973 MS and one Hewlett Packard (HP) Model 7890A GC interfaced to a HP Model 5975 MS. IBM-compatible PCs with EnviroQuant Software are used to handle data acquisition. The resultant data are processed using Target software from ThruPut Corporation. Please note that while HP's instrumentation division has been renamed Agilent Corporation, all of the instruments are referred as HP in this SOP.

6.1.2 The HP gas chromatographs used are fitted with electron pressure controller (EPC) to allow constant carrier gas flow during temperature ramping.

6.1.3 A 30m x 0.25mm ID (0. 25 um film thickness) Rxi-5SilMS fused silica capillary columns (Restek) are used for the analyses.

6.1.4 HP Model 7683 auto-samplers are used for sample injection for 6890 GCs. A CTC leap autosampler is used for sample injection for HP 7890A GC, and a HP Model 7693 auto-sampler is used for the other HP 7890A GC.

6.1.5 Instrument operating conditions are as follows:

Please note that the above are *general* instrument conditions and may be modified to respond to specific project needs. In the event that these conditions are changed, Enviroquant Data Acquisition methods containing the actual GC operating conditions are copied and sent to the network along with all GC/ECD raw data files. They are located in a folder in the sequence batch called "Zacq".

General Gas Chromatography Conditions

Carrier Gas	Helium (99.999%)
Column Flow	about 1 mL/minute
Injector Temperature	275 to 295 °C
Transfer Line Temperature	290°C
Injection Volume	1 µL

General Mass Spectrometry Conditions

Mass Range	35-500 AMU
Scan Speed	at least 1 scan per second
Ionization Mode	70 eV positive ion

GC/MS program for DFTPP tune analysis:

DFTPP

GC/MS Program for Calibration Standards, Blanks, LCS/LCSD, MS/MSD and sample analysis:

BNA

6.2 Maintenance - The semivolatile GC/MS are maintained according to the manufacturer's recommendation. The lab analyst performs preventive maintenance as discussed below.

6.2.1 On a daily basis whenever analyses are to be performed, replace the GC septum and clean the injection port and liner, as well as the gold seal. After prolonged use, or after an analytical sequence in which high concentrations of target compounds are detected, it is at the analysts' discretion to evaluate the condition of the injection liner and gold seal to determine if they require replacement. Also clip up to 6" of the column. All preventative/routine maintenance is recorded in the associated Instrument Run Log.

6.2.2 If needed, the analytical column may be replaced; this is usually indicated by tailing of the polar compounds such as pentachlorophenol/benzidine, and/or when initial and continuing calibration verifications repeatedly fail to meet method requirements (especially for polar acidic or basic compounds). This type of maintenance is recorded in the Instrument Maintenance Logbook. The Instrument Maintenance is located in the LIMS system and can be accessed using the category Analytical and option Instruments. All analysts have access to this function in LIMS. If help is needed, ask the Lab Supervisor for assistance. Document the manufacturer name and lot # of the new column in the LIMS Instrument maintenance log. The certificate for the column may be given to the QA Director for filing or may be scanned.

6.2.3 If the system constantly drifts out of DFTPP tune and/or the initial method requirements, the ion source will need to be cleaned. This maintenance will need to be recorded in the LIMS Instrument Maintenance Logbook.

6.2.4 There are two filaments in the mass spectrometer. If both filaments are blown, the HP 5972A MSD will be vented to replace both filaments. Whenever the ion source is opened for maintenance, the analyst should make sure both filaments are replaced. This would allow uninterrupted operation even if one filament were blown. This maintenance will need to be recorded in the LIMS Instrument Maintenance Logbook.

NOTE: After major maintenance such as the scenarios described in **sections 6.2.2 through 6.2.4**, an Initial Calibration (ICAL) is analyzed. Document the date of the ICAL in the resolution field in the LIMS Maintenance Logbook.

6.2.5 The rough-pump oil should be replaced at least once a year, or as needed. Check the oil level periodically and add oil if needed. Document this maintenance as above.

6.2.6 Once a year, all GC/MS systems may undergo extensive maintenance by a skilled technician. When this occurs, collect all associated paperwork and enter relevant information in the LIMS maintenance log. The paperwork is scanned for .pdf inclusion on the server.

6.2.7 Corrective maintenance is needed if the lab analyst or his/her supervisor fails to diagnose and/or correct the problem. The analyst or lab supervisor will promptly notify the instrument vendor for telephone-consultation and if needed, schedule on-site repair. This information should be documented as in **Section 6.2.6**. In addition, the resolution field in the LIMS Maintenance Logbook should be filled in fully. Enter your initials in the maintenance log entries; do not use administrator or other non-unique identification. Also refer to SOP 110.0040 Instrument Maintenance and Documentation for additional information.

6.3 Troubleshooting - Refer to troubleshooting section of the HP 5972A MSD hardware manual.

6.4 Glassware

6.4.1 Hamilton syringes (10µls, 25µls, 100µls, 250uls, 500µls, and 1000µls). The manufacturer certifies syringe accuracy to ±1%.

6.4.2 Volumetric flasks.

6.4.3 Vials – 2ml and 15ml glass with PTFE-lined screw cap or crimp-cap tops.

6.4.4 Mini-inert vials with on/off valve.

6.4.5 Crimper tool.

7. Reagents and Standards

7.1 Organic solvents – pesticide residue analysis grade methylene chloride (or equivalent) for standard preparation. Solvents are available to the GC/MS lab in 1-gallon liter bottles or in a

smaller volume from the Organic Preparation Lab bulk storage distribution line. Always verify that the lot/serial number of the solvent has been approved before use. Check the server for Agawam's solvent check on same lot. See QA for more information.

7.2 The standards used for this SOP are discussed below. ***Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials***.

7.3 The laboratory will at all time archive or have on order one complete set of unopened ampulated standards (to include internal standards, surrogate standards and target analyte standards).

All primary standards received from vendors are logged into the LIMS Standard Logbook. The standards are labeled *SPyymmddX*,

Where: SP = Semivolatile Primary Standard
yymmdd = date the standard is received
X = the order the standard is logged into the Logbook on that date, in increasing alphabetical order.

7.4 Tune Standard: the tuning standard contains DFTPP, 4,4'-DDT, pentachlorophenol and benzidine. It is purchased from Absolute Standards (Cat. No. 43032) at 1000 ug/mL.

7.5 Internal Standard: the internal standard is obtained from Cambridge Isotope as neat compounds. A 2000ug/mL solution of all compounds (1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthalene-d10, Phenanthrene-d10, Chrysene-d12, and Perylene-d12) is prepared and transferred into 2mL vials marked at the meniscus.

7.6 Primary Calibration Standards:

- 8270 MEGA Mix (Restek, Cat. No. 31850) at 500-1000ug/mL
- Benzidine Mixture (Ultra Cat. No. EPA-1071) at 5000ug/mL
- Acid surrogate (Restek Cat. No. 31087) at 10,000ug/mL
- B/N surrogate (Restek Cat. No. 31086) at 5000ug/mL
- 8270 add-on compounds, neat: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine. (Sigma Aldrich Cat. Nos. B1334, C2204, B34656, A10701 and Supelco Cat. No. 49085).
- Additional special project compounds such as 1,4-dioxane, 2-ethyl-1-hexanol, 2-butoxyethanol, and a-terpinol are also purchased neat from Sigma Aldrich or Ultra Scientific.

7.7 Second Source Standard: the second source standards are prepared at 200 ppm using one or more of the following:

- TCL BNA LCS Spike 100ug/mL (NSI Cat. No. WL-408-25)
- Acid Surrogate Mix (Restek Cat. No. 31083) at 7500ug/mL and
- Base/Neutral Surrogate Mix (Restek Cat. No. 31082) at 5000ug/mL
- Benzaldehyde 2000ug/mL (Restek Cat. No. 33017)

- Atrazine 1000ug/mL (Restek Cat. No. 32208)
- Caprolactam 1000ug/mL (Restek Cat. No. 31833)
- 1,4-Dioxane 2000ug/mL (Restek Cat. No. 31853)
- 1,1' Biphenyl, prepared from neat, (ChemService Cat. No.PS-2032)

7.8 The working tune standard at 50ug/mL is prepared by adding 50uL of the stock standard to a final volume of 1mL(*) with methylene chloride

7.9 The internal standard stock is prepared in-house at 2000ug/mL. A vial is opened and then emptied into a mini-inert vial equipped with an on/off valve for daily use. 20uL are added to each 1.0mL extract or standard.

7.10 An 8270 add-on Intermediate standard is prepared by weighing 0.05g neat compounds into 10mL methanol for a concentration of 5000µg/mL.

7.11 Any additional intermediate standards from neat are prepared by weighing 0.100g into 5mL methanol for a concentration of 20,000µg/mL.

7.12 An intermediate calibration standard at a concentration of 200µg/mL is prepared by combining the following volumes of primary standards and diluting to 5000uL(*) using methylene chloride:

8270 MEGA Mix	1000uL
Benzidine Mix	200uL
Acid surrogate	100uL
B/N surrogate	200uL
8270 Add-on Mix	200uL
Special Project (from 7.11)	50uL

7.13 Multi-level working calibration standards are prepared from the intermediate standard as follows(*):

	Volume Intermediate Standard (uL)	Volume Methylene Chloride (uL)	Volume Internal Standard (uL)
5 ug/mL	25	975	20
10 ug/mL	50	950	20
25 ug/mL	125	875	20
40 ug/mL	200	800	20
60 ug/mL	300	700	20
80 ug/mL	400	600	20

7.14 Second source standard: a working second source standard at 25ug/mL is prepared in a similar manner as the midpoint of the Initial Calibration.

(*) NOTE: Volumes above can be adjusted to make larger or smaller final volume of the Standards.

All of the working standards are labeled SWyymmddX,

Where: S = semivolatile
W = working standard
yymmdd = date the working standard is prepared
X = the order that the working standard is prepared on that date, in increasing alphabetical order

The working standards are protected from light and stored in the freezer (F7) at less than -10°C to -20°C. The standards are stored away from sample extracts to minimize cross contamination. All vials containing working standards must be labeled according to the current version of SOP 80.0001 Standard Preparation, Equivalency and Traceability. Be sure the vial label is not worn or difficult to read. Any vial whose label becomes worn or difficult to read should be re-labeled.

Working standard expiration dates are 12 months after they are prepared. Unopened ampulated standards' expiration dates are based on manufacturer's expiration dates. If no manufacturer's expiration date is provided, the ampulated standards may be retained unopened for up to two years. Once an ampulated standard is opened it may be retained for one year from the date it was opened.

All of the standard preparation information is recorded in the LIMS standard logbook. Document all necessary information related to the preparation of the standard including solvent lot number, volumes, expiration dates and standard IDs.

8. Procedure

To ensure the appropriate analyst is performing the analysis, the analyst's initials should be entered in the Enviroquant acquisition software (do not use the default value). The analyst processing and reviewing data will initial the Instrument Run Logbook when processing data.

8.1 Extraction - The methods in SW-846 typically used for sample extraction are as follows:

- Method 3510 extracts aqueous samples for water-insoluble and slightly water-soluble organics. The samples are serially extracted with methylene chloride using a separatory funnel.

- Method 3520 extracts aqueous samples for water-insoluble and slightly water-soluble organics. The samples are placed in a continuous liquid-liquid extractor and extracted with methylene chloride for a minimum of 18 hours.

- Method 3540 extracts waste, sludge, sediment and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate, placed in an extraction thimble or between plugs of glass wool, and extracted using 1:1 v/v methylene chloride/acetone in a Soxhlet extractor.

- Method 3570 extracts small volumes of waste, sediment and soil samples for water-insoluble and slightly water-soluble organics. The samples are extracted with acetone and then with methylene chloride or hexane in a VOA vial.

- Method 3550 extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate to form a free-flowing powder, and then extracted by ultrasonic extraction using 1:1 v/v methylene chloride/acetone.

- Method 3545 extracts waste, sludge, sediment and soil samples for water-insoluble and slightly water-soluble organics. A 15gram sample is mixed with diatomaceous earth to form a free-flowing mixture. This is added to the sample extraction cell. The cell is loaded on the PFE extractor to perform the extraction.

8.2 Tuning:

The tune standard is prepared at 50µg/mL. The GC/MS must be tuned to meet decafluorotriphenylphosphine (DFTPP) criteria every 12 hours when standards, samples or QC are to be analyzed.

8.2.1 Procedure for performing tune - Use the GC/MS conditions in **Section 6.1.1.4** to perform the tune analysis.

8.2.2 Acceptance criteria for tune - The mass spectrum of DFTPP 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; if needed, background subtraction will be performed and must be accomplished using no more than 20 scans prior to the elution of DFTPP. It is important that the analyst does not selectively add or subtract scans to generate the tune. This is the standard approach used by Target software.

For SW846 projects, the tune can also be obtained using one of the following procedures (1) use one scan at the peak apex, (2) use the one scan either directly preceding or the following scan, (3) use the average across the entire peak Any composite spectrum that is obtained manually (not using the Target software approach) is required to be documented in the Instrument Run log. The analyst is required to document which scans were used for averaging as well as the scan that was used for background subtraction.

A typical mass spectrum and mass spectral listing of the tune in listed in **Figure 2**.

The acceptance criteria are as follows**:**

Mass	Ion Abundance
51	10 - 80% of Base Peak
68	< 2.0% of mass 69
70	< 2.0% of mass 69
127	10 - 80% of Base Peak

197	< 2.0% of mass 198
198	Base peak, or > 50% of mass 442
199	5.0 - 9.0% of mass 198
275	10 - 60% of Base Peak
365	> 1% of mass 198
441	Present, < 24% of mass 442
442	Base Peak or > 50% of mass 198
443	15 - 24% of mass 442

Once the mass spectrometer passes the DFTPP tune, all subsequent standards, samples, and QC associated with the tune must be analyzed using identical mass spectrometer instrument conditions.

Experience working with the instruments has shown the following:

- High m/z 51, this is due to dirty ion source.
- Low to zero abundance of m/z 365, this is due to lack of instrument sensitivity.
- High m/z 68 and/or 70, this is due to high background.
- Incorrect ratio of m/z 199/198 and 441/442/443, this is due to incorrect instrument mass resolution and/or threshold settings.

8.3 % Breakdown –the breakdown analysis is performed to ensure inertness of the GC system such that labile compounds will not decompose or be adsorbed in the chromatographic system. The system inertness is evaluated by assessing the integrity of DDT that is present in the tune solution. The breakdown of DDT to DDE and DDD can not exceed 20%. The breakdown is evaluated as follows: the mass chromatograms for m/z 235/165 are plotted to detect the presence of DDD, the mass chromatograms for m/z 246/318 are plotted to detect the presence of DDE. If these breakdown products are determined to be present, the area count of the total ion chromatogram (not single ion chromatogram) for DDT, DDE and DDD are integrated. The % breakdown is calculated as follows:

$$\% \text{ Breakdown} = \frac{\text{Area count of DDE + DDE}}{\text{Area count of DDT}} \times 100$$

If the breakdown exceeds the QC limit of 20%, consider replacing the injector seal and/or injection liner. Repeat the tune and evaluate the % breakdown.

8.4 Column performance check: the column performance check is performed to ensure the GC column is amenable to the analysis of polar compounds including acids and bases. The column performance check is evaluated by the tailing of benzidine and pentachlorophenol. The maximum tailing factor for each compound is 2. See **Figure 1** for instruction on how to calculate the tailing factor. If tailing factor is above 2, corrective action includes clipping off an additional length of the column. Repeat the tune and evaluate for tailing.

8.5 Initial Calibration - Initial calibration is performed after the instrument passes the tune, % breakdown requirements and column performance check. Initial calibration is required after

major instrument maintenance including source cleaning and/or changing column. Initial calibration will also be performed if continuing calibration analyses do not meet QA/QC criteria.

Six calibration standard solutions are required for all target and surrogate compounds. Standard concentrations at 5, 10, 25, 40, 60 and 80ng/µL are required for the surrogates and all but nine of the target compounds. Nine compounds including 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitro-2-methylphenol, pentachlorophenol and benzoic acid require calibration at 10, 25, 40, 60 and 80ng/µL. The lowest standard concentration is typically correlated with the reporting limits for the target analytes (standard concentration at or below the reporting limit). This is the level closest to the method detection limit (MDL). There may be occasional requests to report results to limits that are below the lowest initial standard concentration. These must be documented and discussed in the project narrative. Any request for non-routine calibration should be discussed with the laboratory Supervisor and Project Manager to insure the resulting data meets project and method requirements and the procedures used and the quality of the data are fully documented.

DoD– the ICAL range shall consist of a minimum of 5 contiguous calibration points for organics, for all analytes reported. The low-level standard must be less than or equal to the reporting limit.

Several state and government programs have specific QA/QC Requirements and Performance Standards for the Initial Calibration. Refer to the individual state/government documents for more details. In particular, Dept. of Defense requires the evaluation of SPCC/CCC compounds in both the ICAL and CCV. See **Attachment 1** for criteria.

After the calibration standards are prepared as directed in **Section 7.12**, the laboratory performs a six level calibration. Please note that for all target analytes except those with poor chromatographic performance discussed above, the relative response factor from the 5ng/uL level is included in averaging the RRF. For the nine poor chromatographic performers, the relative response factor from the 5ng/uL level is not included and the 10ng/uL is used as the lowest calibration level.

8.5.1 Calculation for Initial Calibration:

From the multi-level level calibration, the relative response factor (RRF) for each target compound is determined using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where: A_x = area of the characteristic ion for the target compound to be measured
A_{is} = area of the characteristic ion for the associated internal standard
C_{is} = concentration of the internal standard
C_x = concentration of the compound to be measured

Please refer to **Table 3** for the list of target analytes and their associated internal standard. Please note that this is the Table 5 of the SW-846 Published Method 8270D.

The mean relative response factor is determined by averaging the 6level RRF values. The % relative standard deviation (%RSD) of the RRF is also calculated using:

$$\%\ RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

Where: Standard Deviation = $\sqrt{\sum (Xi - X)^2 / (n-1)}$

Where: Xi = each individual value used to calculate the mean
X = the mean of n values
n = the total number of values = 5

8.5.2 Initial calibration acceptance criteria for SW-846 are as follows:

- The relative retention time (RRT) for each of the target analytes including the surrogates at each calibration level must be within ± 0.06 RRT of the mean RRT for each compound.
- The area response for each internal standard at each calibration level must be within the inclusive range of –50% to +100% of the mean area response of the internal standard in all of the calibration levels.
- The retention time (RT) shift of the internal standards at each calibration level must be within ± 0.5 minutes compared to the mean retention time over the initial calibration range for each internal standard.
- The recommended minimum RF for common compounds is listed in **Table 4** (also Table 4 of Published SW-846 8270D).
- The RSD for all target analytes and/or surrogate compounds must be < 20%. The Target software will flag any compound whose RSD is greater than 20%. If the RSD of any target analytes and/or surrogate compounds is less than 20%, then the RRF is assumed to be constant over the calibration range and the average RRF is used for quantitation. If the calibration is not linear, make sure whether the problem is related to calibration standards or instruments. Experience with instruments S1 and S2 suggested that when the RRF decreases with increasing standard concentration, increasing the emission current from 50 mA to 75 mA and/or adjusting the electron multiplier voltage to make sure the area counts at 160 ng level do not exceed 10 million *will improve* system linearity.
- Given the large number of target analytes, it is likely that some analytes may exceed the acceptance limit. In those instances, the initial calibration is deemed acceptable if the following conditions are met (in order of preference):

  (1) The method allows for a maximum of 10% of the target analytes and/or surrogate compounds to fail the 20% RSD criteria. These allowable outliers should NOT be common compounds or compounds of interest to a specific

project that will utilize this initial calibration. In addition, these outlier RSD have a maximum of 50%.

(2) Linear calibration: a least squares regression may be used. The analyst may employ a regression equation for the analyte(s) that does not pass the earlier approach. The regression will produce the slope and intercept terms for the following linear equation:

$y = mx + b$

Where y = instrument response (peak area)
m = slope of the line
x = concentration of the calibration standard
b = intercept

It is important that the origin (0,0) is not included as the sixth calibration point and that the above equation is not forced through the origin.

The linear regression is deemed acceptable if the correlation coefficient **r ≥ 0.995**.

(3) Non linear calibration: The analyst may employ a non linear regression coefficient of determination (COD). The second order quadratic fit will have the following equation:

$y = ax^2 + bx + c$

Where y = instrument response (peak area or height)
a and b = slope of the curve
x = concentration of the calibration standard
c = intercept

In performing second order quadratic fit, the analyst should not force the curve to pass through the origin (0, 0). In addition, the origin should not be used as an additional calibration point.

From the quadratic fit, the "goodness of fit" is evaluated by calculating the coefficient of determination (COD). In order to be acceptable, the COD of the polynomial must be **≥0.99**.

8.5.3 Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. Benzaldehyde is prepared in a separate solution due to reactivity. This is performed by analyzing the 25ug/mL standard prepared in **Section 7.13.** The acceptance criteria are as follows:

For routine SW 8270 analyses, the calculated value of the analyte in the ICV must be 70 – 130% of the expected value (35 – 65 ng/uL).

DoD QSM: the calculated value of the analyte in the ICV should be 80-120% of the expected value (40-60 ng/uL), with no allowance for poor performing compounds.

If the above criteria are not met, the analyst has to evaluate the integrity of the primary and second source standards. First, reanalyze the ICV. Preparation and analysis of a new initial calibration may be required.

8.5.4 Corrective Action for Initial Calibration - Depending on which compound failed the criteria, corrective action included preparing fresh standards, source cleaning, and changing GC column or injection liners. Document the actions and resolution in the LIMS maintenance log.

8.5.5 Initial calibration acceptance criteria must be met before any sample, blanks or QC is to be analyzed. There may be circumstances where project-specific criteria allow the use of an initial calibration where one or more compound exceeds the acceptance criteria. For example, work performed under the Massachusetts Contingency Plan (MCP) allows up to 20% of the non-CCC analytes (calibration check compounds are acenaphthene, 1,4-dichlorobenzene, hexachlorobutadiene, N-nitroso-di-phenylamine, di-n-octylphthalate, fluoranthene, benzo(a)pyrene, 4-chloro-3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol and 2,4,6-trichlorophenol) to have %RSD > 30 or r < 0.99. This situation is to be discussed with the Technical Director or Project Manager for approval. Any compound not passing the calibration criteria will be flagged on Form 7 and the information included in the data report. This information will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.

8.5.6 Upon the successful completion of the initial calibration, select the mid-point calibration (25ug/mL standard) and update the reference spectra in Target.

8.5.7 Refer to SOP No. 110.0008 for details on the need for and documentation of manual integration.

8.6 Calibration verification - Calibration verification standards containing all of the target and surrogate compounds at 25ng on-column injection is performed every time samples are to be analyzed to ensure that the GC/MS system continues to meet instrument sensitivity and linearity requirements. An example of a continuing calibration chromatogram and quantitation report is included in **Figure 3**.

8.6.1 Frequency of Continuing Calibration - The continuing calibration standard must be performed once every 12 hours. If time remains in the 12-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed.

8.6.2 Procedure for Performing Continuing Calibration Verification - The continuing calibration verification (CCV) is performed at 25ng injection. Calculate the % difference between the continuing calibration RRF and those from the most recent initial calibration. The % difference is determined as follow:

$$\% \text{ Difference} = \frac{RRF_c - RRF_i}{RRF_i} \times 100$$

Where:

RRF_c = relative response factor from continuing calibration
RRF_i = mean relative response factor from the most recent initial calibration which meets acceptance criteria

Use % drift when using least-squares calibration.

$$\% \text{ Drift} = \frac{Conc_c - Conc_t}{Conc_t} \times 100$$

Where:

$Conc_c$ = concentration obtained from continuing calibration
$Conc_t$ = theoretical concentration of standard

8.6.3 Continuing calibration acceptance criteria:

- The recommended minimum RF for common compounds is listed in **Table 4**.
- The % D must be ≤ 20% (use % drift if using a regression fit model). Given the large number of target analytes, it is likely that some analytes may exceed the acceptance limit. In those instances, the continuing calibration is deemed acceptable if the following condition is met:
- A maximum of 20% of the target analytes and/or surrogate compounds to fail the 20% RSD criteria. These allowable outliers should NOT be common compounds or compounds of interest to a specific project that will utilize this initial calibration. In addition, these outlier %Ds have a maximum of 50%.
- No quantitation ion may saturate the detector.
- The internal standard retention time of the CCV must be within 30 seconds from that of the mid-point calibration (25ug/mL) of the associated initial calibration when run on the same day. Otherwise the CCV will be used to set the day's RRT to account for potential changes due to GC column maintenance.
- The internal standard area counts must be within +100% to –50% from that of the mid-point calibration (25 ug/mL) of the associated initial calibration when run on the same day. Otherwise the CCV will be used for comparison of IS areas.

Several states have specific QA/QC Requirements and Performance Standards for the Continuing Calibration. Refer to the individual state documents for more details.

8.6.4 Corrective Action for Continuing Calibration - Depending on which compound(s) fail(s) the criteria, corrective action includes preparing fresh standards, source cleaning, changing the GC column or injection liners. Repeated failure to pass continuing

calibration may necessitate performing new initial calibration. See **Attachments 1-3** for specific QC criteria and corrective action.

8.6.5 Continuing calibration acceptance criteria must be met before any samples or QC is to be analyzed. There may be circumstances where project-specific criteria allow the use of a continuing calibration where one or more compound exceeds the acceptance criteria. For example, work performed under the Massachusetts Contingency Plan (MCP) allows up to 10% of the non-CCC analytes (calibration check compounds are acenaphthene, 1,4-dichlorobenzene, hexachlorobutadiene, N-nitroso-di-phenylamine, di-n-octylphthalate, fluoranthene, benzo(a)pyrene, 4-chloro-3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol and 2,4,6-trichlorophenol) to have %D > 30. This is to be discussed with the Technical Director or Project Manager for approval. Any compound not passing the calibration criteria will be flagged on Form 7 and the information included in the data report. This information will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.

8.7 Sample Analysis:

Prior to sample analysis, the sample extract and the internal standard prepared in **Section 7.9** are allowed to warm to room temperature to ensure complete dissolution of the high molecular weight internal standards.

Twenty microliters of the 2000ppm internal standard solution is added to each of the sample extracts at 1mL final volume to ensure 40ng on-column amount. The internal standard volume will be adjusted for smaller extract volume. A 1uL aliquot of the extract in injected onto the GC/MS via an auto-sampler.

Target compounds identified in the sample extracts at concentration above the calibration range will be reanalyzed at dilution. These samples will be labeled with the DL suffix. For these re-analyses, an aliquot of the original extract will be diluted with methylene chloride. Additional aliquots of internal standards are then added to ensure an on-column injection of 40ng of the internal standards. For the dilution analysis, the target compounds that exceeded the calibration range in the initial analysis should be detected above the mid-level calibration level (40ng on-column).

8.7.1 Criteria for reporting dilution: the final dilution analysis is always reportable. This analysis should have the concentration of the most concentrated compound near or above the mid-level point of the calibration range.

8.7.2 Normally, the initial analysis result will be reported as long as QC criteria are met.

8.7.3 If the laboratory has prior information that a sample may contain concentrations of target and/or non-target compounds exceeding the instrument calibration range, or if the extracts are dark and viscous, the initial analysis may be performed at dilution. The initial analysis at dilution is noted on the data review checklist included with the data submitted for review, to allow discussion in the project narrative.

8.7.4 If the initial and dilution analysis together demonstrate matrix interference, such as surrogate/internal standard area counts out of the QC limit in both analyses, both sets of data are typically reported. Also, if the initial analysis provides important information to the project, it should be reported, with any QC exceedences noted on the data sheets (e.g.: flagged surrogate recovery in Form 2, flagged internal standard area counts in Form 8 and "E" data qualifier in Form 1). These will be incorporated and discussed in the project narrative.

8.8 Analytical Sequence: The following sequence is recommended:

Initial Batch	**Middle/Final Batch**
1. Tune	1. Tune
2. ICal Standard #1	2. CCV
3. ICal Standard #2	3. Method Blank
4. ICal Standard #3	4. LCS
5. ICal Standard #4	5-12. Samples (< 8)
6. ICal Standard #5	13. CCV
7. ICal Standard #6	14. Method Blank
8. ICV (second source)	15. MS
9. Method Blank	16. MSD
10. LCS	17-24. Samples (< 8)
11-18. Samples (< 8)	
19. Tune (as required per 12 hr.)	
20. CCV (as required per 12 hr.)	
21. Method Blank	
22. MS	
23. MSD	
24-31. Samples (< 8)	

In instances where there is no Method Blank, LCS, MS/MSD with the batch of samples to be analyzed, the lab may analyze an instrument blank after the successful completion of the calibration verification. Analyze colorless or light colored extracts first and arrange the dirty sample extracts towards the end of the analytical sequence.

9. Data Reduction and Calculations

9.1 Identification of Target Compounds - Two criteria are used to identify target compounds:

9.1.1 Relative Retention Time (RRT) - The sample component RRT must agree within ± 0.06 RRT units of the RRT of the component in the associated continuing calibration standard.

The relative retention time is determined as follows:

$$RRT = \frac{\text{Retention of target compound}}{\text{Retention of internal standard}}$$

Retention time of associated internal standard

9.1.2 Comparability of mass spectra - The requirements for qualitative verification by comparison of mass spectra is as follows:

- all ions present in the standard mass spectra at a relative abundance greater than 30% must be present in the sample spectrum
- the relative intensities of ions specified above must agree within ± 30 % between the standard and sample spectra
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered; this may be due to potential co-eluting interferences
- if the criteria above are not met but in the technical judgment of the analyst that the identification is correct, the lab will report the identification and proceed with the quantitation

9.2 Identification of non-target compounds [tentatively identified compounds (TICs)] - Client may request the analysis of TICs. If not client requested, the TICs will NOT be reported. Non-target compounds will be searched using the NIST/EPA/NIH library. The non-target compound will be reported as part of the analysis requirement if:

9.2.1 The client requires a full data package deliverable, including level 4 or New York ASP-B reporting format (exceptions are projects that have a short list of target analytes such as TCLP, PAH, acid compounds only, base-neutral compounds only, STAR list or projects that the client specified no TIC reporting).

9.2.2 The non-target compounds will be identified and reported if:

- its response is greater than 10% of the closest eluting interference free internal standard
- its retention time is within the range of 30 seconds before the elution of the first target compounds (early eluting aldol condensation products are thus not reported), and 3 minutes after the elution of the last target compound
- Unless specified, up to **20** TIC are to be reported

9.2.3 Guidelines for making tentative identification:

- Ions greater than 10% in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%.
- Molecular ions present in reference spectrum should be present in sample spectrum.
- Ions present in sample spectrum but not in the reference spectrum should be reviewed for co-eluting interferences.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed with caution because of background contamination and/or co-eluting interferences.
- The lab shall report pesticide target compounds as semivolatile TIC.

- The lab shall not report volatile target compounds.

The non-target compounds will be reported as "unknown" if no valid tentative identification can be made (as based on analysts' interpretation**)**. If possible, try to give classification of the unknown, eg. Unknown aliphatic hydrocarbon, unknown halogenated etc.

If the Quality (Qual) of the match as determined by the library search program is above 85%, it typically meets the criteria above, and is considered a tentative identification. If the Qual is less than 85%, the match typically does not meet the criteria above, and is usually identified as "unknown".

9.3 Determining the Concentration of Target Compounds - Sample data are reported in units of µg/L for aqueous samples and ug/Kg dry weight basis for solid samples. For aqueous samples, results are reported to one significant figure if the value is ≤10ug/L. At greater concentration, the results are reported to two significant figures. Solid sample results will be reported in dry weight unless otherwise specified for unusual sample matrices (tissue). Results for solid sample are reported to two significant figures.

9.4 Rounding Rule – Analysis results are to be rounded according to the current EPA guidelines.

9.5 The average RRF from the initial calibration is used to quantitate the target compounds. It is important to note that the concentrations of the target compounds do not exceed the calibration range. Any target analyte that exceeds the calibration range will be diluted and re-analyzed as discussed in **Section 8.7**.

Target compounds identified are quantitated using the following equations:

$$\text{For aqueous samples, Concentration } ug/L = \frac{(A_x)\,(I_s)\,(V_t)\,(Df)}{(A_{is})\,(RRF)\,(V_o)\,(V_i)}$$

$$\text{For solid samples, Concentration } ug/Kg = \frac{(A_x)\,(I_s)\,(V_t)\,(Df)}{(A_{is})\,(RRF)\,(W_s)\,(V_i)\,(S)}$$

Where:

A_x = area of the characteristic ion for the compound to be measured
A_{is} = area of the characteristic ion of the associated internal standard
I_s = amount of internal standard added in nanogram
RRF = relative response factor
V_o = volume of water extracted in milliliters = 1,000
V_t = volume of the sample extract in milliliters = 1
Df = dilution factor
W_s = weight of soil extracted in grams
S = % solid

9.6 Determining the concentration of non-target compounds - An estimated concentration for non-target compounds is determined using the closest eluting internal standard free of interference. The formula to calculate the concentration is the same as those for water and soil samples described above. Total area counts from the total ion chromatograms are to be used for both the compound to be measured and the associated internal standard. A RRF of one (1) is assumed. An estimated concentration must be calculated for all tentatively identified compounds as well as these identified as unknown.

9.7 Acceptance Criteria for Sample Analysis - Acceptance criteria are as follows:

- The sample must meet both extraction and analysis holding time.
- The sample must have a compliant tune, initial calibration and continuing calibration.
- The sample must have a compliant method blank.
- The sample must have a compliant LCS and/or LCSD
- The surrogate recovery per this SOP (**Section 10.6**) or client-specified criteria must be met.
- All of the target analyte concentrations should be within the calibration range.
- Excluding the solvent front or the Aldol condensation peak for solid extract analysis, no ion should saturate the detector.

In addition,

- Area count of each of the internal standards in the inclusive range of - 50% to + 100% of the response of the continuing calibration.
- Retention time of each of the internal standards must not shift more than ± 0.5 minute from the continuing calibration.

When the above two criteria are not met, the laboratory will re-analyze the sample at the same concentration or at dilution, unless similar results were found for its associated MS/MSD. Dark and viscous extracts with an unresolved complex mixture consisting of hydrocarbons usually results in the depression of the last two eluting internal standards, and subsequent high recovery of terphenyl-d14. In these instances, the samples may not require re-analysis. The supervisor or project manager should be consulted for further guidance.

See **Attachments 1-3** for specific guidelines. Please note that these two criteria must be met for interference-free QC samples including method blank and LCS/LCSD.

9.8 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

$$\%\ \text{Recovery}\ (\%R) = 100 \times (SSR - SR)/(SA)$$

Where: SSR = spiked sample result
SR = sample concentration
SA = spike added

9.9 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

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

Where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

9.10 Manual integrations are performed, reviewed and documented per SOP No. 110.0008 Manual Integration of IC, GC and GC/MS Chromatograms.

10. Quality Assurance/Quality Control

10.1 All standards made from a primary standard expire on or before the primary standard's expiration date. All standards must be labeled with the expiration date.

10.2 Use of this method is restricted to analysts who are knowledgeable in the operation of the instrumentation and have performed a proficiency test with acceptable accuracy and precision results. All analysts must have read this SOP and asked questions and received explanation for the areas they are not familiar with. This SOP should be referred to often, and used as a reference for this procedure. Details of the procedure for documenting analyst proficiency can be found in the current revision of SOP 80.0016.

10.3 Method blanks - A method blank is extracted with every batch not to exceed 20 samples (excluding LCS/LCSD, MS/MSD).

Acceptance criteria for the method blank are as follows:

- The recovery of the surrogates must be within the calculated acceptance limits discussed in **Section 10.6.**

- The concentration of the target compounds in the method blank must be less than the Reporting Limit; the concentration for common laboratory contaminants such as phthalate esters, must not exceed the 5 times the Reporting Limit.

DoD QSM: the concentration of the target compounds in the method blank must be less than one-half of the Limit of Quantitation (LOQ); the concentration for common laboratory contaminants such as phthalate esters, must not exceed the Limit of Quantitation (LOQ).

Any sample associated with a blank that fails these criteria checks shall be re-extracted in a subsequent preparation batch, except when the sample analysis resulted in a non-detect for the compound that failed the criteria.

If no sample volume remains for re-extraction, the results shall be reported with appropriate data qualifying codes. The "B" qualifier is applied to positive sample results on Form 1 or LIMS Level 2 data sheet when the same compound is detected in the blank. For semivolatile analysis, the most common lab contaminant is bis (2-ethylhexyl) phthalate (BEHP).

10.4 Lab Control Sample (LCS) – A Lab Control Sample is a weight or volume of a clean reference matrix (sodium sulfate or DI water) that is spiked with all target analytes and surrogates and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples. Where applicable, a Lab Control Sample Duplicate (LCSD) will also be performed to evaluate reproducibility.

10.4.1 Acceptance criteria for LCS:

- General acceptance: compliant surrogate recovery
- For regular SW8270 projects, the recovery is evaluated against the DoD QC limits for those compounds listed in QSM, and established in-house limits for all other compounds. Refer to the LIMS Test Information category/Test option/ specs for the most current QC control limits. See **Attachment 2** for DoD QC limits.
- If target analytes are outside of the acceptance limits, corrective action is required. Project-specific requirements, if available, will dictate the corrective action performed. See **Attachment 3** for further guidance.
- Due to the large number of target analytes, some recoveries may be outside the control limits. Poor performing compounds such as those that are reactive or highly polar (such as hexachloropentadiene or pentachlorophenol) should not be used to base batch acceptance on.

DoD QSM requirements: analyses of <11 analytes, no marginal exceedences (ME) are allowed. For the analysis of 11-30 analytes (typical PAH analysis), one ME is allowed; for the analysis of 31-50 analytes, two ME are allowed; for 51-70 analytes, three ME are allowed; for the analysis of 71-90 analytes (typical routine 8270D analysis), four ME are allowed; for the analysis of >90 analytes, five ME are allowed. See **Attachments 1 and 2** for further guidance.

- Reporting LCS Results – If any compounds are outside of the acceptance limits, their recoveries are qualified with the "*" flag on the LCS recovery summary report (Form 3) for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

10.5 Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicate are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain elevated concentrations of target analytes, the laboratory should perform one matrix spike and a separate un-spiked duplicate. For clean samples and those without documented history, matrix spike/matrix spike duplicate analyses are performed. Since the majority of the samples received at Spectrum RI do not have any documented history, Spectrum RI will routinely perform matrix spike and matrix spike duplicate.

10.5.1 Acceptance criteria for Duplicate Matrix Spike:

Matrix spike and matrix spike duplicate are used to assess the effect of matrix interferences on the analysis of the target analytes and the recovery should be used as advisory guidelines to answer the question posed above.

Control limits are the same as those discussed in **Section 10.4** (same as LCS aqueous and soil), but are used as advisory guidelines. This is especially true when the native sample exhibited a matrix effect or the spike concentrations are less than the native sample concentration. If the MS/MSD does not meet the in-house criteria, see **Attachment 3** for corrective action guidelines.

For *DoD* projects, the %RPD limits for the duplicate set is 30%. See **Attachment 1** for corrective action.

10.5.2 Reporting the Duplicate Matrix Spikes-If any compounds are outside of the acceptance limits, their recoveries and/or RPD are qualified with the "*" flag on the recovery MS/MSD summary report (Form 3) for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

10.6 Surrogate recoveries - The recovery of each surrogate compound in all samples, blanks and LCS/LCSD, MS/MSD will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.6.1 Acceptance criteria - The percent recovery of each of the surrogate compounds in blanks, samples, duplicate matrix spikes and LCS must be within the in-house acceptance windows with the exception of one surrogate per fraction allowed out. Outliers should have at least 10% recovery with the exception of Phenol-d5 in an aqueous sample. This compound is an extremely poor performer *.

	Solid	Aqueous
• 2-Fluorophenol	35-105	20-110
• Phenol-d_5	40-105	10-115
• 2,4,6-Tribromophenol	35-125	40-125
• Nitrobenzene-d_5	35-100	40-110
• 2-Fluorobiphenyl	45-105	50-110
• p-Terphenyl-d_{14}	30-125	50-135

* For *DoD* projects (unless superseded by client request): Phenol-d_5 in an aqueous sample is considered a poor performing analyte with a marginal exceedence (ME) limit of 0-135%.DoD does not allow any other ME for surrogates.

10.6.2 Any **sample**, which fails to meet the above criteria, will be subjected to re-extraction. There may be instances when recovery exceedence is matrix related. For example, samples with high concentration of non-target hydrocarbons may have depressed internal standard area counts and resulting "elevated" recovery of the associated surrogate compound such as p-Terphenyl-d_{14}. In these instances where the recovery has a high bias due to sample matrix, the sample is not re-extracted. The sample extract may be re-analyzed to confirm the matrix effect. Once confirmed, both data sets are reported and the occurrence mentioned in the report narrative.

Any **method blank**, which fails to meet the above criteria, will trigger re-extraction of the entire preparation batch. In the event that the surrogate recoveries are above the upper QC limits and no target analytes are detected, the Technical Director or Laboratory Director should be notified for guidance regarding re-extraction. In the event that re-extraction is warranted but no sample volume remains, notify the Lab Director and Project Manager, report the data and note the issues in detail on the data review checklist for inclusion in the project narrative. See **Attachment 3** for corrective action guidelines.

10.6.2.1 If re-extraction and re-analysis of the sample demonstrate similar recovery performance, both sets of results will be reported to demonstrate matrix- related problems.

10.6.2.2 Re-extraction is not required for the sample, if the recovery is out of the QC limits for both the sample and its duplicate matrix spikes.

10.6.3 Reporting of Surrogate Recoveries-All surrogate outliers will be flagged with an "*" on the surrogate recovery report (Form 2) for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports.

10.7 MDL studies are conducted to establish the limit of detection applicable to this method. MDL verification at approximately 1-4 x MDL is analyzed after the study which also sets the DoD Limit of Detection (LOD). LOD verification must be analyzed quarterly on each instrument used for DoD program work. An annual verification of the detection and reporting limits must be performed for the method per NELAC. Please refer to the Spectrum RI SOP No. 80.0005 Determination of Method Detection Limits for more detail.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and samples results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, and instrument logs are reviewed and signed by the appropriate area supervisor. 100% of the data is reviewed. The QA Director randomly reviews 10% of the data reported by the laboratory.

11.2 Analysts transfer organic data report forms, data review checklist(s) and raw data electronically to the reporting group for assembly into a final report. The data submitted for report preparation is dependent on project requirements and is subjected to further review by a data reviewer. The project manager reviews the data for reasonableness and finalizes the project narrative prior to releasing the report to the customer.

12. Data Management and Records Management

12.1 Electronic data generated from the analysis of Semivolatile 8270 extracts (calibrations, QC, samples) is saved and managed per SOP 110.0029 Electronic Data Management.

12.2 All analysis information is documented in the individual Instrument Run/Injection Logbook regardless of run acceptance. No injections are deleted from the sequence.

13. Corrective Action Procedures

13.1 Corrective actions to be implemented in the event QC results are outside of the acceptance range are covered in **Sections 8, 9, and 10**. QC corrective action tables for *DoD* are presented in **Attachments 1 and 2**. An overview of the corrective actions and associated documentation is listed in **Attachment 3**.

13.2 Discrepancy reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a discrepancy report for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007 Corrective Action Procedures. Starting in 2006, corrective actions were recorded in the LIMS system in the Quality Control section/corrective action reports. All employees have access to LIMS and may initiate a corrective action. If help is needed, see the QA Director for assistance.

14. Health and Safety

The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is archived by the health and safety officer and available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Health and Safety Plan. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

15. Pollution Prevention, Waste Management, Acronyms and Definitions

See sections 19.0 (Waste Management) and 20.0 (Definitions, Acronyms, and Abbreviations) of the current Quality Assurance Plan.

16. References

1. U.S. Environmental Protection Agency. Gas Chromatography/Mass Spectrometry Method 8270D, SW-846 Test Methods for Evaluating Solid Wastes, Revision 4, February 2007.

2. "Quality Systems Manual for Environmental Laboratories" Department of Defense, Final Version 4.1, April 2009 or current version.

Attachments:

1. **Table 1**: List of Acronyms
2. **Table 2**: Semivolatile Analyte list and Method Reporting Limits (RLs).
3. **Table 3**: Internal Standard/Analyte list (Table 5 from Published Method).
4. **Table 4**: Suggested RRF per SW8270D (Table 4 from Published Method).
5. **Figure 1**: Calculation of Peak Tailing Factors.
6. **Figure 2**: DFTPP Tune and Chromatogram.
7. **Figure 3**: Continuing Calibration Standard Chromatogram and Quantitation Report.
8. **Attachment 1**: DoD Specific QC Requirements: DoD-B SW846 box, Table F-4.
9. **Attachment 2**: DoD Specific QC Control Limits: Tables G-6 and G-7.
10. **Attachment 3:** Overview of Corrective Action and Documentation Examples.
11. **Attachment 4:** Additional QA/QC Requirements for MA-DEP.

Table 1
List of Acronyms

1,2-DCB	1,2-Dichlorobenzene
1,3-DCB	1,3-Dichlorobenzene
1,4-DCB	1,4-Dichlorobenzene
2,4-DNT	2,4-Dinitrotoluene
2,6-DNT	2,6-Dinitrotoluene
AFCEE	Air Force Center for Environmental Excellence—procedures used for selected projects—to be replaced by DOD QSM procedures.
DFTPP	Decafluorotriphenylphosphine
DoD	Department of Defense (including Army, Navy, Air Force)
LCS	Lab control sample
MDL	Method detection limit
MQL	Method quantitation limit
ME	Marginal Exceedence
MS	Matrix spike
MSD	Matrix spike duplicate
QSM	Quality Systems Manual for DoD work
RL	Reporting Limit (occasionally referred to as PQL or Practical Quantitation Limit, or MRL or Method Reporting Limit)
LOD	Limit of Detection
LOQ	Limit of Quantitation

Table 2

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W **DefaultPrep:** BNA_W_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Aqueous **Units:** ug/L **Updated:** 01/03/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
A	0001	0001	Phenol	0.75000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0002	0001	Bis(2-chloroethyl)ether	0.75000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0003	0001	2-Chlorophenol	0.61000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0004	0001	1,3-Dichlorobenzene	0.71000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0005	0001	1,4-Dichlorobenzene	1.10000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0006	0001	1,2-Dichlorobenzene	0.84000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0007	0001	2-Methylphenol	0.96000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0008	0001	2,2´-oxybis(1-Chloropropane)	0.78000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0009	0001	4-Methylphenol	1.40000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0010	0001	N-Nitroso-di-n-propylamine	0.63000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0011	0001	Hexachloroethane	0.55000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0011	0002	Nitrobenzene	1.60000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0012	0002	Isophorone	0.47000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0014	0002	2-Nitrophenol	0.60000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0015	0002	2,4-Dimethylphenol	1.80000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0016	0002	2,4-Dichlorophenol	0.57000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0017	0002	1,2,4-Trichlorobenzene	0.93000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0018	0002	Naphthalene	0.96000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0019	0002	4-Chloroaniline	2.00000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0020	0002	Bis(2-chloroethoxy)methane	1.10000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0021	0002	Hexachlorobutadiene	0.75000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0022	0002	4-Chloro-3-methylphenol	0.60000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0023	0002	2-Methylnaphthalene	0.94000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0024	0003	Hexachlorocyclopentadiene	1.00000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0025	0003	2,4,6-Trichlorophenol	0.53000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0026	0003	2,4,5-Trichlorophenol	0.26000	20.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0027	0003	2-Chloronaphthalene	0.81000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0028	0003	2-Nitroaniline	0.71000	20.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0029	0003	Dimethylphthalate	0.37000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0030	0003	Acenaphthylene	0.42000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0031	0003	2,6-Dinitrotoluene	0.52000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0032	0003	3-Nitroaniline	0.97000	20.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0033	0003	Acenaphthene	0.65000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W **DefaultPrep:** BNA_W_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Aqueous **Units:** ug/L **Updated:** 01/03/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
A	0034	0003	2,4-Dinitrophenol	3.50000	20.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0035	0003	4-Nitrophenol	0.53000	20.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0036	0003	Dibenzofuran	0.52000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0037	0003	2,4-Dinitrotoluene	0.41000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0038	0003	Diethylphthalate	0.45000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0039	0003	4-Chlorophenyl-phenylether	0.41000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0040	0003	Fluorene	0.44000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0041	0003	4-Nitroaniline	0.96000	20.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0042	0004	4,6-Dinitro-2-methylphenol	0.79000	20.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0043	0004	N-Nitrosodiphenylamine	1.10000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0044	0004	4-Bromophenyl-phenylether	0.54000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0045	0004	Hexachlorobenzene	0.44000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0046	0004	Pentachlorophenol	1.70000	20.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0047	0004	Phenanthrene	0.45000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0048	0004	Anthracene	0.48000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0049	0004	Carbazole	0.64000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0050	0004	Di-n-butylphthalate	0.48000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0051	0004	Fluoranthene	0.33000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0052	0005	Pyrene	0.44000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0053	0005	Butylbenzylphthalate	0.32000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0054	0005	3,3´-Dichlorobenzidine	1.70000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0055	0005	Benzo(a)anthracene	0.40000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0056	0005	Chrysene	0.42000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0057	0005	Bis(2-ethylhexyl)phthalate	1.30000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0058	0006	Di-n-octylphthalate	0.47000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0059	0006	Benzo(b)fluoranthene	0.94000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0060	0006	Benzo(k)fluoranthene	1.20000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0061	0006	Benzo(a)pyrene	1.20000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0062	0006	Indeno(1,2,3-cd)pyrene	0.38000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0063	0006	Dibenzo(a,h)anthracene	0.44000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
A	0064	0006	Benzo(g,h,i)perylene	0.39000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
I	0001	0001	1,4-Dichlorobenzene-d4	0.10000	10.00000	160.00000	N/A	N/A		09/07/10
I	0002	0002	Naphthalene-d8	0.10000	10.00000	160.00000	N/A	N/A		09/07/10

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W **DefaultPrep:** BNA_W_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Aqueous **Units:** ug/L **Updated:** 01/03/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
I	0003	0003	Acenaphthene-d10	0.10000	10.00000	160.00000	N/A	N/A		09/07/10
I	0004	0004	Phenanthrene-d10	0.10000	10.00000	160.00000	N/A	N/A		09/07/10
I	0005	0005	Chrysene-d12	0.10000	10.00000	160.00000	N/A	N/A		09/07/10
I	0006	0006	Perylene-d12	0.10000	10.00000	160.00000	N/A	N/A		09/07/10
S	0001	0002	Nitrobenzene-d5	0.00000	10.00000	160.00000	N/A	N/A		09/07/10
S	0002	0003	2-Fluorobiphenyl	0.00000	10.00000	160.00000	N/A	N/A		09/07/10
S	0003	0005	Terphenyl-d14	0.00000	10.00000	160.00000	N/A	N/A		09/07/10
S	0004	0001	Phenol-d5	0.00000	10.00000	160.00000	N/A	N/A		09/07/10
S	0005	0001	2-Fluorophenol	0.00000	10.00000	160.00000	N/A	N/A		09/07/10
S	0006	0004	2,4,6-Tribromophenol	0.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	3-Methylphenol + 4-Methylphenol	1.40000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	2-Picoline	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	3,3´-Dimethylbenzidine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	2-Nitrophenol-d4	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	4-Aminobiphenyl	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0003	1,2,3,4-Tetrachlorobenzene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0002	1,2,3,5-Tetrachlorobenzene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	1,2,3-Trimethylbenzene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0002	4-Chlorophenol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	4-Chloroaniline-d4	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0003	1,2,4,5-Tetrachlorobenzene	0.92000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0002	3-Chlorophenol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	1,2-Dichlorobenzene-d4	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0003	3,4-Dichlorophenol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	4,6-Dinitro-2-methylphenol-d2	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	1,2-Diphenylhydrazine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	1,3,5-Trichlorobenzene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0002	3/4-Chlorophenol	2.00000	20.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	3-Methylcholanthrene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0003	3,5-Dichlorophenol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	1,2,4-Trimethylbenzene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	2-Butoxyethanol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	2,4-Dichlorobenzotrifluoride	1.00000	10.00000	160.00000	N/A	N/A		09/07/10

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W **DefaultPrep:** BNA_W_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Aqueous **Units:** ug/L **Updated:** 01/03/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0002	2,4,5-Trichlorotoluene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0003	2,3-Dichlorophenol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	2,5-Dichlorobenzotrifluoride	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0003	2,3,6-Trichlorophenol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	2,3,5-Trimethylnaphthalene	0.00999	0.00999	0.00000	N/A	N/A		09/07/10
X	0999	0002	2,3,5-Trichlorophenol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0003	2,3,4-Trichlorophenol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0005	Benzidine	1.80000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0003	2,3,4,6-Tetrachlorophenol	0.65000	25.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	1-Naphthylamine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0002	2,6-Dichlorophenol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	2-Naphthylamine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	2-Acetylaminofluorene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	1,3-Dinitrobenzene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0002	2-Chloroaniline	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	1-Methylphenanthrene	0.00999	0.00999	0.00000	N/A	N/A		09/07/10
X	0999	0002	1-Methylnaphthalene	0.81000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	1-Chloronaphthalene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	2-Chlorophenol-d4	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0002	1-Chloro-2-nitro-4-(trifluoromethyl)benzene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	2-Ethyl-1-hexanol	0.56000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	1,4-Naphthoquinone	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	1,4-Naphthoquinoline,1-oxide	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	1,4-Dioxane-d8	1.00000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	2,4-Dichlorophenol-d3	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	1,4-Dioxane	5.70000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0002	2,5-Dichlorophenol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	2,6-Dimethylnaphthalene	0.00999	0.00999	0.00000	N/A	N/A		09/07/10
X	0999	0000	Missouri - Oil Range Organics	0.00999	0.00999	0.00000	N/A	N/A		09/07/10
X	0999	0000	O,O,O-Triethylphosphorothioate	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	N-Nitrosopyrrolidine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	N-Nitrosopiperidine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	N-Nitrosomorpholine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W **DefaultPrep:** BNA_W_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Aqueous **Units:** ug/L **Updated:** 01/03/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0000	N-Nitrosomethylethylamine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	N-Nitrosodimethylamine	0.56000	10.00000	160.00000	N/A	N/A	01/21/09	09/07/10
X	0999	0000	N-Nitrosodiethylamine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Hexachloropropene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	N,N-Dimethylformamide	2.60000	10.00000	160.00000	N/A	N/A	02/06/09	09/07/10
X	0999	0004	Octachlorocyclopentene	0.62000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Missouri - Diesel Range Organics	50.00000	50.00000	5000.00000	N/A	N/A		09/07/10
X	0999	0000	Methyl Parathion	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Methyl methane sulfonate	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Methapyrilene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	m-Toluic Acid	2.00000	20.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Kepone	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Isosafrole	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0004	Azobenzene	0.71000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	N-Nitroso-di-n-butylamine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Phenol-d6	0.00999	0.00999	160.00000	N/A	N/A		09/07/10
X	0999	0000	Thionazin	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	Tetramethyllead	1.00000	10.00000	250.00000	N/A	N/A		09/07/10
X	0999	0001	Tetraethyllead	1.00000	10.00000	250.00000	N/A	N/A		09/07/10
X	0999	0000	Sym-Trinitrobenzene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Safrole	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	Pyridine	0.57000	20.00000	160.00000	N/A	N/A	01/21/09	09/07/10
X	0999	0000	Pyrene-d10	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Pronamide	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	o-Toluic Acid	2.00000	20.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Phorate	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	o-Toluidine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Phenacetin	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Perylene	0.00999	0.00999	0.00000	N/A	N/A		09/07/10
X	0999	0004	Pentachloronitrobenzene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0003	Pentachlorobenzene	0.89000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	p-Toluic Acid	2.00000	20.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	p-Phenylenediamine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W **DefaultPrep:** BNA_W_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Aqueous **Units:** ug/L **Updated:** 01/03/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0000	p-(Dimethylamino)azobenzene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Hexachlorophene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0002	Phthalic anhydride	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0002	alpha-Terpineol	1.20000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	Total SVOC	10.00000	10.00000	0.00000	N/A	N/A		09/07/10
X	0999	0001	Benzaldehyde	0.51000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0003	1,1´-Biphenyl	0.65000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0004	Atrazine	1.30000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	Aramite-2	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Aramite-1	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Aramite	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Isodrin	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	Aniline	1.30000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	Benzo(e)pyrene-d12	0.00999	0.00999	0.00000	N/A	N/A		09/07/10
X	0999	0001	Acetophenone	0.51000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	Acenaphthylene-d8	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	a,a-Dimethylphenethylamine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	7,12-Dimethylbenz(a)anthracene	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	5-Nitro-o-toluidine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0003	4-tert-Octylphenol	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	4-Nitroquinoline-1-oxide	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	4-Nitrophenol-d4	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Anthracene-d10	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Dibenz(a,j)acridine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Fluorene-d10	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Famphur	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Ethyl parathion	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Ethyl methane sulfonate	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Disulfoton	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Diphenylamine	1.10000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	Dimethylphthalate-d6	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Dimethoate	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Benzo(a)pyrene-d12	1.00000	10.00000	160.00000	N/A	N/A		09/07/10

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W **DefaultPrep:** BNA_W_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Aqueous **Units:** ug/L **Updated:** 01/03/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0000	Dibenzothiophene	0.00999	0.00999	0.00000	N/A	N/A		09/07/10
X	0999	0000	Benzo(e)pyrene	0.00999	0.00999	0.00000	N/A	N/A		09/07/10
X	0999	0000	Diallate	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	Chlorobenzilate	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0002	Caprolactam	1.10000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0000	Bis(2-chloroisopropyl)ether	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	bis(2-Chloroethyl)ether-d8	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	Benzyl alcohol	0.87000	10.00000	160.00000	N/A	N/A	07/29/10	09/07/10
X	0999	0002	Benzoic acid	2.00000	20.00000	160.00000	N/A	N/A		09/07/10
X	0999	0000	4-Methylphenol-d8	1.00000	10.00000	160.00000	N/A	N/A		09/07/10
X	0999	0001	Diethylenetriamine	1.00000	10.00000	160.00000	N/A	N/A		09/07/10

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_S **DefaultPrep:** BNA_S_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Soil **Units:** ug/Kg **Updated:** 08/11/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
A	0001	0001	Phenol	37.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0002	0001	Bis(2-chloroethyl)ether	42.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0003	0001	2-Chlorophenol	41.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0004	0001	1,3-Dichlorobenzene	40.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0005	0001	1,4-Dichlorobenzene	36.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0006	0001	1,2-Dichlorobenzene	42.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0007	0001	2-Methylphenol	38.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0008	0001	2,2´-oxybis(1-Chloropropane)	51.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0009	0001	4-Methylphenol	35.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0010	0001	N-Nitroso-di-n-propylamine	32.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0011	0001	Hexachloroethane	35.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0011	0002	Nitrobenzene	38.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0012	0002	Isophorone	34.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0014	0002	2-Nitrophenol	36.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0015	0002	2,4-Dimethylphenol	36.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0016	0002	2,4-Dichlorophenol	38.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0017	0002	1,2,4-Trichlorobenzene	46.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0018	0002	Naphthalene	41.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0019	0002	4-Chloroaniline	24.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0020	0002	Bis(2-chloroethoxy)methane	39.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0021	0002	Hexachlorobutadiene	45.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0022	0002	4-Chloro-3-methylphenol	26.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0023	0002	2-Methylnaphthalene	42.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0024	0003	Hexachlorocyclopentadiene	96.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0025	0003	2,4,6-Trichlorophenol	39.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0026	0003	2,4,5-Trichlorophenol	37.00000	670.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0027	0003	2-Chloronaphthalene	38.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0028	0003	2-Nitroaniline	21.00000	670.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0029	0003	Dimethylphthalate	30.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0030	0003	Acenaphthylene	37.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0031	0003	2,6-Dinitrotoluene	28.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0032	0003	3-Nitroaniline	24.00000	670.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0033	0003	Acenaphthene	39.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_S **DefaultPrep:** BNA_S_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Soil **Units:** ug/Kg **Updated:** 08/11/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
A	0034	0003	2,4-Dinitrophenol	180.00000	670.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0035	0003	4-Nitrophenol	22.00000	670.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0036	0003	Dibenzofuran	36.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0037	0003	2,4-Dinitrotoluene	23.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0038	0003	Diethylphthalate	24.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0039	0003	4-Chlorophenyl-phenylether	40.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0040	0003	Fluorene	33.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0041	0003	4-Nitroaniline	25.00000	670.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0042	0004	4,6-Dinitro-2-methylphenol	25.00000	670.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0043	0004	N-Nitrosodiphenylamine	29.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0044	0004	4-Bromophenyl-phenylether	32.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0045	0004	Hexachlorobenzene	32.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0046	0004	Pentachlorophenol	140.00000	670.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0047	0004	Phenanthrene	26.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0048	0004	Anthracene	27.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0049	0004	Carbazole	28.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0050	0004	Di-n-butylphthalate	28.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0051	0004	Fluoranthene	29.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0052	0005	Pyrene	32.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0053	0005	Butylbenzylphthalate	26.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0054	0005	3,3´-Dichlorobenzidine	35.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0055	0005	Benzo(a)anthracene	33.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0056	0005	Chrysene	29.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0057	0005	Bis(2-ethylhexyl)phthalate	29.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0058	0006	Di-n-octylphthalate	28.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0059	0006	Benzo(b)fluoranthene	40.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0060	0006	Benzo(k)fluoranthene	43.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0061	0006	Benzo(a)pyrene	31.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0062	0006	Indeno(1,2,3-cd)pyrene	37.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0063	0006	Dibenzo(a,h)anthracene	35.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
A	0064	0006	Benzo(g,h,i)perylene	38.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
I	0001	0001	1,4-Dichlorobenzene-d4	0.33000	330.00000	80.00000	N/A	N/A		08/11/11
I	0002	0002	Naphthalene-d8	0.33000	330.00000	80.00000	N/A	N/A		08/11/11

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_S **DefaultPrep:** BNA_S_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Soil **Units:** ug/Kg **Updated:** 08/11/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
I	0003	0003	Acenaphthene-d10	0.33000	330.00000	80.00000	N/A	N/A		08/11/11
I	0004	0004	Phenanthrene-d10	0.33000	330.00000	80.00000	N/A	N/A		08/11/11
I	0005	0005	Chrysene-d12	0.33000	330.00000	80.00000	N/A	N/A		08/11/11
I	0006	0006	Perylene-d12	0.33000	330.00000	80.00000	N/A	N/A		08/11/11
S	0001	0002	Nitrobenzene-d5	0.00000	330.00000	80.00000	N/A	N/A		08/11/11
S	0002	0003	2-Fluorobiphenyl	0.00000	330.00000	80.00000	N/A	N/A		08/11/11
S	0003	0005	Terphenyl-d14	0.00000	330.00000	80.00000	N/A	N/A		08/11/11
S	0004	0001	Phenol-d5	0.00000	330.00000	80.00000	N/A	N/A		08/11/11
S	0005	0001	2-Fluorophenol	0.00000	330.00000	80.00000	N/A	N/A		08/11/11
S	0006	0004	2,4,6-Tribromophenol	0.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	3-Methylphenol + 4-Methylphenol	35.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	2-Nitrophenol-d4	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	2-Picoline	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	3,3´-Dimethylbenzidine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	4-Aminobiphenyl	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0003	1,2,3,4-Tetrachlorobenzene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	1,2,3,5-Tetrachlorobenzene	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	4-Chlorophenol	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	4-Chloroaniline-d4	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	1,2,3-Trimethylbenzene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0003	1,2,4,5-Tetrachlorobenzene	59.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	3-Chlorophenol	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0001	1,2-Dichlorobenzene-d4	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	3,4-Dichlorophenol	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	4,6-Dinitro-2-methylphenol-d2	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	1,2-Diphenylhydrazine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	3/4-Chlorophenol	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	1,3,5-Trichlorobenzene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	3-Methylcholanthrene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	3,5-Dichlorophenol	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0001	1,2,4-Trimethylbenzene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	2-Acetylaminofluorene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	2,4-Dichlorobenzotrifluoride	0.00999	0.00999	0.00000	N/A	N/A		08/11/11

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_S **DefaultPrep:** BNA_S_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Soil **Units:** ug/Kg **Updated:** 08/11/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0000	2,4,5-Trichlorotoluene	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	2,3-Dichlorophenol	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	2,3,6-Trichlorophenol	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	2,3,5-Trimethylnaphthalene	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	2,3,5-Trichlorophenol	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	2,3,4-Trichlorophenol	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	2,5-Dichlorobenzotrifluoride	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0001	Benzaldehyde	44.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0003	2,3,4,6-Tetrachlorophenol	31.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	1-Naphthylamine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	2,6-Dichlorophenol	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	1,4-Dioxane	50.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	1-Methylphenanthrene	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	2-Naphthylamine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	2-Butoxyethanol	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0002	2-Chloroaniline	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0002	1-Methylnaphthalene	41.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	1-Chloronaphthalene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0002	1-Chloro-2-nitro-4-(trifluoromethyl)benzene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	2-Chlorophenol-d4	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	2-Ethyl-1-hexanol	140.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	1,4-Naphthoquinone	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	1,4-Naphthoquinoline,1-oxide	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	2,4-Dichlorophenol-d3	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	1,4-Dioxane-d8	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	2,5-Dichlorophenol	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	1,3-Dinitrobenzene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	2,6-Dimethylnaphthalene	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	Missouri - Oil Range Organics	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0004	Atrazine	47.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	N-Nitrosopyrrolidine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	N-Nitrosopiperidine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	N-Nitrosomorpholine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_S **DefaultPrep:** BNA_S_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Soil **Units:** ug/Kg **Updated:** 08/11/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0001	N-Nitrosomethylethylamine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	N-Nitrosodimethylamine	34.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	N-Nitrosodiethylamine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	o-Toluic Acid	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	N,N-Dimethylformamide	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	o-Toluidine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Missouri - Diesel Range Organics	1699.99000	1700.00000	500.00000	N/A	N/A		08/11/11
X	0999	0000	Methyl Parathion	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Methyl methane sulfonate	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Methapyrilene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	m-Toluic Acid	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Kepone	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Isosafrole	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Isodrin	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	N-Nitroso-di-n-butylamine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Phenol-d6	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	Tetramethyllead	1.00000	330.00000	250.00000	N/A	N/A		08/11/11
X	0999	0001	Tetraethyllead	1.00000	330.00000	250.00000	N/A	N/A		08/11/11
X	0999	0000	Sym-Trinitrobenzene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Safrole	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	Pyridine	27.00000	670.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	Pyrene-d10	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Pronamide	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	O,O,O-Triethylphosphorothioate	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Phorate	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Fluorene-d10	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Phenacetin	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Perylene	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0004	Pentachloronitrobenzene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Pentachlorobenzene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	p-Toluic Acid	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	p-Phenylenediamine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	p-(Dimethylamino)azobenzene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_S **DefaultPrep:** BNA_S_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Soil **Units:** ug/Kg **Updated:** 08/11/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0000	Octachlorocyclopentene	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0002	Phthalic anhydride	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0002	alpha-Terpineol	38.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0005	Benzidine	130.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	Thionazin	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0004	Azobenzene	32.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0003	1,1´-Biphenyl	42.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	Aramite-2	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Aramite-1	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Aramite	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Hexachloropropene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	Aniline	24.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	Benzo(e)pyrene-d12	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0001	Acetophenone	31.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	Acenaphthylene-d8	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	a,a-Dimethylphenethylamine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	7,12-Dimethylbenz(a)anthracene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	5-Nitro-o-toluidine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0003	4-tert-Octylphenol	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	4-Nitroquinoline-1-oxide	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	4-Nitrophenol-d4	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Anthracene-d10	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Dibenz(a,j)acridine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	4-Methylphenol-d8	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Famphur	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Ethyl parathion	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Ethyl methane sulfonate	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Disulfoton	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Diphenylamine	29.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	Dimethylphthalate-d6	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Dimethoate	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Benzo(a)pyrene-d12	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Dibenzothiophene	0.00999	0.00999	0.00000	N/A	N/A		08/11/11

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_S **DefaultPrep:** BNA_S_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS **Conversion:** 1000.0000

Matrix: Soil **Units:** ug/Kg **Updated:** 08/11/2011

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0000	Benzo(e)pyrene	0.00999	0.00999	0.00000	N/A	N/A		08/11/11
X	0999	0000	Diallate	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Chlorobenzilate	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0002	Caprolactam	21.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0000	Bis(2-chloroisopropyl)ether	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	bis(2-Chloroethyl)ether-d8	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	Benzyl alcohol	35.00000	330.00000	80.00000	N/A	N/A	07/29/10	08/11/11
X	0999	0002	Benzoic acid	67.00000	670.00000	80.00000	N/A	N/A		08/11/11
X	0999	0000	Hexachlorophene	33.00000	330.00000	80.00000	N/A	N/A		08/11/11
X	0999	0001	Diethylenetriamine	33.00000	330.00000	80.00000	N/A	N/A		08/11/11

Table 3
(Table 5 from Published Method)

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-	Dimethyl phthalate
Ethyl methanesulfonate	phenethylamine	2,4-Dinitrophenol
2-Fluorophenol (surr)	2,4-Dimethylphenol	2,4-Dinitrotoluene
Hexachloroethane	Hexachlorobutadiene	2,6-Dinitrotoluene
Methyl methanesulfonate	Isophorone	Fluorene
2-Methylphenol	2-Methylnaphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Naphthalene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene	1-Naphthylamine
N-Nitroso-di-n-propylamine	Nitrobenzene-d_8 (surr)	2-Naphthylamine
Phenol	2-Nitrophenol	2-Nitroaniline
Phenol-d_6 (surr)	N-Nitrosodi-n-butylamine	3-Nitroaniline
2-Picoline	N-Nitrosopiperidine	4-Nitroaniline
	1,2,4-Trichlorobenzene	4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (surr)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

(surr) = surrogate

TABLE 5
(continued)

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
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-methylphenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene
Fluoranthene	p-Dimethyl aminoazobenzene	7,12-Dimethylbenz(a) anthracene
Hexachlorobenzene	Pyrene	Di-n-octyl phthalate
N-Nitrosodiphenylamine	Terphenyl-d_{14} (surr)	Indeno(1,2,3-cd) pyrene
Pentachlorophenol		3-Methylcholanthrene
Pentachloronitrobenzene		
Phenacetin		
Phenanthrene		
Pronamide		

(surr) = surrogate

Table 4
(Table 4 from Published Method)

TABLE 4

RECOMMENDED MINIMUM RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION USING THE SUGGESTED IONS FROM TABLE 1

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800

TABLE 4
(continued)

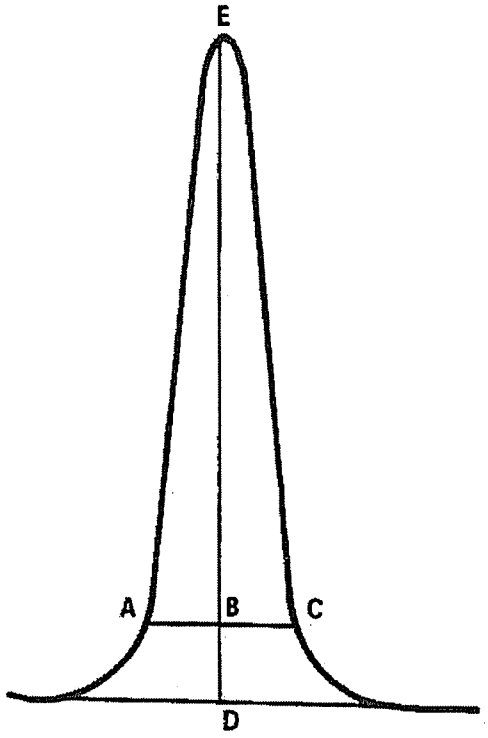
Semivolatile Compounds	Minimum Response Factor (RF)
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800

TABLE 4
(continued)

Semivolatile Compounds	Minimum Response Factor (RF)
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Figure 1

FIGURE 1
TAILING FACTOR CALCULATION



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Figure 2

Spectrum Analytical, Inc. RI Division

Data file : \\Avogadro\Organics\S4.I\120131.B\S4E9688.D
Lab Smp Id: DFTPP4H Client Smp ID: DFTPP4H
Inj Date : 31-JAN-2012 10:34
Operator : MMS SRC: MMS Inst ID: S4.i
Smp Info : DFTPP4H,DFTPP4H
Misc Info : 3
Comment :
Method : \\Avogadro\Organics\S4.I\120131.B\S4_dftppSOM.m
Meth Date : 13-Jan-2012 10:54 bmaczewska Quant Type: ESTD
Cal Date : Cal File:
Als bottle: 100 QC Sample: DFTPP
Dil Factor: 1.00000
Integrator: HP RTE Compound Sublist: all.sub
Target Version: 4.14 Sample Matrix: WATER
Processing Host: TARGET104

Concentration Formula: Amt * DF * Uf * Vf/Vi * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	ng unit correction factor
Vf	1.000	Volumetric correction factor
Vi	2.000	Injection Volume (uL)
Cpnd Variable		Local Compound Variable

RT	EXP RT	DLT RT	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/L)	CONCENTRATIONS FINAL (ug/L)	TARGET RANGE	RATIO
1 dftpp						CAS #: 5074-71-5		
1.940	2.361	-0.421	198	3016			0.00- 100.00	100.00
1.940	2.361	-0.421	51	1002			10.00- 80.00	33.22
1.940	2.361	-0.421	68	0	0.0	0.0	0.00- 2.00	0.00
1.940	2.361	-0.421	69	1247			0.00- 0.00	41.35
1.940	2.361	-0.421	70	0	0.0	0.0	0.00- 2.00	0.00
1.940	2.361	-0.421	127	1464			10.00- 80.00	48.54
1.940	2.361	-0.421	197	0	0.0	0.0	0.00- 2.00	0.00
1.940	2.361	-0.421	199	212			5.00- 9.00	7.03
1.940	2.361	-0.421	275	995			10.00- 60.00	32.99
1.940	2.361	-0.421	365	170			1.00- 0.00	5.64
1.940	2.361	-0.421	441	334			0.01- 99.99	90.27
1.940	2.361	-0.421	442	1952			50.00- 100.00	64.72
1.940	2.361	-0.421	443	370			15.00- 24.00	18.95

Data File: \\Avogadro\Organics\S4.I\120131.B\S4E9688.D

Date : 31-JAN-2012 10:34

Client ID: DFTPP4H

Instrument: S4.i

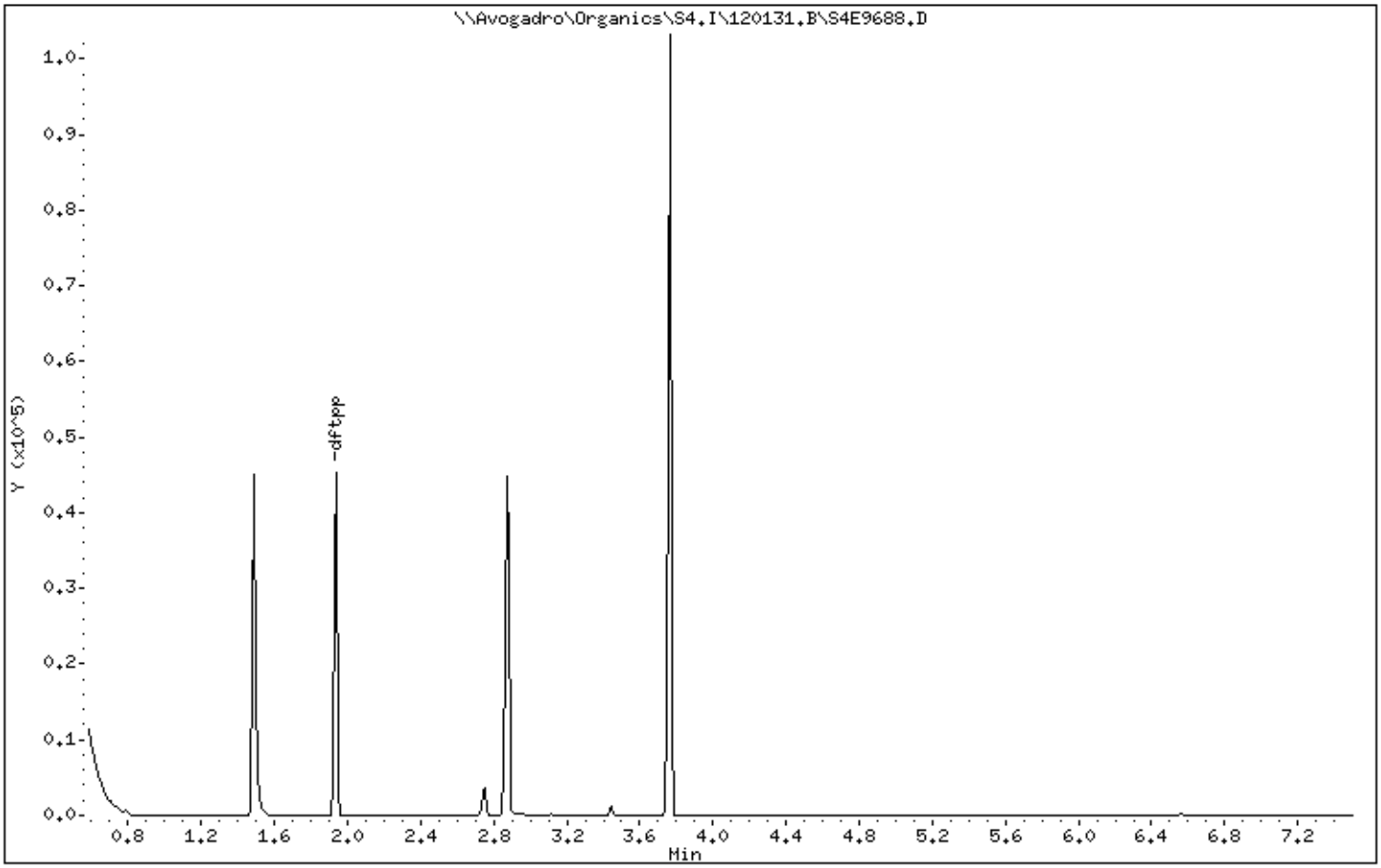
Sample Info: DFTPP4H,DFTPP4H

Volume Injected (uL): 2.0

Operator: MMS SRC: MMS

Column phase: RXi-5SILMS

Column diameter: 0.25



Data File: \\Avogadro\Organics\S4.I\120131.B\S4E9688.D

Date : 31-JAN-2012 10:34

Client ID: DFTPP4H

Instrument: S4.i

Sample Info: DFTPP4H,DFTPP4H

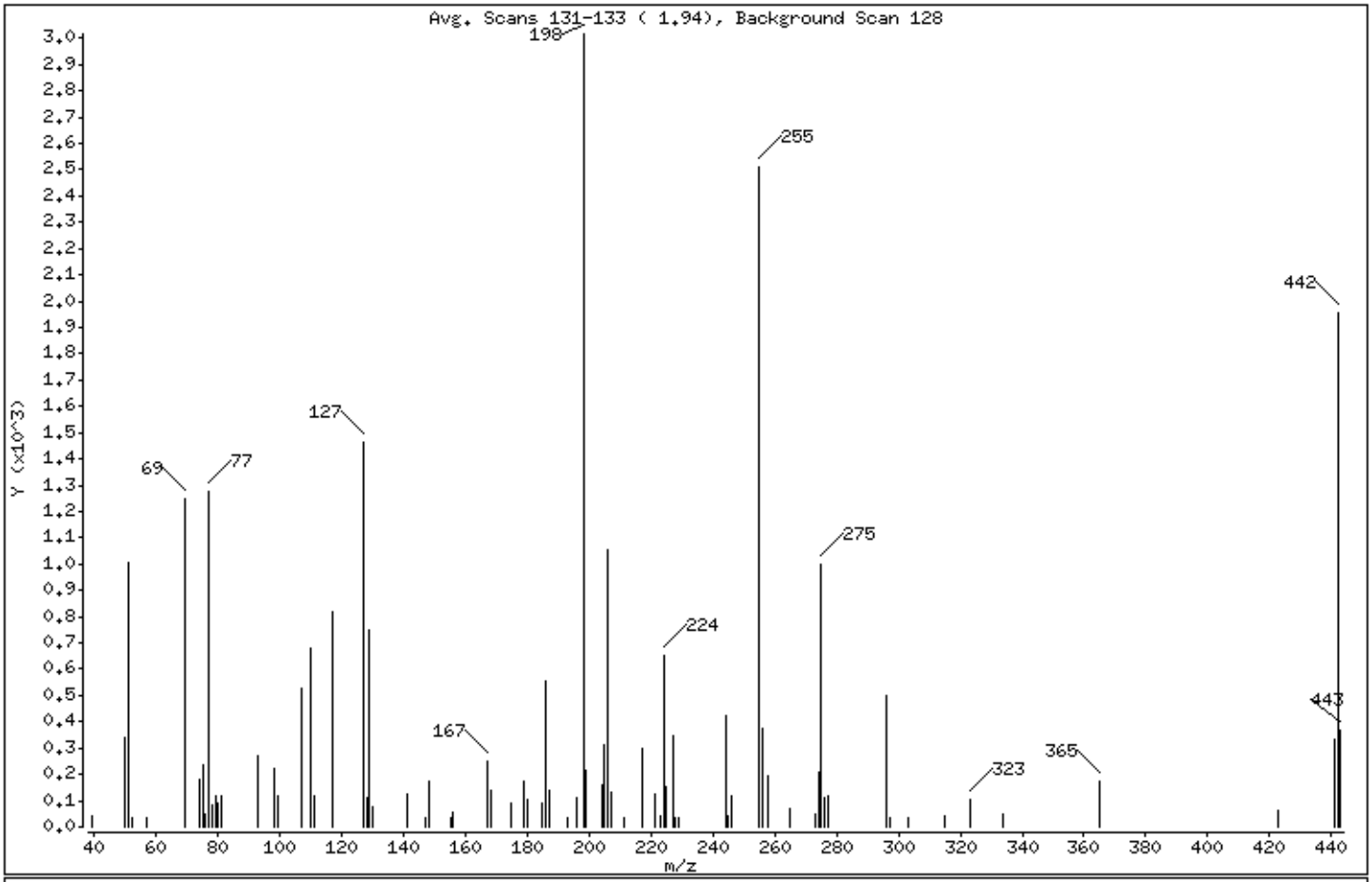
Volume Injected (uL): 2.0

Operator: MMS SRC: MMS

Column phase: RXi-5SILMS

Column diameter: 0.25

1 dftpp



m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
198	Base Peak, 100% relative abundance	100.00
51	10.00 - 80.00% of mass 198	33.22
68	Less than 2.00% of mass 69	0.00 (0.00)
69	Mass 69 relative abundance	41.35
70	Less than 2.00% of mass 69	0.00 (0.00)
127	10.00 - 80.00% of mass 198	48.54
197	Less than 2.00% of mass 198	0.00
199	5.00 - 9.00% of mass 198	7.03
275	10.00 - 60.00% of mass 198	32.99
365	Greater than 1.00% of mass 198	5.64
441	Present, but less than mass 443	11.07
442	50.00 - 100.00% of mass 198	64.72
443	15.00 - 24.00% of mass 442	12.27 (18.95)

Date : 31-JAN-2012 10:34

Client ID: DFTPP4H Instrument: S4.i

Sample Info: DFTPP4H,DFTPP4H

Volume Injected (uL): 2.0 Operator: MMS SRC: MMS

Column phase: RXi-5SILMS Column diameter: 0.25

Data File: S4E9688.D
Spectrum: Avg. Scans 131-133 (1.94), Background Scan 128
Location of Maximum: 198.00
Number of points: 78

m/z	Y	m/z	Y	m/z	Y	m/z	Y
39.00	42	117.00	815	198.00	3016	258.00	197
50.00	339	127.00	1464	199.00	212	265.00	67
51.00	1002	128.00	109	204.00	158	273.00	49
52.00	34	129.00	750	205.00	309	274.00	210
57.00	34	130.00	73	206.00	1051	275.00	995
69.00	1247	141.00	125	207.00	129	276.00	113
74.00	178	147.00	37	211.00	33	277.00	119
75.00	233	148.00	172	217.00	298	296.00	499
76.00	46	155.00	34	221.00	123	297.00	37
77.00	1279	156.00	53	223.00	42	303.00	35
78.00	84	167.00	250	224.00	654	315.00	41
79.00	119	168.00	136	225.00	154	323.00	107
80.00	89	175.00	91	227.00	346	334.00	48
81.00	118	179.00	174	228.00	37	365.00	170
93.00	269	180.00	101	229.00	34	423.00	59
98.00	224	185.00	90	244.00	425	441.00	334
99.00	120	186.00	553	245.00	40	442.00	1952
107.00	527	187.00	140	246.00	116	443.00	370
110.00	680	193.00	36	255.00	2509		
111.00	117	196.00	110	256.00	371		

Figure 3

Data File: \\avogadro\organics\S3.I\110623.B\S3H4311A.D
Report Date: 24-Jun-2011 10:21

Spectrum Analytical, Inc. RI Division

Data file : \\avogadro\organics\S3.I\110623.B\S3H4311A.D
Lab Smp Id: SSTD0253C Client Smp ID: SSTD0253C
Inj Date : 23-JUN-2011 13:17
Operator : PK   SRC: PK Inst ID: S3.i
Smp Info : SSTD0253C,SSTD0253C
Misc Info : 2,3
Comment   :
Method   : \\avogadro\organics\S3.I\110623.B\s3_8270C_N.m
Meth Date : 24-Jun-2011 10:21 S3.i Quant Type: ISTD
Cal Date : 19-MAY-2011 13:48 Cal File: S3H3775.D
Als bottle: 1 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: HP RTE Compound Sublist: allnew.sub
Target Version: 4.14
Processing Host: TARGET113

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi) * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Vo	1000.000	Volume of sample extracted (mL)
Vt	1000.000	Volume of final extract (uL)
Vi	1.000	Volume injected (uL)
Uf	1.000	ng unit correction factor
Cpnd Variable		Local Compound Variable

	Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)
	1 N-Nitrosodimethylamine	74	0.624	0.624	(0.233)	59864	25.0000	33
	2 Pyridine	79	0.624	0.624	(0.233)	103741	25.0000	34
$	3 2-Fluorophenol	112	1.468	1.468	(0.549)	61527	25.0000	24
	101 Benzaldehyde	77	2.178	2.178	(0.814)	56364	25.0000	23
$	5 Phenol-d5	99	2.499	2.499	(0.934)	90794	25.0000	25
	6 Phenol	94	2.515	2.515	(0.940)	99276	25.0000	24
	7 Aniline	93	2.333	2.333	(0.872)	113226	25.0000	25
	8 bis(2-Chloroethyl)Ether	93	2.451	2.451	(0.916)	72906	25.0000	23
	10 2-Chlorophenol	128	2.477	2.477	(0.926)	78018	25.0000	24
	11 1,3-Dichlorobenzene	146	2.595	2.595	(0.970)	85950	25.0000	24
*	12 1,4-Dichlorobenzene-d4	152	2.675	2.675	(1.000)	90804	40.0000	
	13 1,4-Dichlorobenzene	146	2.691	2.691	(1.006)	87350	25.0000	24
	117 2-Ethyl-1-hexanol	57	2.915	2.915	(1.090)	83091	25.0000	24
	15 Benzyl Alcohol	108	2.958	2.958	(1.106)	53081	25.0000	24
	16 1,2-Dichlorobenzene	146	2.862	2.862	(1.070)	84343	25.0000	24
	17 2-Methylphenol	108	3.220	3.220	(1.204)	73101	25.0000	23
	18 2,2'-oxybis(1-Chloropropane)	45	3.124	3.124	(1.168)	108474	25.0000	21
	99 Acetophenone	105	3.220	3.220	(1.204)	94047	25.0000	24
	19 N-Nitroso-di-n-propylamine	70	3.295	3.295	(1.232)	62036	25.0000	22
	20 4-Methylphenol	108	3.450	3.450	(1.290)	80507	25.0000	23

Data File: \\avogadro\organics\S3.I\110623.B\S3H4311A.D
Report Date: 24-Jun-2011 10:21

							AMOUNTS	
		QUANT SIG					CAL-AMT	ON-COL
	Compounds	MASS	RT	EXP RT	REL RT	RESPONSE	(ng)	(ng)
	21 Hexachloroethane	117	3.263	3.263	(1.220)	34334	25.0000	26
$	22 Nitrobenzene-d5	82	3.391	3.391	(0.785)	90649	25.0000	24
	23 Nitrobenzene	77	3.412	3.412	(0.790)	82056	25.0000	22
	24 Isophorone	82	3.770	3.770	(0.873)	161924	25.0000	23
	25 2-Nitrophenol	139	3.845	3.845	(0.890)	45988	25.0000	24
	26 2,4-Dimethylphenol	107	4.123	4.123	(0.954)	69677	25.0000	19
	27 bis(2-Chloroethoxy)methane	93	4.176	4.176	(0.967)	90715	25.0000	23
	28 Benzoic Acid	105	4.481	4.481	(1.037)	68751	25.0000	23
	29 2,4-Dichlorophenol	162	4.267	4.267	(0.988)	73377	25.0000	24
	30 1,2,4-Trichlorobenzene	180	4.278	4.278	(0.990)	79502	25.0000	24
*	31 Naphthalene-d8	136	4.320	4.320	(1.000)	363184	40.0000	
	32 Naphthalene	128	4.347	4.347	(1.006)	229438	25.0000	24
	115 alpha-Terpineol	59	4.491	4.491	(1.040)	55379	25.0000	23
	33 4-Chloroaniline	127	4.534	4.534	(1.049)	94961	25.0000	23
	34 Hexachlorobutadiene	225	4.593	4.593	(1.063)	46940	25.0000	26
	102 Caprolactam	113	5.004	5.004	(1.158)	30208	25.0000	21
	35 4-Chloro-3-Methylphenol	107	5.245	5.245	(1.214)	86206	25.0000	24
	36 2-Methylnaphthalene	142	5.181	5.181	(1.199)	164472	25.0000	23
	114 1-Methylnaphthalene	142	5.271	5.271	(1.220)	174908	25.0000	25
	38 Hexachlorocyclopentadiene	237	5.362	5.362	(0.875)	43231	25.0000	23
	112 1,2,4,5-Tetrachlorobenzene	216	5.357	5.357	(0.874)	84099	25.0000	27
	39 2,4,6-Trichlorophenol	196	5.533	5.533	(0.903)	60275	25.0000	25
	40 2,4,5-Trichlorophenol	196	5.587	5.587	(0.912)	66143	25.0000	26
$	41 2-Fluorobiphenyl	172	5.587	5.587	(0.912)	194030	25.0000	25
	98 1,1'-Biphenyl	154	5.661	5.661	(0.924)	201564	25.0000	25
	42 2-Chloronaphthalene	162	5.640	5.640	(0.921)	168097	25.0000	24
	43 2-Nitroaniline	65	5.795	5.795	(0.946)	63818	25.0000	24
	44 Dimethylphthalate	163	6.009	6.009	(0.981)	216171	25.0000	24
	45 2,6-Dinitrotoluene	165	6.035	6.035	(0.985)	48598	25.0000	22
	46 Acenaphthylene	152	5.993	5.993	(0.978)	283846	25.0000	25
	47 3-Nitroaniline	138	6.169	6.169	(1.007)	50950	25.0000	23
*	48 Acenaphthene-d10	164	6.126	6.126	(1.000)	245719	40.0000	
	49 Acenaphthene	153	6.153	6.153	(1.004)	174803	25.0000	24
	50 2,4-Dinitrophenol	184	6.254	6.254	(1.021)	23840	25.0000	18
	51 4-Nitrophenol	109	6.452	6.452	(1.053)	49997	25.0000	26
	53 2,4-Dinitrotoluene	165	6.366	6.366	(1.039)	68830	25.0000	22
	52 Dibenzofuran	168	6.302	6.302	(1.029)	244645	25.0000	24
	110 2,3,4,6-Tetrachlorophenol	232	6.457	6.457	(1.054)	59506	25.0000	26
	54 Diethylphthalate	149	6.607	6.607	(1.078)	223399	25.0000	23
	56 4-Chlorophenyl-phenylether	204	6.628	6.628	(1.082)	103286	25.0000	25
	55 Fluorene	166	6.586	6.586	(1.075)	212626	25.0000	24
	57 4-Nitroaniline	138	6.671	6.671	(1.089)	51379	25.0000	20
	58 4,6-Dinitro-2-methylphenol	198	6.692	6.692	(0.914)	43324	25.0000	22
	59 N-Nitrosodiphenylamine	169	6.746	6.746	(0.921)	191687	25.0000	25
	97 Azobenzene	77	6.751	6.751	(0.922)	263271	25.0000	29
$	60 2,4,6-Tribromophenol	330	6.794	6.794	(0.928)	31814	25.0000	30
	61 4-Bromophenyl-phenylether	248	7.013	7.013	(0.958)	66013	25.0000	27
	62 Hexachlorobenzene	284	7.024	7.024	(0.959)	70348	25.0000	28
	100 Atrazine	200	7.227	7.227	(0.987)	66624	25.0000	27
	63 Pentachlorophenol	266	7.221	7.221	(0.986)	43296	25.0000	24
	111 Pentachloronitrobenzene	237	7.211	7.211	(0.985)	33409	25.0000	26
*	64 Phenanthrene-d10	188	7.323	7.323	(1.000)	467674	40.0000	
	65 Phenanthrene	178	7.339	7.339	(1.002)	329393	25.0000	25
	66 Anthracene	178	7.376	7.376	(1.007)	333692	25.0000	25

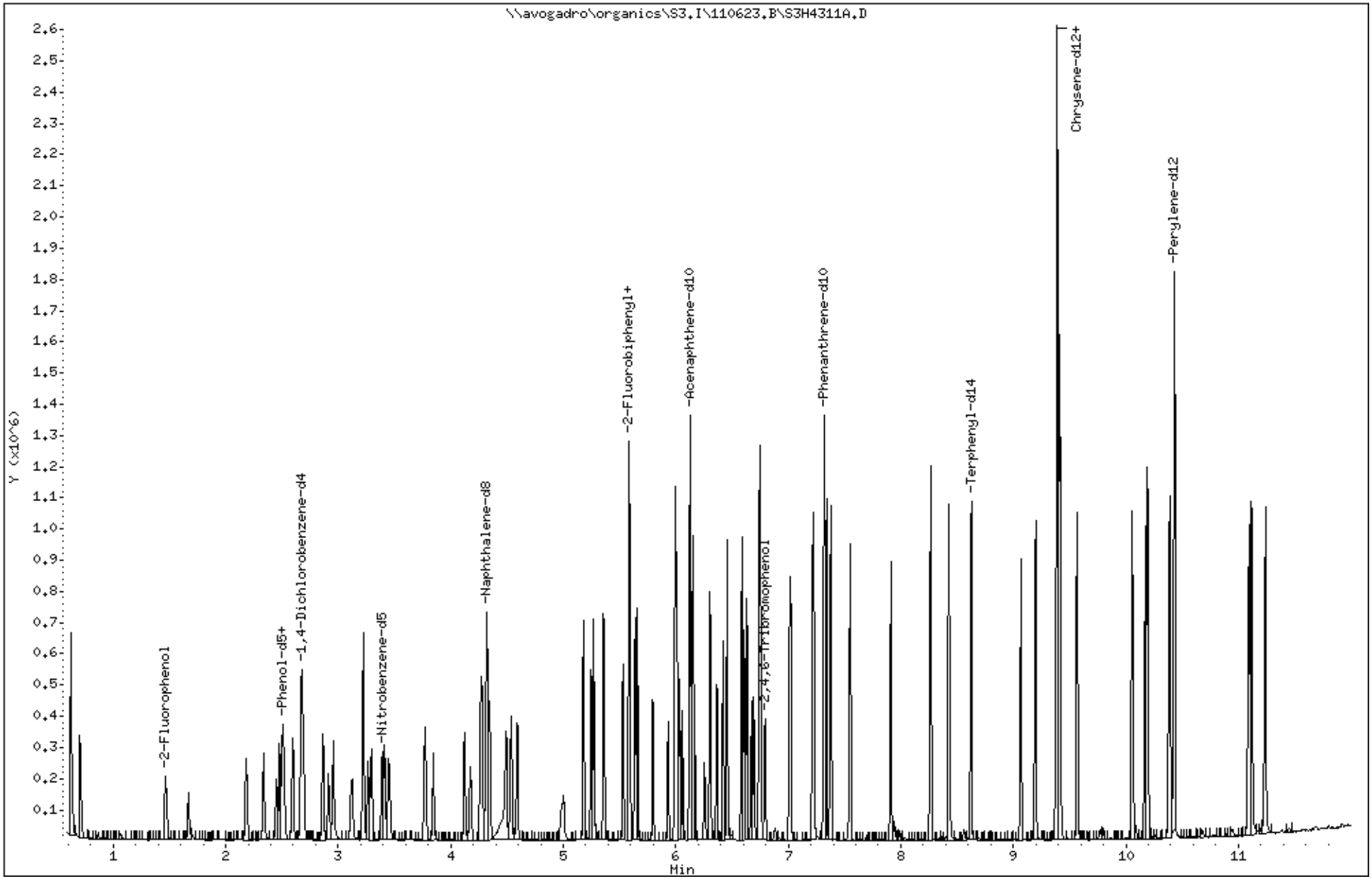
Data File: \\avogadro\organics\S3.I\110623.B\S3H4311A.D
Report Date: 24-Jun-2011 10:21

	Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)
67	Carbazole	167	7.547	7.547	(1.031)	320352	25.0000	24
68	Di-n-butylphthalate	149	7.910	7.910	(1.080)	389702	25.0000	25
69	Fluoranthene	202	8.263	8.263	(1.128)	376713	25.0000	25
70	Benzidine	184	8.434	8.434	(0.898)	33366	25.0000	4(a)
71	Pyrene	202	8.429	8.429	(0.898)	394939	25.0000	22
$ 72	Terphenyl-d14	244	8.626	8.626	(0.919)	263496	25.0000	23
73	Butylbenzylphthalate	149	9.070	9.070	(0.966)	194529	25.0000	22
74	3,3'-Dichlorobenzidine	252	9.417	9.417	(1.003)	121326	25.0000	24
78	bis(2-Ethylhexyl)phthalate	149	9.566	9.566	(1.019)	271559	25.0000	23
75	Benzo(a)anthracene	228	9.385	9.385	(0.999)	439964	25.0000	24
* 76	Chrysene-d12	240	9.390	9.390	(1.000)	649523	40.0000	
77	Chrysene	228	9.412	9.412	(1.002)	442157	25.0000	25
79	Di-n-octylphthalate	149	10.058	10.058	(0.964)	471970	25.0000	24
80	Benzo(b)fluoranthene	252	10.175	10.175	(0.975)	412987	25.0000	22
81	Benzo(k)fluoranthene	252	10.191	10.191	(0.977)	512836	25.0000	28
82	Benzo(a)pyrene	252	10.389	10.389	(0.996)	407525	25.0000	24
* 83	Perylene-d12	264	10.432	10.432	(1.000)	626653	40.0000	
84	Indeno(1,2,3-cd)pyrene	276	11.094	11.094	(1.063)	505364	25.0000	25
85	Dibenzo(a,h)anthracene	278	11.121	11.121	(1.066)	418551	25.0000	25
86	Benzo(g,h,i)perylene	276	11.239	11.239	(1.077)	438927	25.0000	25

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: \\avogadro\organics\S3.I\110623.B\S3H4311A.D
Date : 23-JUN-2011 13:17
Client ID: SSTD0253C
Instrument: S3.i
Sample Info: SSTD0253C,SSTD0253C
Volume Injected (uL): 1.0
Operator: PK SRC: PK
Column phase: Rxi-5Sil MS
Column diameter: 0.25



Attachment 1

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050. 2. RSD for RFs for CCCs: VOCs and SVOCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression–coefficient of determination (COD) r^2 ≥ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Attachment 2

Table G-5. LCS Control Limits for Volatile Organic Compounds SW-846 Method 8260 Solid Matrix[3] (continued)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Chloroform	98	9	70	125	65	135
Chloromethane	90	13	50	130	40	140
cis-1,2-Dichloroethene	96	10	65	125	55	135
cis-1,3-Dichloropropene	99	9	70	125	65	135
Dibromomethane	100	9	75	130	65	135
Dichlorodifluoromethane[4]	85	17	35	135	15	155
Ethylbenzene	101	9	75	125	65	135
Hexachlorobutadiene	98	15	55	140	40	155
Isopropylbenzene	103	9	75	130	70	140
m,p-Xylene	102	8	80	125	70	135
Methylene chloride	97	14	55	140	40	155
Naphthalene	84	14	40	125	25	140
n-Butylbenzene	101	12	65	140	50	150
n-Propylbenzene	99	12	65	135	50	145
o-Xylene	101	8	75	125	70	135
p-Isopropyltoluene	104	10	75	135	65	140
sec-Butylbenzene	97	11	65	130	50	145
Styrene	101	9	75	125	65	135
tert-Butylbenzene	99	11	65	130	55	145
Tetrachloroethene	103	12	65	140	55	150
Toluene	99	9	70	125	60	135
trans-1,2-Dichloroethene	100	11	65	135	55	145
trans-1,3-Dichloropropene	96	10	65	125	55	140
Trichloroethene	101	8	75	125	70	130
Trichlorofluoromethane	106	27	25	185	10	215
Vinyl chloride	92	11	60	125	45	140

Table G-6. LCS Control Limits for Semivolatile Organic Compounds SW-846 Method 8270 Water Matrix[5]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	75.0	9.5	45	105	35	115
Acenaphthene	77.6	10.1	45	110	35	120
Acenaphthylene	78.5	9.4	50	105	40	115
Anthracene	83.0	9.7	55	110	45	120
Benz[a]anthracene	82.7	8.9	55	110	45	120
Benzo[a]pyrene	81.3	9.5	55	110	45	120

[5] A number of sporadic marginal exceedances of the control limits are allowed depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, and N-nitrosopyrrolidine. Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section G.5.

Table G-6. LCS Control Limits for Semivolatile Organic Compounds SW-846 Method 8270 Water Matrix[5] (continued)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Benzo[b]fluoranthene	81.8	12.1	45	120	35	130
Benzo[k]fluoranthene	84.6	13.2	45	125	30	135
Benzo[g,h,i]perylene	80.5	14.1	40	125	25	135
Chrysene	82.1	8.9	55	110	45	120
Dibenz[a,h]anthracene	84.7	14.1	40	125	30	140
Fluoranthene	85.2	10.4	55	115	45	125
Fluorene	80.6	10.3	50	110	40	120
Indeno[1,2,3-cd]pyrene	84.3	13.6	45	125	30	140
Naphthalene	70.8	10.5	40	100	30	115
Phenanthrene	84.0	11.0	50	115	40	130
Pyrene	88.6	13.2	50	130	35	140
Phenolic/Acidic						
2,4,5-Trichlorophenol	79.7	10.3	50	110	40	120
2,4,6-Trichlorophenol	80.7	10.7	50	115	40	125
2,4-Dichlorophenol	76.3	9.6	50	105	40	115
2,4-Dimethylphenol	68.8	13.5	30	110	15	125
2,4-Dinitrophenol	75.8	20.6	15	140	10	160
2-Chlorophenol	71.3	11.4	35	105	25	115
2-Methylphenol	73.3	11.7	40	110	25	120
2-Nitrophenol	75.8	12.4	40	115	25	125
3-Methylphenol/4-Methylphenol	71.3	13.0	30	110	20	125
4,6-Dinitro-2-methylphenol	84.9	15.0	40	130	25	145
4-Chloro-3-methylphenol	78.6	10.7	45	110	35	120
Pentachlorophenol	77.6	13.3	40	115	25	130
Basic						
3,3'-Dichlorobenzidine	65.2	15.3	20	110	10	125
4-Chloroaniline	62.2	15.6	15	110	10	125
Phthalate Esters						
Bis(2-ethylhexyl) phthalate	84.2	14.0	40	125	30	140
Butyl benzyl phthalate	81.1	11.7	45	115	35	130
Di-n-butyl phthalate	84.8	10.3	55	115	45	125
Di-n-octyl phthalate	87.4	16.6	35	135	20	155
Diethyl phthalate	79.2	12.9	40	120	30	130
Dimethyl phthalate	75.9	16.9	25	125	10	145
Nitrosoamines						
N-Nitrosodi-n-propylamine	80.9	15.7	35	130	20	145
N-Nitrosodimethylamine	67.9	14.1	25	110	10	125
N-Nitrosodiphenylamine	79.6	10.6	50	110	35	120
Chlorinated Aliphatics						
Bis(2-chlorethoxy)methane	76.2	10.2	45	105	35	115
Bis(2-chloroethyl) ether	73.3	12.3	35	110	25	120
Bis(2-chloroisopropyl) ether	78.2	17.5	25	130	10	150
Hexachlorobutadiene	65.2	12.6	25	105	15	115
Hexachloroethane	60.9	11.1	30	100	15	105

Table G-6. LCS Control Limits for Semivolatile Organic Compounds SW-846 Method 8270 Water Matrix[5] (continued)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Halogenated Aromatics						
1,2,4-Trichlorobenzene	71.7	11.6	35	105	25	120
1,2-Dichlorobenzene	67.3	11.4	35	100	20	115
1,3-Dichlorobenzene	64.8	10.9	30	100	20	110
1,4-Dichlorobenzene	64.8	10.9	30	100	20	110
2-Chloronaphthalene	76.5	9.3	50	105	40	115
4-Bromophenyl phenyl ether	82.9	10.2	50	115	40	125
4-Chlorophenyl phenyl ether	80.6	10.3	50	110	40	120
Hexachlorobenzene	82.3	10.0	50	110	40	120
Nitroaromatics						
2,4-Dinitrotoluene	84.3	11.2	50	120	40	130
2,6-Dinitrotoluene	82.7	11.3	50	115	35	130
2-Nitroaniline	81.8	11.2	50	115	35	125
3-Nitroaniline	72.6	17.7	20	125	10	145
4-Nitroaniline	77.2	13.7	35	120	20	130
Nitrobenzene	76.8	10.8	45	110	35	120
Neutral Aromatics						
Carbazole	82.5	11.4	50	115	35	130
Dibenzofuran	80.3	8.8	55	105	45	115
Others						
1,2-Diphenylhydrazine	84.8	9.4	55	115	45	120
Benzyl alcohol	71.0	13.8	30	110	15	125
Isophorone	81.0	10.5	50	110	40	125

Table G-7. LCS Control Limits for Semivolatile Organic Compounds SW-846 Method 8270 Solid Matrix[6]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	77.3	10.0	45	105	35	115
Acenaphthene	77.3	10.3	45	110	35	120
Acenaphthylene	75.7	10.4	45	105	35	115
Anthracene	79.9	9.0	55	105	45	115
Benz[a]anthracene	81.6	9.8	50	110	40	120
Benzo[a]pyrene	80.7	10.3	50	110	40	120
Benzo[b]fluoranthene	79.7	11.4	45	115	35	125
Benzo[k]fluoranthene	83.8	12.9	45	125	30	135
Benzo[g,h,i]perylene	81.8	14.7	40	125	25	140
Chrysene	82.6	9.9	55	110	45	120

[6] A number of sporadic marginal exceedances (ME) of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, 1,2-Diphenylhydrazine, and N-nitrosopyrrolidine. Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section G.5.

Table G-7. LCS Control Limits for Semivolatile Organic Compounds
SW-846 Method 8270 Solid Matrix[6] (continued)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Dibenz[a,h]anthracene	82.9	13.9	40	125	25	140
Fluoranthene	83.9	10.1	55	115	45	125
Fluorene	78.3	9.8	50	110	40	115
Indeno[1,2,3-cd]pyrene	79.7	13.8	40	120	25	135
Naphthalene	73.4	11.1	40	105	30	120
Phenanthrene	80.1	10.0	50	110	40	120
Pyrene	84.4	12.8	45	125	35	135
Phenolic/Acidic						
2,4,5-Trichlorophenol	80.1	10.4	50	110	40	120
2,4,6-Trichlorophenol	76.3	11.0	45	110	30	120
2,4-Dichlorophenol	77.2	10.9	45	110	35	120
2,4-Dimethylphenol	67.3	11.9	30	105	20	115
2,4-Dinitrophenol	72.6	20.0	15	130	10	150
2-Chlorophenol	74.7	10.3	45	105	35	115
2-Methylphenol	71.7	10.6	40	105	30	115
2-Nitrophenol	76.2	11.5	40	110	30	120
3-Methylphenol/4-Methylphenol	73.9	10.9	40	105	30	120
4,6-Dinitro-2-methylphenol	83.1	18.0	30	135	10	155
4-Chloro-3-methylphenol	79.5	11.1	45	115	35	125
4-Nitrophenol	77.0	20.2	15	140	10	160
Pentachlorophenol	71.9	15.6	25	120	10	135
Phenol	69.7	10.2	40	100	30	110
Phthalate Esters						
Bis(2-ethylhexyl) phthalate	87.4	13.3	45	125	35	140
Butyl benzyl phthalate	86.4	12.3	50	125	35	135
Di-n-butyl phthalate	83.2	9.1	55	110	45	120
Di-n-octyl phthalate	86.4	15.2	40	130	25	145
Diethyl phthalate	82.2	10.6	50	115	40	125
Dimethyl phthalate	79.6	10.2	50	110	40	120
Nitrosoamines						
N-Nitrosodi-n-propylamine	76.8	12.3	40	115	30	125
N-Nitrosodimethylamine	66.1	15.9	20	115	10	130
N-Nitrosodiphenylamine	82.4	11.1	50	115	40	125
Chlorinated Aliphatics						
Bis(2-chlorethoxy)methane	75.5	10.9	45	110	30	120
Bis(2-chloroethyl) ether	71.1	11.2	40	105	25	115
Bis(2-chloroisopropyl) ether	68.4	15.7	20	115	10	130
Hexachlorobutadiene	78.2	12.9	40	115	25	130
Hexachloroethane	71.9	12.6	35	110	20	120
Halogenated Aromatics						
1,2,4-Trichlorobenzene	77.4	11.2	45	110	30	120
1,2-Dichlorobenzene	70.9	8.7	45	100	35	105
1,3-Dichlorobenzene	69.7	10.3	40	100	30	110
1,4-Dichlorobenzene	69.0	11.4	35	105	25	115

Table G-7. LCS Control Limits for Semivolatile Organic Compounds SW-846 Method 8270 Solid Matrix (continued)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
2-Chloronaphthalene	75.2	9.9	45	105	35	115
4-Bromophenyl phenyl ether	81.7	11.8	45	115	35	130
4-Chlorophenyl phenyl ether	79.6	10.7	45	110	35	120
Hexachlorobenzene	82.5	11.7	45	120	35	130
Nitroaromatics						
2,4-Dinitrotoluene	82.0	11.4	50	115	35	130
2,6-Dinitrotoluene	80.2	10.7	50	110	35	125
2-Nitroaniline	81.0	12.2	45	120	30	130
3-Nitroaniline	68.8	13.8	25	110	15	125
4-Nitroaniline	73.6	13.1	35	115	20	125
Nitrobenzene	77.2	11.9	40	115	30	125
Neutral Aromatics						
Carbazole	80.4	12.3	45	115	30	130
Dibenzofuran	77.1	8.8	50	105	40	110
Others						
Benzyl alcohol	70.9	17.4	20	125	10	140
Isophorone	77.0	11.4	45	110	30	125

Table G-8. LCS Control Limits for Chlorinated Herbicides SW-846 Method 8151 Water Matrix[7]

Analyte	Median	Lower Control Limit	Upper Control Limit
2,4-D	88	35	115
2,4-DB	99	45	130
2,4,5-T	83	35	110
2,4,5-TP (Silvex)	87	50	115
Dalapon	62	40	110
Dicamba	86	60	110
Dichloroprop	91	70	120
Dinoseb	65	20	100
MCPA	93	60	145

[7] LCS control limits were generated using non-parametric statistics (see section G.1 for further explanation). LCS control limits are not available for MCPP. Sufficient data to perform statistically significant analyses were not received for the analyte during the LCS study.

Attachment 3
Corrective Action and Documentation Examples

OCCURRENCE	ACTION	DOCUMENTATION
1. Initial calibration does not meet QC criteria.	1. Investigate source of problem, determine if source is an instrument problem or a standard solution problem. If problem is with a single point of the ICAL, reanalyze the bad standard and reevaluate. Depending on extent of problem, major maintenance or invoking manufacturer service contract for instrument repair will be performed.	1. Notation in instrument run log book, and if necessary notation in instrument maintenance log book.
2. Initial calibration verification check does not meet QC criteria.	2. Investigate source of problem, determine if source is with ICAL or ICV, is it an instrument problem or a standard solution problem, reanalyze ICV or perform new ICAL.	2. Notation in instrument run log book, and if necessary notation in instrument maintenance log book. If source determined to be bad standard solution, formal corrective action form must be initiated.
3. Continuing calibration verification check does not meet QC criteria.	3. Investigate source of problem. If source is instrument, perform instrument maintenance and reanalyze CCV. If CCV still will not pass, repeat the above, or perform new initial calibration. Depending on extent of problem, major maintenance or invoking manufacturer service contract for instrument repair will be performed.	3. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
4. GC/MS tune does not meet method criteria.	4. Investigate source of problem, evaluate instrument response to cal gas (PFTBA), when instrument response to PFTBA is improved, re-inject DFTPP tune.	4. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
5. Method blank contains target compound above reporting limit (or 5x RL for common contam). Check if samples contain 10X the amount in MB as the data may be reportable.	5. Investigate source of problem. Reanalyze all affected samples. If reanalysis is within holding time, report only these analyses. If they are beyond holding time, report both sets and notify project manager. If contaminant is not present in samples, data may be released with commentary. 6. Investigate source of problem. If it is determined to be an	5. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book. 6. If only reanalysis is reported, notation in instrument run log.

6. Surrogate standard outside of acceptable range.	instrument problem, reanalyze sample. If it is determined to be a preparation problem, re-extract/re-analyze the sample. If it can be determined to be an obvious matrix problem (masking of surrogate by target or non-target compound at significantly greater concentration, excessive hydrocarbons in sample, other knowledge of sample matrix, etc.) the sample may be reanalyzed at dilution to reduce interference or reported with notation in narrative, depending on project objectives.	If both sets of data are to be reported, notation in instrument run log, re-extraction request form, preparation logbooks, commentary on data review checklist to be included in project narrative, flagging all non-compliant values on Form 2 of data report. If source of problem found to be systematic (bad spike solution, etc). A formal corrective action form must be initiated.
7. Compound out of acceptance range in laboratory control sample.	7. Investigate source of problem. If LCS is acceptable per method/SOP specifications, associated sample data can be reported. If LCS recovery is above upper QC limit, and if analyte is not detected in associated samples, data may be flagged and reported. If LCS is not acceptable per method/SOP requirements, re-extract all associated samples. If insufficient sample volume, notify project manager to discuss with client, report initial data if no other sample can be provided.	7. If LCS is acceptable per method/SOP, flag all compounds out of range on Form 3 of data report, if samples are reanalyzed within holding times, note in instrument run logbook. If samples are beyond holding time and both sets of data are to be reported, note in instrument run logbook and commentary in data review checklist to be included in project narrative. If reanalysis cannot be performed due to insufficient sample, commentaries in data review checklist to be included in project narrative. If source of problem found to be systematic (bad spike solution, etc). A formal corrective action form must be initiated.
8. Compound in sample exceeds upper calibration standard concentration.	8. Reanalyze sample at dilution. If calibration limit exceedence is the only QC problem, report both initial and dilution analyses. If initial analysis has multiple QC problems, evaluate further to determine if initial run is to be reported (often this cannot be determined until the results of the dilution are evaluated). Instrument must be shown to be free of carryover contamination prior to acceptable analysis of next sample. If running instrument-using autosampler, evaluate following sample. If following sample contains less than reporting limit of compound, the analysis is valid, and no instrument blank is required. If following sample(s) contain compound (typically in decreasing concentration—carryover typically	8. Notation in instrument run log book. If initial analysis is reported, flag compound exceeding calibration limit with "E" on data report and commentary on data review checklist to be included in project narrative. If only diluted analysis is to be reported, commentary in data review checklist to be included in project narrative. If both initial and dilution are to be reported, all of the above.

	occurs at 1% of concentration of high sample in following analysis, with effect more pronounced for later-eluting compounds. Effected samples must be reanalyzed if sufficient volume exists.	
9. Instrument blank (GC) contains contamination above QC criteria.	9. Investigate source of problem, decontaminate purge and trap instrument, reanalyze all effected samples.	9. Notation in instrument run logbook, and if instrument maintenance performed, in instrument maintenance logbook.
10. Matrix spike recovery out of QC range.	10. Evaluate problem. If duplicate spike (MSD) shows same effect, it is generally matrix interference. If concentration of spike analyte is significantly (approx. 4 times) greater in unspiked sample, this is matrix interference masking quantitation of spike concentration. If source cannot be determined, reanalyze spike sample.	10. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.
11. Duplicate (or MSD) relative percent difference exceeds QC limit.	11. Evaluate problem. If concentration of analyte is close to reporting limit, variation of analysis is acceptable. If sample is soil or other heterogeneous matrix, high RPD is typical. If sample is a typically homogeneous matrix, reanalyze duplicate sample.	11. Flag RPD on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.
12. Internal Standard areas exceed QC criteria. (-50% to +100%) A. CCV. B. QC (blank, LCS) C. Samples and MS/MSD.	12. A. Evaluate problem. Re-analyze CCV. If the CCV does not meet criteria, re-analyze the initial calibration and proceed with CCV/QC/samples. B. Evaluate CCV and QC. As blank and LCS are “interference-free matrix” the IS areas should be within the same limits as the CCV. Evaluate for potential problems. If time allows (data not required on a rush basis), reanalyze QC prior to sample analysis. If insufficient time due to client deadline, data may be reported (as they meet method requirements) but the issue should be noted for the data reviewer and for the client. C. Evaluate CCV and QC. The IS areas may indicate a	12. A. Document in the analytical run log. B. If the criteria are met after re-analysis, document in run log. If the criteria have not been met and the results of the sample batch is reported, document in the run log and the Corrective Action Logbook. Have the supervisor review situation, initial/date, and include a comment on the data review checklist for the data reviewer and for inclusion in the narrative information to the client. C. If the CCV and QC meet criteria, document in the run log and on the package checklist for inclusion in the narrative submitted to the client. Note that certain

	potential problem, or matrix interference. If the CCV and batch QC meet criteria, document and report results as matrix interference. In particular, if the recovery of the surrogate standard associated with the IS compound is within the recovery range, then the internal standard method is effectively quantifying the compounds. If the associated surrogate is outside of the recovery criteria, the IS issue is impacting quantitation. Evaluate whether this indicates a potential high or low bias for the associated compound results (low IS=high surrogate=high bias; high IS=low surrogate=low bias) This requires notation and communication of the effect to the data reviewer and the client. Based on the severity of the problem, discuss with supervisor, technical director and /or reanalyze effected samples. If results are reported as is, document per 12C.	compounds may be potentially high bias or potentially low bias due to IS recoveries outside of range. If QC and samples do not meet criteria and the results are reported, document in the run log, document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer and for inclusion in the narrative.

Attachment 4

Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
GC/MS Tunes with DFTPP	Inter-laboratory consistency and comparability	(1) Criteria for DFTPP listed in Table 3 of SW-846 8270C (the same criteria must be used for all analyses) (2) Every 12 hours (3) DDT breakdown should be evaluated and should be <20%. (4) Pentachlorophenol and benzidine peak tailing should be evaluated. Peak tailing factor must be <3 for benzidine and <5 for pentachlorophenol. *NOTE: Tune must be performed in full scan mode for SIM analyses.*	No	Perform instrument/injection port maintenance as necessary; retune instrument	Suspend all analyses until tuning non-compliance is rectified. Report DDT breakdown and peak tailing exceedances in the case narrative.
Initial Calibration	Laboratory Analytical Accuracy	(1) Minimum of 5 standards (2) Low standard must be ≤ reporting limit (3) *Full scan:* %RSD should be ≤15 or "r" should be ≥0.99 for all compounds except CCCs which must be ≤30 % RSD or "r" ≥0.99 *SIM:* %RSD should be ≤20 or "r" should be ≥0.99 for all compounds (4) Must contain all target analytes (5) If regression analysis is used, the curve must not be forced through the origin. (6) *SIM:* Laboratory must monitor a minimum of two ions per analyte (the primary ion or quantitation ion and a minimum of one confirmation ion); this is required for all target analytes, surrogates and internal standards	No	Recalibrate as required by method (1) if any of CCC %RSDs >30 or any of CCC "r" <0.99 or (2) if >20% of remaining analytes have %RSDs >30 or "r" <0.99.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds in case narrative. If the average response factor or linear regression are not used for analyte quantitation (e.g., use of a quadratic equation), this must be noted in the case narrative with a list of the affected analytes.
Continuing Calibration (CCAL)	Laboratory Analytical Accuracy	(1) Every 12 hours prior to the analysis of samples (2) Concentration level near midpoint of curve (3) Must contain all target analytes (4) *Full scan:* Percent difference or percent drift must be ≤20 for CCCs and should be ≤30 for other compounds *SIM:* Percent difference or percent drift should be ≤30 for all compounds	No	Recalibrate as required by method (1) if %D of any of CCCs >20 or (2) if %D of >10% of other analytes >30.	Report non-conforming compounds in case narrative.

Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Method Blanks	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent (2) Matrix-specific (e.g., water, soil) (3) Target analytes must be < RL except for common laboratory contaminants (such as phthalates) which must be <5x RL	Yes	Locate source of contamination; correct problem; re-extract associated samples if uncommon contaminants are present in the method blank.	(1) Report non-conformances in case narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" flag or some other convention, should qualify the sample results. Blank contamination should also be documented in the case narrative. (3) If re-extraction is performed within holding time, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
Laboratory Control Spikes (LCSs)	Laboratory Method Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Prepared using standard source different than used for initial calibration (3) Concentration level should be between low and mid-level standard (4) Must contain all target analytes (5) Matrix-specific (e.g., soil, water) (6) Percent recoveries must be between 40 – 140 for the base-neutral compounds and between 30 -130 for the acid compounds (7) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above.	Yes	Recalculate the percent recoveries; Re-extract associated samples if >20% of all analytes fall outside the acceptance criteria or if >15% of analytes from a particular class (base-neutral or acid) fall outside the acceptance criteria.	(1) Report non-conformances in case narrative. (2) Individual laboratories should identify and document "difficult" (**) analytes for which laboratory-determined recovery ranges routinely exceed the 100 ± 30% criterion. Exceedances for these "difficult" analytes should be qualified in case narrative. Analytical data to support the "difficult" analyte classification are to be available for review during an audit. (3) If re-extraction is performed within holding time, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Method Precision	(1) Every 20 samples or for each new tune clock, whichever is more frequent. (2) Prepared using same standard source and concentration as LCS. (3) Must contain all target analytes. (4) Recommended to be run immediately after LCS in analytical sequence. (5) Laboratory–determined percent recoveries must be between 40 – 140 for the base-neutral compounds and between 30 -130 for the acid compounds (6) Matrix-specific (e.g., soil, water,. etc.) (7) Laboratory–determined Relative Percent Difference (RPD) must be ≤20 for waters and ≤30 for solids except for "difficult" (**) analytes which must be ≤ 50.	Yes	Recalculate RPD; Locate source of problem; Narrate non-conformances	(1) Locate and rectify source of non-conformance before proceeding with the analyses of subsequent sample batches. (2) Individual laboratories must identify and document "difficult" (**) analytes for which laboratory-determined RPDs routinely exceed the ≤ 25 criterion. (3) Exceedances for these "difficult" analytes must be qualified in Environmental Laboratory case narrative. Analytical data to support the "difficult" analyte classification must be available for review during an audit. (4) Narrate non-conformances

Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
MS/MSDs	Method Accuracy in Sample Matrix Method Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data-user) (2) Matrix-specific (3) Prepared by fortifying field sample with standard from source different than source used for initial calibration (4) Concentration level should be between low and mid-level standard (5) Must contain all target analytes. (6) Percent recoveries should be between 40 – 140 for the base-neutral compounds and between 30 -130 for the acid compounds, or develop laboratory in-house limits. (7) RPDs should be ≤20 for waters and ≤30 for solids	Yes Only when requested by the data-user,	Check LCS; if recoveries acceptable in LCS, evaluate alternate cleanup techniques for samples associated with MS/MSD and/or narrate non-conformance.	Note exceedances in case narrative.
Surrogates	Accuracy in Sample Matrix	(1) Minimum of 3 base-neutral and 3 acid, at retention times across GC run Recommended base-neutral surrogates: nitrobenzene-d5, 2-fluorobiphenyl, terphenyl-d14 Recommended acid surrogates: phenol-d5, 2-fluorophenol, 2,4,6-tribromophenol *SIM Note:* Surrogates used must be representative of compound class of target analytes (e.g., use base-neutral surrogates if analyzing for PAHs, use acid surrogates if analyzing for pentachlorophenol). (2) Percent recoveries in soil must be between 30-130 for all surrogates. Percent recoveries in water must be between 30-130 for base-neutral surrogates and between 15-110 for acid surrogates. (3) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above.	Yes	If two or more surrogates for any one fraction (base-neutral or acid) are outside limits or if any one surrogate recovers at <10%, reextract the sample. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.	(1) Note exceedances in case narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory should report results of both extractions. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial and re-extraction. (5) If sample is not re-extracted due to obvious interference, the laboratory must provide the chromatogram in the data report.

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Internal Standards	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) *Full scan:* Minimum of 6 at retention times across GC run. *SIM:* Number of internal standards used will be dependent on the analytes of interest. Internal standards must elute in close proximity to the analytes of interest. (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard (Section 5.4.2 of 8270C) (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard	No	If one or more internal standards are outside limits, re-analyze sample unless obvious interference present (e.g., UCM)	(1) Note exceedances in case narrative. (2) If re-analysis yields similar internal standard non-conformances, the laboratory should report both results of both analyses. (3) If re-analysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on internal standard calibration. (2) The laboratory must use the average response factor or linear regression curve generated from the associated initial calibration for quantitation of each analyte (3) The internal standard used for quantitation shall be the one nearest the retention time of the subject analyte.	NA	NA	If the average response factor or linear regression are not used for analyte quantitation (e.g. quadratic equation), this must be noted in the case narrative with a list of the affected analytes.

Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported (3) Refer to Section 3.3, TIC Compounds by GC/MS for guidance			(1) Qualification of the data is required if reporting values below the sample-specific reporting limit. (2) Complete analytical documentation for diluted and undiluted analyses is to be available for review during an audit. (3) TICs will be evaluated at the discretion of the LSP consistent with the guidelines presented in Appendix II B–3. (4) The performance of dilutions must be documented in the case narrative.

GC/MS = Gas Chromatography/Mass Spectrometry
DFTPP = Decafluorotriphenylphosphine
MS/MSDs = Matrix Spikes/Matrix Spike Duplicates
%RSD = Percent Relative Standard Deviation
UCM = Unresolved Complex Mixture

"r" = Correlation Coefficient
CCCs = Calibration Check Compounds
RPDs = Relative Percent Differences
TIC = Tentatively Identified Compound
NA = Not Applicable

Potentially "difficult" analytes include: dimethyl phthalate, 4-nitrophenol, phenol, 4-methylphenol, 2-methlphenol, 2,4-dinitrophenol, pentachlorophenol, and 4-chloroaniline

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by Selective Ion Monitoring (SIM) using Modified Method SW-846 8270

Contents SOP NO. 70.0033

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		8/22/11
Quality Assurance Director		8/22/11
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	Analyst	9/1/12			

Revision Record

Revision Date	Revision Description	Comments	Initials
3/19/03	Added control page and renamed SOP	Was SOP O33A	
12/20/07	Updated SOP to reflect SW846 methods	SW8270D, full edit	SBL
10/18/10	Updated ICV limits based on QSM4.1	Minor edit	SBL
07/28/11	Lab name change, added new sect12 and info about zacq,	Full revision	SBL
8/12/11	Intermediate and working std have 12 month not 6 month HT	Minor	SBL
8/18/11	Add optional 1,4-dioxane QC. Replaced Figure 2 with example LIMS std log. Replaced tune figure with correct ranges	full	SBL
9/6/12	Updated instrument configs	Minor	SBL

Procedure Superseded By: _____ Date: _____
Procedure Discontinued By: _____ Date: _____
Procedure Archived By: _____ Date: _____

SPECTRUM ANALYTICAL, INC.

Featuring Hanibal Technology
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by Selective Ion Monitoring (SIM) using Modified Method SW-846 8270

Rev. 7

	Signature	Date
QA Director:	Thomas B. Lawler	8/22/11
Lab Director:		8/22/11
Effective Date:	8/29/11	

SPECTRUM ANALYTICAL, INC.

Featuring Hanibal Technology
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by Selective Ion Monitoring (SIM) using Modified Method SW-846 8270 Rev 7

1. Scope and Application

This Standard Operating Procedure (SOP) describes the analysis of Semivolatile Polycyclic Aromatic Hydrocarbons (PAHs) in aqueous and solid sample using gas chromatography/mass spectrometry (GC/MS) and Selective Ion Monitoring (SIM). The SOP follows the analyses as discussed in current USEPA SW-846 Final Update IV, Method 8270D.

The semivolatile Polycyclic Aromatic Hydrocarbons that can be analyzed using this SOP are listed in **Table 1**. Additional SW-846 8270 semivolatile compounds can be analyzed by this method.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors** review the logbooks and data generated from this procedure and approve all reported results. The **Project Manager** evaluates laboratory reports for reasonableness of the results and signs the reports. The **QA Director** reviews quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure/Instrumentation

3.1 The samples are extracted using appropriate sample extraction methods (see SOPs for sample extraction) and, if necessary, sample clean-up procedures.

3.2 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 connected to the gas chromatograph.

3.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by coelution of the ions that are selected at the correct retention times. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a minimum of a five-point calibration curve.

3.4 A list of acronyms used in this SOP is included in **Section 20** of the current QAP.

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. In some instances, clients will provide their own containers. For semivolatile organic compound analysis by the SIM method, water samples are collected in 1- liter amber glass bottles. Solid samples are collected in 8-ounce amber glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 Sample extracts are transferred to the semi-volatile organics analysis lab with appropriate sample preparation information. Extracts are stored at 4° ± 2°C, protected from light, in sealed vials (screw-cap or crimp-cap) equipped with un-pierced PTFE-lined septa and stored in a separate location from the analytical standards.

4.3 The holding time of the extracts for semivolatile organic compounds analysis by the SIM method is 40 days from date of sample extraction. The sample preparation holding times are covered in the corresponding extraction procedures SOPs.

5. Interferences and Potential Problems

5.1 Evaluate the raw GC/MS data to verify that interferences were not introduced during the extraction and/or clean up of the samples.

5.2 The SIM method is not appropriate for multi-component analytes (Aroclor, toxaphene, chlordane, etc.). Refer to Methods 8081 and 8082.

5.3 This method is not appropriate for some of the routine SW8270 compounds. Semivolatile PAH are routinely analyzed by the SIM method.

6. Equipment and Apparatus

6.1 Equipment: There are four GC/MS in the semivolatile organic analysis lab. The instruments used in the laboratory include two HP Model 6890 GCs interfaced to ~~a~~ HP Model 5973 MSs, ~~and~~ one Hewlett Packard (HP) Model 7890A GC interfaced to a HP Model 5973 MS and one Hewlett Packard (HP) Model 7890A GC interfaced to a HP Model 5975 MS. IBM-compatible PCs with EnviroQuant Software are used to handle data acquisition. The resultant data are processed using Target software from ThruPut Corporation. Please note that while HP's instrumentation division has been renamed Agilent Corporation, all of the instruments are referred as HP in this SOP.

6.1.1 The HP GC is fitted with an electron pressure controller (EPC) to allow constant carrier gas flow during the temperature ramp.

6.1.2 A 30m x 0.25mm ID (0. 25 um film thickness) Rxi-5SilMS fused silica capillary columns (Restek) are used for the analyses.

6.1.3 Model 7683 auto-samplers are used for sample injection for 6890 GCs. A CTC leap autosampler is used for sample injection for HP 7890A GC, and a HP Model 7693 auto-sampler is used for the other HP 7890A GC.

6.2 Instrument operating conditions are as follows: In the event that these conditions are changed, Enviroquant Data Acquisition methods containing the actual GC operating conditions are copied and sent to the network along with all GC/ECD raw data files. They are located in a folder in the sequence batch called “Zacq”.

General Gas Chromatography Conditions

Carrier Gas	Helium (99.999%)
Column Flow	about 1 mL/minute
Injector Temperature	290°C
Transfer Line Temperature	280°C
Injection Volume	1 µL

GC Temperature program

Ramp	Rate	Initial Temp	Hold Time	Run Time
1		45°C	2.0 min	
2	15°C/min	225°C	0 min	
3	25°C/min	310°C	12.25min	29.65min

General Mass Spectrometry Conditions

Mass Range	SIM scans specific to the masses for the individual compounds and surrogate.
Scan Speed	at least 1 scan per second
Ionization Mode	70 eV positive ion

GC/MS program for DFTPP tune analysis:

DFTPP

GC Temperature program for analyzing DFTPP

Ramp	Rate	Initial Temp	Hold Time	Run Time
1		150°C	1.0 min	
2	13 °C/min	300°C	2.0 min	14.54 min

GC/MS Program for Standards, Blanks, LCS, MS/MSD and sample analysis:

BNA_SIM_W or BNA_SIM_S

6.3 The MS program method scans for specific SIM masses in different time groups.

Group 1 scan should begin after the solvent delay and continue until the elution of 2-methylnapthalene.

Group 2 scan should start right after Group 1 and continue until after fluorene.

Group 3 scan should begin right after Group 2 and continue until after anthracene.

Group 4 scan should begin right after Group 3 and continue until after chrysene.

Group 5 should begin right after Group 5 and continue until after benzo(g,h,i)perylene.

6.4 A primary and secondary ion for each analyte and surrogate will be collected. For some compounds an additional secondary ion will be collected to assist with identification. **Table 2** lists the primary and confirmation ions for target and surrogate analytes.

6.5 Preventative Maintenance - GC/MS are maintained according to the manufacturer's recommendation. The lab analyst performs preventive maintenance as discussed below.

6.5.1 On a daily basis whenever analyses are to be performed, replace the GC septum and clean the injection liner. Also clip up to 6" of the column.

6.5.2 If needed, the analytical column is replaced; this is usually indicated by the tailing of the polar compounds such as pentachlorophenol/benzidine and/or initial and continuing calibration verifications that repeatedly fail to meet method requirements.

6.5.3 If the system constantly drifts out of DFTPP tune and/or the initial and continuing calibration verifications repeatedly fail to meet method requirements, the ion source will need to be cleaned.

6.5.4 There are two filaments in the mass spectrometer. If both filaments are blown, the method sample will be vented to replace both filaments.

NOTE: After major maintenance such as the scenarios described in **sections 6.5.2-6.5.4** an Initial Calibration (ICAL) is analyzed. Document the date of the ICAL in the resolution field in the LIMS Maintenance Logbook.

6.5.5 The rough-pump oil should be replaced at least once a year, or as needed. Check the oil level periodically and add oil if needed. Document this maintenance as above.

6.5.6 Once a year, all GC/MS systems may undergo extensive maintenance by a skilled technician. When this occurs, collect all associated paperwork and enter relevant information in the LIMS maintenance log. The paperwork can be brought to the data reporting area for PDF inclusion on the server.

6.5.7 Corrective maintenance is needed if the lab analyst or his/her supervisor fails to diagnose and/or correct the problem. The analyst or lab supervisor will promptly notify the instrument vendor for telephone-consultation and if needed, schedule on-site repair. This information should be documented as in **Section 6.5.6**. In addition, the resolution field in the LIMS Maintenance Logbook should be filled in fully. Enter your initials in the maintenance log entries; do not use administrator or other non-unique identification. Also refer to SOP 110.0040 Instrument Maintenance and Documentation for additional information.

6.6 Troubleshooting - Refer to troubleshooting section of the HP 5972A MSD hardware manual.

6.7 Glassware:

6.7.1 Hamilton syringes (10 µl, 25 µl, 100 µl, 500 µl, 1000 µl). Syringe accuracy is certified to ±1% by the manufacturer.

6.7.2 Vials – 2ml glass with PTFE-lined screw-cap or crimp-cap tops.

7. Reagents and Standards

7.1 Organic solvents – Pesticide Residue analysis grade (or equivalent) methylene chloride for standard preparation. Solvents are available to the GC/MS lab in 1-gallon liter bottles or in a smaller volume from the Organic Preparation Lab bulk storage distribution line. Always verify that the lot/serial number of the solvent has been approved before use. Check the server for Agawam's solvent check on same lot. See QA for more information.

7.2 The standards used for this SOP are discussed below. ***Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*** These standards are obtained as ampulated mixture. Preparation of surrogate and spike mixes for the preparatory lab are detailed in the preparation SOPs.

7.3 The laboratory will at all time archive or have on order one complete set of unopened ampulated standards (to include internal standards, surrogate standards and target analyte standards).

7.4 All **Primary** standards received from vendors are logged into the LIMS Standard Logbook. The standards are labeled *SPyymmddX*,

Where SP = Semivolatile Primary Standard
yymmdd = date the standard is received
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

7.4.1 Tune Standard: the tuning standard contains DFTPP, 4,4'-DDT, Pentachlorophenol and benzidine. It is purchased from Absolute Standards (Cat. No. 43032) at 1000ug/mL.

7.4.2 Internal Standard: the internal standards are obtained from Cambridge Isotope as neat compounds. A 2000ug/mL solution of all compounds (1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthalene-d10, Phenanthrene-d10, Chrysene-d12, and Perylene-d12) is prepared and

transferred into 1mL aliquoted vials. An ampule is opened and then emptied into a mini-inert vial equipped with an on/off valve.

7.4.3 Primary Calibration Standards: the primary calibration standards are:

- 8270 MEGA Mix (Restek, Cat. No. 31850) at 500-1000ug/mL
- 1,4-Dioxane from Ultra (Cat. No. RCC-180) in neat form.

7.4.4 Second Source Standards: the second source standards are:

- TCL BNA LCS Spike 100ug/mL (NSI Cat No. WL-408-25)
- 1,4-Dioxane at 2000ug/mL (Restek Cat.No.31853)

7.4.5 Surrogate Standards: Benzo(e)pyrene d-12 standard is obtained from Cambridge (Cat.No.DLM-257-S) at 200ug/mL. When analysis includes 1,4-Dioxane, an additional surrogate (1,4-Dioxane-d8) is required. The primary 1,4-Dioxane-d8 standard is also obtained from Cambridge (Cat. No. DLM-28-5) in neat form.

7.5 All **Intermediate** standards are logged into the LIMS Standard Logbook. The standards are labeled *SIyymmddX*,

Where SI = Semivolatile IntermediateStandard
yymmdd = date the standard is prepared
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

7.5.1 An intermediate 8270 standard is prepared by making a 5 times dilution of the 8270 MEGA Mix in **Section 7.4.3** such that most compounds are at 200ug/mL. A 20,000ug/mL standard is prepared from the neat 1,4-dioxane and 1,4-dioxane-d8 standards. 50uL of each of these solutions are added to the diluted 8270MEGA Mix. A secondary intermediate calibration standard at 10ug/mL is prepared by adding the following standards, and diluting to 10mL with methylene chloride See **Attachment 1** for example of LIMS standard Log for 8270 SIM Intermediate standard.

8270 Intermediate (200 ppm)	500uL
Benzo(e)pyrene-d12 (200 ppm)	500uL

7.5.1.1 The Initial Calibration Working Standards are prepared as follows using the standard prepared above:

Levels of Initial Calibration (ug/mL)	Volume (uL) Intermediate Standard	Volume (uL) Methylene Chloride
10	1000	0
5.0	500	500
1 (L3)	100	900

0.5	50	950
0.1	10	990

7.5.1.2 When 1,4-Dioxane is analyzed separately, the initial calibration is prepared using a 20ug/mL intermediate standard containing both 1,4-Dioxane and the surrogate 1,4-Dioxane-d8, using the following scheme:

Levels of Initial Calibration (ug/mL)	Volume (uL) Intermediate Standard	Volume (uL) Methylene Chloride
10	500	500
5	100	900
1(L3)	50	950
0.5	10	990
0.1	5	995

7.5.2 The ICV is prepared in a similar manner as the Midpoint (L3) of the ICAL. Serial dilutions of the second source standards in **Section 7.4.4** are made with methylene chloride until the result is a final 1ug/mL standard.

7.6 All **Working** standards are logged into the LIMS Standard Logbook. The standards are labeled *SWyymmddX*,

Where SW = Semivolatile Working Standard
yymmdd = date the standard is prepared.
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

7.6.1 The working tune standard at 50ug/mL is prepared by adding 50uL of the stock standard to a final volume of 1mL with methylene chloride

7.6.2 The internal standard working standard at 500ug/mL is prepared by adding 250uL of the primary standard (2000ug/mL) and diluting it to 1mL with methylene chloride.

Please note that the volumes can be adjusted to make larger or smaller volume of the standards.

The standards are protected from light and stored in the freezer (F7) at -10°C to -20° C. The standards are stored away from sample extracts to minimize cross contamination.

Unopened ampulated standards' expiration dates are based on manufacturer's expiration dates. If no manufacturer's expiration date is provided the ampulated standards may be retained unopened for up to two years. Once an ampulated standard is opened it may be retained for one year from the date it was opened. Intermediate and Working Standard expiration dates are up to 12 months after they are prepared.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8. Procedure

8.1 The SW-846 methods for sample extraction are as follows:

Method 3510 (SOP# 50.0051) extracts aqueous samples for water-insoluble and slightly water-soluble PAHs. The samples are serially extracted with methylene chloride using a separatory funnel. The final volume for the extracts is 0.5mL to achieve the lower reporting limit.

Method 3520 (SOP# 50.0050) extracts aqueous samples for water-insoluble and slightly water-soluble PAHs. The samples are placed in a continuous liquid-liquid extractor and extracted with methylene chloride for 18 hours.

Method 3540 (SOP# 50.0053) extracts waste, sludge, sediment and soil samples for water-insoluble and slightly water-soluble PAHs. The samples are mixed with anhydrous sodium sulfate, placed in an extraction thimble or between plugs of glass wool, and extracted using 1:1 v/v methylene chloride/acetone in a Soxhlet extractor.

Method 3550 (SOP# 50.0052) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble PAHs. The samples are mixed with anhydrous sodium sulfate to form a free-flowing powder, and then extracted by ultrasonic extraction using 1:1 v/v methylene chloride/acetone.

8.2 Tuning:

The tune standard is prepared at 50µg/mL. The GC/MS must be tuned to meet decafluorotriphenylphosphine (DFTPP) criteria every 12 hours when standards, blanks, LCS and/or samples are to be analyzed.

All of the analysis information is to be recorded in the Semivolatile Instrument Logbook. The logbook is issued by the QA officer and will be archived upon its completion.

8.2.1 Procedure for performing tune - Use the GC/MS conditions in **section 6.2** to perform the tune analysis.

8.2.2 Acceptance criteria for tune - The DFTPP tune will be analyzed using scan for m/z of 35 to 500 amu, based on the full scan. The mass spectrum of DFTPP must be acquired in the following manner for analysis: three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged; if needed background subtraction will be performed and must be accomplished using no more than 20 scans prior to the elution of DFTPP. It is important that the analyst does not selectively add or subtract scans to generate the tune.

For SW846 projects, tune can also be obtained using one of the following procedures (1) use one scan at the peak apex, (2) scan immediately before or after the apex, (3) use the average across the entire peak

A typical mass spectrum and mass spectral listing of the tune in listed in **Figure 1**.

The acceptance criteria are as follows**:**

Mass	Ion Abundance
51	10 - 80% of Base Peak
68	< 2.0% of mass 69
70	< 2.0% of mass 69
127	10 - 80% of Base Peak
197	< 2.0% of mass 198
198	Base peak, or > 50% of mass 442
199	5.0 - 9.0% of mass 198
275	10 - 60% of Base Peak
365	> 1% of mass 198
441	Present, < 24% of mass 442
442	Base Peak or > 50% of mass 198
443	15 - 24% of mass 442

Once the mass spectrometer passes the DFTPP tune, all subsequent standards, samples, and blanks associated with the tune must be analyzed using identical mass spectrometer instrument conditions.

8.3 Initial Calibration - Initial calibration is performed after the instrument passes the tune, % breakdown requirements and column performance check. Initial calibration is required after major instrument maintenance including source cleaning and/or changing column. Initial calibration will also be performed if continuing calibration analyses do not meet QA/QC criteria.

Five calibration standard solutions are required for all target and surrogate compounds. Standard concentrations of 10, 5.0, 1.0, 0.5, and 0.1ng/µL are required for the surrogate and all target compounds.

8.3.1 Calculation for Initial Calibration:

A typical chromatogram of a 2.0ng standard is shown in **Figure 2** attached with the quantitation report.

From the multi-level level calibration, the relative response factor (RRF) for each target compound is determined using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where A_x = area of the selected ion for the target compound to be measured
A_{is} = area of the selected ion for the associated internal standard
C_{is} = concentration of the internal standard
C_x = concentration of the compound to be measured

The mean relative response factor is determined by averaging the 5 level values.

The % relative standard deviation (%RSD) of the RRF is also calculated using:

$$\%\ RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

Where Standard Deviation = $\sqrt{\sum (Xi - X)^2 / (n-1)}$

Where Xi = each individual value used to calculate the mean
X = the mean of n values
n = the total number of values = 5

8.3.2 Initial calibration acceptance criteria for SW-846 is as follows:

- The relative retention time (RRT) for each of the target analyte including the surrogates at each calibration level must be within ± 0.06 RRT of the mean RRT for each compound.
- The area response for each internal standard at each calibration level must be within the inclusive range of –50% to +100% of the mean area response of the internal standard in all of the calibration level.
- The retention time (RT) shift of the internal standards at each calibration level must be within ± 0.5 minutes compared to the mean retention time over the initial calibration range for each internal standard.
- The RSD for all target analytes and surrogate compound must be < 20%. The Target software will flag any compound whose RSD is greater than 20%. If the RSD of any target analytes and/or surrogate compounds is less than 20%, then the RRF is assumed to be constant over the calibration range and the average RRF is used for quantitation. If the calibration is not linear, make sure whether the problem is related to calibration standards or instruments.

  (1) The method allows for a maximum of 10% of the target analytes and/or surrogate compound to fail the 20% RSD criteria. These allowable outliers should NOT be common compounds or compounds of interest to a specific project that will utilize this initial calibration. In addition, these outlier RSDs have a maximum of 50%.
  (2) Linear calibration: a least squares regression may be used. The analyst may employ a regression equation for the analyte(s) that does not pass the earlier approach. The regression will produce the slope and intercept terms for the following linear equation:

$$y = mx + b$$

Where y = instrument response (peak area)
m = slope of the line
x = concentration of the calibration standard
b = intercept

It is important that the origin (0,0) is not included as the sixth calibration point and that the above equation is not forced through the origin.

The linear regression is deemed acceptable if the correlation coefficient **r ≥ 0.995**.

(3) Non linear calibration: The analyst may employ a non linear regression coefficient of determination (COD). The second order quadratic fit will have the following equation:

$$y = ax^2 + bx + c$$

Where y = instrument response (peak area or height)
a and b = slope of the curve
x = concentration of the calibration standard
c = intercept

In performing second order quadratic fit, the analyst should not force the curve to pass through the origin (0,0). In addition, the origin should not be used as an additional calibration point.

From the quadratic fit, the "goodness of fit" is evaluated by calculating the coefficient of determination (COD). In order to be acceptable, the COD of the polynomial must be **≥0.99**.

8.3.3 Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. This is performed by analyzing the 1ug/mL standard prepared in **Section 7.5.2.** The acceptance criteria are as follows:

8.3.3.1 For routine SW8270 SIM analyses, the calculated value of the analyte in the ICV must be 75-125% of the expected value. DoD **full scan SW8270** analyses have an ICV recovery limit of 80-120%. These limits will be applied to PAH compounds by SIM for DoD.

8.3.4 Initial calibration acceptance criteria must be met before any sample, blank or LCS is to be analyzed.

8.3.5 Corrective Action for Initial Calibration - Depending on which compound failed the criteria, corrective action includes preparing fresh standards, source cleaning, and changing GC column or injection liners.

8.4 Continuing Calibration - Continuing calibration standards containing all of the target and surrogate compounds at 1ng on-column injection is performed every time samples are to be analyzed to ensure that the GC/MS system continues to meet instrument sensitivity and linearity requirements.

8.4.1 Frequency of Continuing Calibration- The continuing calibration standard must be performed once every 12 hours. If time remains in the 12-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed. The continuing calibration is also required whenever blanks, LCS, MS/MSD and samples are analyzed.

8.4.2 Continuing calibration acceptance criteria:

- The % D must be ≤ 20% (use % drift if using a regression fit model). A maximum of 20% of the target analytes and/or surrogate compound are allowed to fail the 20% RSD criteria. These allowable outliers should NOT be common compounds or compounds of interest to a

specific project that will utilize this initial calibration. In addition, these outlier %Ds have a maximum of 50%.

- No quantitation ion may saturate the detector.
- The internal standard retention time of the calibration verification standard must be within 30 seconds from that of the mid-point calibration (1ug/mL) of the associated initial calibration when run on the same day. Otherwise the CCV will be used to set the day's RRT to account for potential changes due to GC column maintenance.
- The internal standard area counts must be within +100% to –50% from that of the mid-point calibration (1ug/mL) of the associated initial calibration when run on the same day. Otherwise the CCV will be used for comparison of IS areas.

8.4.3 Corrective Action for Continuing Calibration - Depending on which compound(s) fail(s) the criteria, corrective action included preparing fresh standards, source cleaning, and changing GC column or injection liners. Repeated failure to pass continuing calibration may necessitate performing new initial calibration.

8.4.4 Continuing calibration acceptance criteria must be met before any samples or blanks are to be analyzed for generation of acceptable data.

8.5 Sample Analysis:

Prior to sample analysis, the sample extract and the internal standard are allowed to warm to room temperature to ensure complete dissolution of the high molecular weight internal standards. 10uL of the internal standard solution is added to each of the sample extracts at 1.0mL final volume to ensure 5.0ng on-column amount. Shake the extract slightly to mix. The internal standard volume will be adjusted for smaller extract volume.

8.5.1 Analytical Sequence: The following QC protocol is recommended for analyses:

Initial Batch		Middle/Final Batch	
1.	Tune including Breakdown	1.	Tune
2.	ICal Standard #1	2.	%Breakdown
3.	ICal Standard #2	3.	CCV
4.	ICal Standard #3	4.	Method Blank
5.	ICal Standard #4	5.	LCS
6.	ICal Standard #5	6-13.	Samples (< 8)
7.	ICV(second source)	14.	CCV
8.	Method Blank	15.	Method Blank
9.	LCS	16.	MS
10.	LCSD	17.	MSD
11-18.	Samples (< 8)	18-25.	Samples (< 8)
19.	Tune (as required per 12 hr.)	26.	CCV (Final Batch Only)
20.	CCV (as required per 12 hr.)		
21.	Method Blank		
22.	MS		
23.	MSD		
24-31.	Samples (< 8)		

9. Data Reduction and Calculations

9.1 Identification of Target Compounds - Two criteria are used to identify target compounds:

9.1.1 Relative Retention Time (RRT) - The sample component RRT must agree within ± 0.06 RRT units of the RRT of the component in the associated continuing calibration standard. The relative retention time is determined as follows:

$$RRT = \frac{\text{Retention of target compound}}{\text{Retention time of associated internal standard}}$$

9.1.2 Coelution of the primary and confirmation ions.

9.2 Determining the Concentration of Target Compounds - Sample data should be reported in units of µg/L for aqueous samples and µg/Kg dry weight basis for solid samples.

9.2.1 Target Quantitation: Compounds identified are quantitated using the following equations:

For **aqueous** samples, Concentration *ug/L* $= \dfrac{(A_x)\,(I_s)\,(V_t)\,(Df)}{(A_{is})\,(RRF)\,(V_o)\,(V_i)}$

For **solid** samples, Concentration *ug/Kg* $= \dfrac{(A_x)\,(I_s)\,(V_t)\,(Df)}{(A_{is})\,(RRF)\,(W_s)\,(V_i)\,(S)}$

Where A_x = area of the characteristic ion for the compound to be measured
A_{is} = area of the characteristic ion of the associated internal standard
I_s = amount of internal standard added in nanogram (ng)
RRF = relative response factor
V_o = volume of water extracted in milliliters = 1,000
V_t = volume of the sample extract in milliliters = 1
Df = dilution factor
W_s = weight of soil extracted in grams
S = % solid

9.3 Rounding Rule – Use the most current EPA rounding rules.

9.4 Acceptance Criteria for Sample Analysis:

- The sample must meet both extraction and analysis holding time.
- The sample has to have a compliant tune, initial calibration and continuing calibration.
- The sample has to have a compliant method blank.
- The sample has to have a compliant LCS.
- The surrogate recovery per this SOP (**Section 10.5**) or client-specified criteria.
- All of the target analyte concentration should be within the calibration range.

- The area count of each of the internal standards in the inclusive range of - 50% and +100% of the response of the continuing calibration
- Retention time of each of the internal standards must not shift more than ± 0.5 minute from the continuing calibration.
- excluding the solvent front or the aldol condensation peak for solid extract analysis, no ion should saturate the detector

9.5 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

% Recovery (%R) = 100 x (SSR-SR)/ (SA)

Where: SSR = spiked sample result
SR = sample concentration
SA = spike added

9.6 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

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

Where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

9.7 Manual integrations are performed, reviewed and documented per SOP No. 110.0008 Manual Integration of IC, GC and GC/MS Chromatograms.

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results (IPANDA). To ensure the appropriate analyst is performing the analysis, the analyst's initials should be entered in the Enviroquant acquisition software (do not use the default value). The analyst processing and reviewing data will initial the Run Logbook when processing data.

10.2 Method blanks - A method blank is prepared and analyzed with every batch not to exceed 20 samples. Acceptance criteria for the method blank are as follows:

- The recovery of Benzo(e) pyrene-d12 (and/or 1,4-Dioxane-d8) must be within the acceptance limits discussed in **Section 10.5.**
- Method blank concentration for DoD full scan SW8270 projects must be less than one half the reporting limit. This may or may not be achievable at the low levels of SIM analysis although every attempt is made to provide the cleanest MB.

10.2.1 The "B" qualifier is applied to positive sample results on Form 1 or LIMS Level 2 data sheet when the same compound is detected in the method blank.

10.3 Lab Control Sample (LCS) – A Lab Control Sample is a weight or volume of a clean reference matrix (anhydrous sodium sulfate or DI water) that is spiked with target analytes and surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1 Acceptance criteria for LCS:

- General acceptance: compliant Benzo(e)pyrene-d12 (and/or 1,4-Dioxane-d8) recovery

  Recovery of individual compounds within 45-135% with the exception of pentachlorophenol and 1,4-Dioxane which use a 10-150% limit, or established in-house limits. These 45-135% limits are currently being used per the ACOE guidelines until enough points are collected for both soil and water matrices, at which time in-house derived QC limits may be substituted. Pentachlorophenol and 1,4-Dioxane use a wider limit during the data point collection period due to their low extraction efficiency. Refer to the LIMS Test Information category/Test option/ specs for the most current QC control limits.

10.3.2 If any compounds are outside of the acceptance limits, their recoveries are qualified with the "*" flag on the LCS recovery summary report (Form 3) for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

10.4 Duplicate Matrix Spikes: Matrix spikes and matrix spike duplicate are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received do not have any documented history, we will perform matrix spike and matrix spike duplicate.

10.4.1 Acceptance criteria for Duplicate Matrix Spike:

Matrix spike and matrix spike duplicate are used to assess the effect of matrix interferences on the analysis of the target analytes and the recovery should be used as advisory guidelines to answer question posed above. Control limits are the same as discussed in **Section 10.3.1** but are used as advisory guidelines.

If any compounds are outside of the acceptance limits, their recoveries and/or RPD are qualified with the "*" flag on the recovery MS/MSD summary report (Form 3) for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

10.5 Surrogate recoveries:

10.5.1 The recovery of Benzo (e)pyrene-d12 (and/or 1,4-Dioxane-d8) in all samples, blanks and LCS will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.5.2 Acceptance criteria - The percent recovery of benzo(e) pyrene-d12 in blanks, samples, duplicate matrix spikes and LCS must be within the in-house set QC limits. 1, 4-Dioxane-d8 (when required) has initial limits set at 10-150% recovery. Once enough points are collected, in-house limits will be set per SOP 80.0009 Newly Implemented Methods. These limits may be updated by the QA Department on an annual basis.

10.5.3 Corrective Action for Recovery Failures:

10.5.3.1 Any sample which fails the above will be subjected to re-extraction. Any method blank which fails the above will be re-extracted with the associated samples.

10.5.3.2 If re-extraction and re-analysis of the sample demonstrate similar recovery performance, both sets of results will be reported to demonstrate matrix- related problems. Re-extraction is not required if the recovery is out of the above range for both the native sample and its duplicate matrix spikes.

10.5.3.3 All surrogate outliers will be flagged with an "*" on the surrogate recovery report (Form 2) for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports.

10.6 Internal Standard Response and Retention Times:

10.6.1 The area count of the characteristic ion of each of the internal standards in the samples, blanks, duplicate matrix spikes and LCS must be within the inclusive range of -50.0% and +100% of the response of the internal standards of the continuing calibration.

10.6.2 The retention shift for each of the internal standards in the samples, blanks and LCS must be within ± 0.5 minute of those obtained from the associated Continuing Calibration.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and samples results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the supervisor. The laboratory supervisor reviews 100% of the data prior to report generation. The QA Director randomly reviews 10% of the data reported by the laboratory.

11.2 Reports are generated by the reporting group. The data submitted for report preparation is dependent on project requirements and is subjected to further review by the project manager for reasonableness prior to release to the customer.

12. Data Management and Records Management

12.1 Electronic data generated from the analysis of Semivolatile 8270 SIM extracts (calibrations, QC, samples) is saved and managed per SOP 110.0029 Electronic Data Management.

12.2 All analysis information is documented in the individual Instrument Run/Injection Logbook regardless of run acceptance. No injections are deleted from the sequence.

13. Corrective Action Procedures

Discrepancy reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a discrepancy report for the purpose of identifying the appropriate corrective action is covered in Corrective Action Procedure SOP No. 80.0007. Corrective actions are recorded in the LIMS system in the Quality Control section/corrective action reports. All employees have access to LIMS and may initiate a corrective action. If help is needed, see the QA Director for assistance.

14. Health and Safety

14.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is archived by the health and safety officer and available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Health and Safety Plan. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

14.2 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

15. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 (Waste Management) and 20.0 (Definitions, Acronyms, and Abbreviations) of the current Quality Assurance Plan.

16. References

U.S. Environmental Protection Agency. Gas Chromatography/Mass Spectrometry Method 8270D, SW-846 Test Methods for Evaluating Solid Wastes, Update IV, Revision 4, February 2007.

Attachments:

1. **Table 1**: PAH Target Analyte List for Method 8270 SIM
2. **Table 2:** Selected Ions for Target Compounds and Surrogate.
4. **Figure 1**: DFTPP Tune and Chromatogram.
5. **Figure 2**: Continuing Calibration Standard Chromatogram and Quantitation Report.
6. **Attachment 1**: Example LIMS Standard log for 8270 SIM Intermediate Standard.

Table 1. PAH Target Analyte List for Method 8270 SIM

Target Analyte	CAS Registry No.
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo(a)anthracene	56-55-3
Benzo(b)fluoranthene	205-99-2
Benzo(k)fluoranthene	207-08-9
Benzo(g,h,i)perylene	191-24-2
Benzo(a)pyrene	50-32-8
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Indeno(1,2,3-cd)pyrene	193-39-5
2-Methylnaphthalene	91-57-6
Naphthalene	91-20-3
Phenanthrene	85-01-8
Pyene	129-00-0
1,4-Dioxane	123-91-1

* Note: Additional non-PAH compounds may be analyzed under SIM mode on a client requested basis

Table 2. Selected Ions for PAH Target Compounds and Surrogates*

Target Analyte	Primary Quantitation Ion	Confirmation Ion
Acenaphthene	154	153
Acenaphthylene	152	151
Anthracene	178	89
Benzo(a)anthracene	228	114
Benzo(b)fluoranthene	252	126
Benzo(k)fluoranthene	252	126
Benzo(g,h,i)perylene	276	138
Benzo(a)pyrene	252	126
Chrysene	228	114
Dibenz(a,h)anthracene	278	139
Fluoranthene	202	101
Fluorene	166	165
Indeno(1,2,3-cd)pyrene	276	138
2-Methylnaphthalene	142	141
Naphthalene	128	129
Phenanthrene	178	89
Pyrene	202	101
1, 4-Dioxane	88	58
Surrogate:		
Benzo(e)pyrene-d12	264	132
1,4-Dioxane-d8	96	64

* Note: Additional non-PAH compounds may be analyzed under SIM mode on a client requested basis

Figure 1

Data File: \\Avogadro\Organics\S6.I\110713.B\S6A3290.d

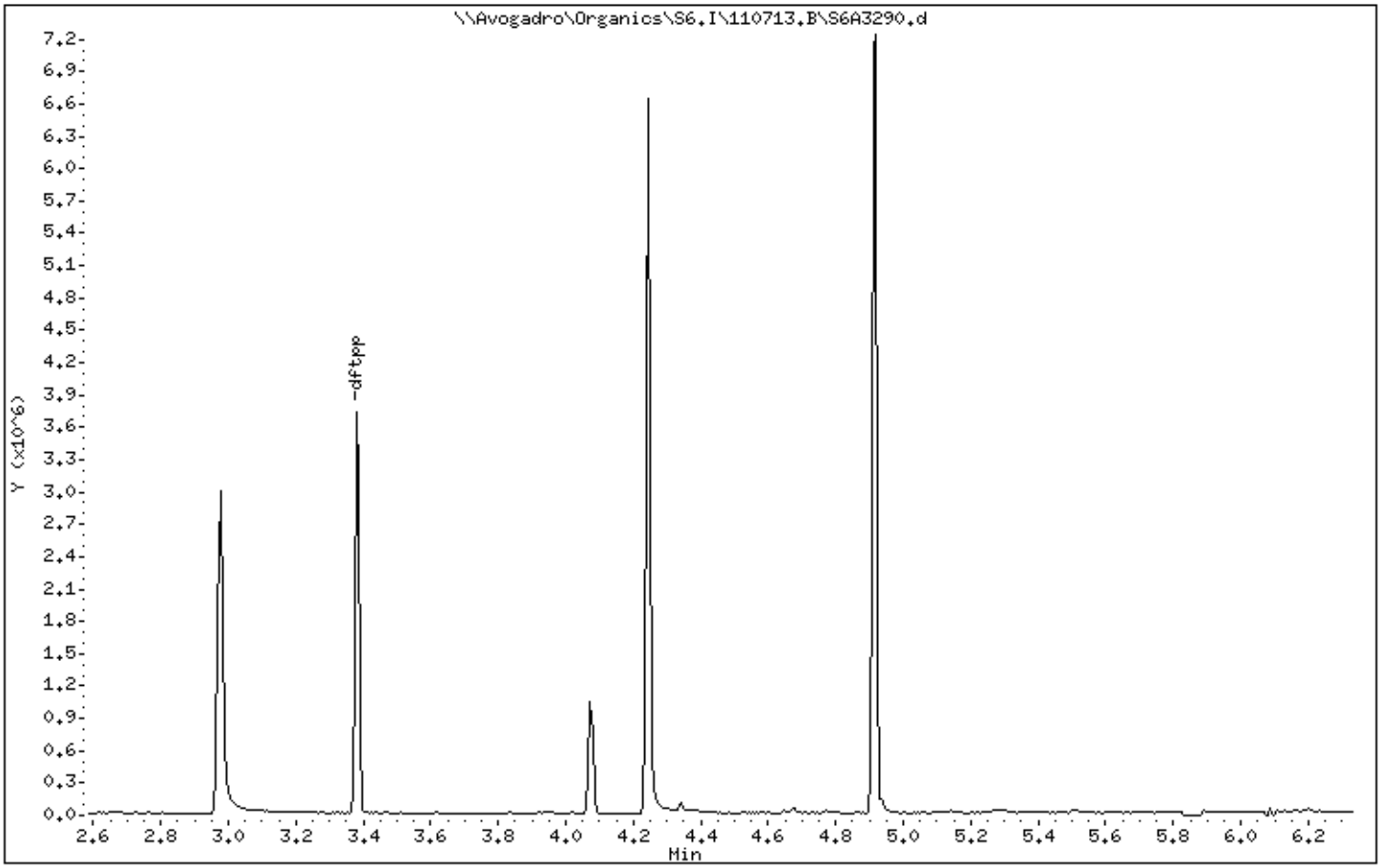
Date : 13-JUL-2011 09:08

Client ID: DFTPP6K | Instrument: S6.i

Sample Info: DFTPP6K,DFTPP6K

Volume Injected (uL): 2.0 | Operator: SRC:

Column phase: RXi-5SILMS | Column diameter: 0.25



Data File: \\Avogadro\Organics\S6.I\110713.B\S6A3290.d

Date : 13-JUL-2011 09:08

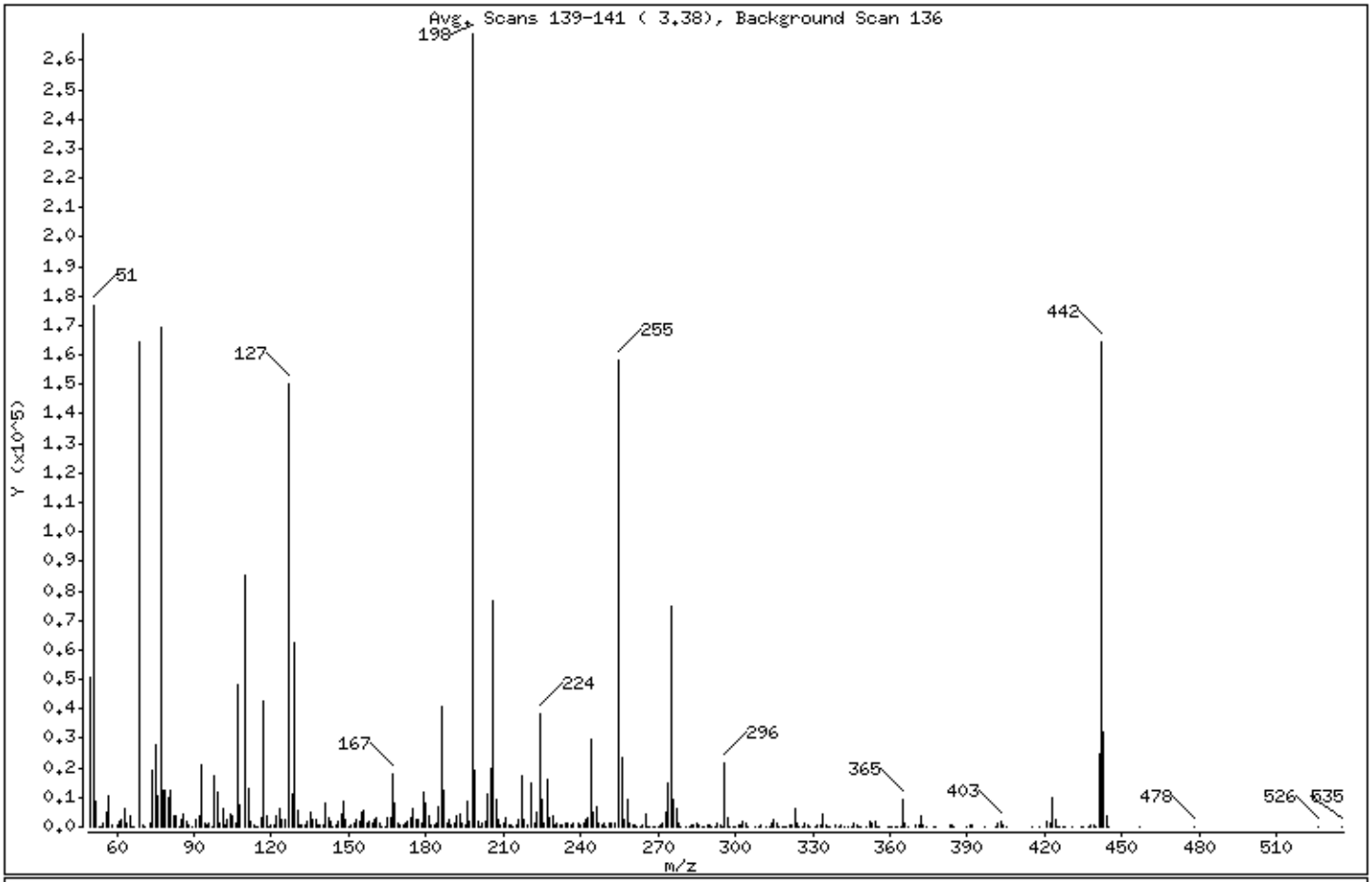
Client ID: DFTPP6K Instrument: S6.i

Sample Info: DFTPP6K,DFTPP6K

Volume Injected (uL): 2.0 Operator: SRC:

Column phase: RXi-5SILMS Column diameter: 0.25

1 dftpp



m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
198	Base Peak, 100% relative abundance	100.00
51	10.00 - 80.00% of mass 198	65.77
68	Less than 2.00% of mass 69	0.00 (0.00)
69	Mass 69 relative abundance	61.06
70	Less than 2.00% of mass 69	0.29 (0.48)
127	10.00 - 80.00% of mass 198	55.89
197	Less than 2.00% of mass 198	0.67
199	5.00 - 9.00% of mass 198	7.23
275	10.00 - 60.00% of mass 198	27.77
365	Greater than 1.00% of mass 198	3.34
441	Present, but less than mass 443	9.16
442	50.00 - 100.00% of mass 198	61.10
443	15.00 - 24.00% of mass 442	12.04 (19.70)

Data File: \\Avogadro\Organics\S6.I\110713.B\S6A3290.d

Date : 13-JUL-2011 09:08

Client ID: DFTPP6K Instrument: S6.i

Sample Info: DFTPP6K,DFTPP6K

Volume Injected (uL): 2.0 Operator: SRC:

Column phase: RXi-5SILMS Column diameter: 0.25

Data File: S6A3290.d

Spectrum: Avg. Scans 139-141 (3.38), Background Scan 136

Location of Maximum: 198.00

Number of points: 344

m/z	Y	m/z	Y	m/z	Y	m/z	Y
50.00	50544	143.00	1951	231.00	1273	324.00	1381
51.00	176832	144.00	307	232.00	365	325.00	113
52.00	8777	145.00	559	233.00	551	326.00	255
53.00	129	146.00	1696	234.00	1322	327.00	1483
54.00	63	147.00	4162	235.00	1263	328.00	791
55.00	1008	148.00	8831	236.00	789	329.00	68
56.00	4756	149.00	2479	237.00	1501	331.00	56
57.00	10214	150.00	156	239.00	978	332.00	469
58.00	753	151.00	755	240.00	492	333.00	398
60.00	327	152.00	1130	241.00	949	334.00	4099
61.00	1777	153.00	2743	242.00	2223	335.00	883
62.00	2580	154.00	2018	243.00	3334	336.00	170
63.00	6446	155.00	4992	244.00	29856	337.00	79
64.00	1037	156.00	5686	245.00	5145	339.00	395
65.00	3574	157.00	1321	246.00	7060	340.00	127
66.00	62	158.00	1975	247.00	1298	341.00	380
67.00	284	159.00	1081	248.00	662	342.00	265
69.00	164160	160.00	2709	249.00	1274	344.00	121
70.00	783	161.00	3335	250.00	306	345.00	62
71.00	229	162.00	1107	251.00	986	346.00	1360
73.00	1527	163.00	256	252.00	1038	347.00	437
74.00	19288	164.00	305	253.00	1166	348.00	156
75.00	28120	165.00	3040	255.00	158464	349.00	164
76.00	10660	166.00	2866	256.00	23712	351.00	113
77.00	169472	167.00	17704	257.00	2484	352.00	2103
78.00	12410	168.00	7849	258.00	8964	353.00	1296
79.00	12128	169.00	1123	259.00	1311	354.00	2046
80.00	10135	170.00	924	260.00	356	355.00	293
81.00	12449	171.00	875	261.00	358	356.00	153
82.00	3846	172.00	1275	262.00	99	359.00	175
83.00	3470	173.00	1970	263.00	77	360.00	252
84.00	16	174.00	3158	264.00	358	361.00	60
85.00	2548	175.00	6400	265.00	4104	362.00	68
86.00	4086	176.00	2566	266.00	275	363.00	109
87.00	1847	177.00	2580	267.00	219	364.00	243

Data File: \\Avogadro\Organics\S6.I\110713.B\S6A3290.d

Date : 13-JUL-2011 09:08

Client ID: DFTPP6K Instrument: S6.i

Sample Info: DFTPP6K,DFTPP6K

Volume Injected (uL): 2.0 Operator: SRC:

Column phase: RXi-5SILMS Column diameter: 0.25

Data File: S6A3290.d
Spectrum: Avg. Scans 139-141 (3.38), Background Scan 136
Location of Maximum: 198.00
Number of points: 344

m/z	Y	m/z	Y	m/z	Y	m/z	Y
88.00	841	178.00	1141	268.00	122	365.00	8976
89.00	187	179.00	11965	269.00	281	366.00	1341
91.00	2506	180.00	7825	270.00	247	367.00	124
92.00	3936	181.00	3669	271.00	805	370.00	421
93.00	21024	182.00	634	272.00	1158	371.00	878
94.00	1131	183.00	650	273.00	5022	372.00	3598
95.00	850	184.00	1036	274.00	14960	373.00	911
96.00	1058	185.00	6493	275.00	74656	374.00	197
97.00	137	186.00	40648	276.00	9365	377.00	153
98.00	17216	187.00	12267	277.00	6176	378.00	53
99.00	11567	188.00	1271	278.00	1353	383.00	694
100.00	1234	189.00	2457	279.00	204	384.00	476
101.00	6432	190.00	776	281.00	181	385.00	164
102.00	786	191.00	1054	282.00	266	390.00	305
103.00	2391	192.00	3788	283.00	733	391.00	310
104.00	4040	193.00	4413	284.00	700	392.00	312
105.00	3863	194.00	1165	285.00	1240	397.00	55
106.00	962	195.00	870	286.00	380	401.00	238
107.00	48136	196.00	8785	287.00	168	402.00	1071
108.00	7661	197.00	1813	288.00	96	403.00	2079
110.00	85576	198.00	268864	289.00	357	404.00	764
111.00	13106	199.00	19440	290.00	513	405.00	137
112.00	1635	200.00	1747	291.00	259	415.00	166
113.00	848	201.00	256	292.00	242	418.00	50
114.00	137	202.00	1536	293.00	1422	421.00	1805
115.00	292	203.00	2062	294.00	633	422.00	1045
116.00	3027	204.00	10967	295.00	110	423.00	10053
117.00	42720	205.00	19992	296.00	21752	424.00	2296
118.00	3609	206.00	76768	297.00	2916	425.00	120
119.00	207	207.00	9189	298.00	70	426.00	50
120.00	431	208.00	2590	299.00	132	427.00	104
121.00	396	209.00	566	300.00	78	428.00	97
122.00	3821	210.00	1498	301.00	482	431.00	59
123.00	5878	211.00	3323	302.00	492	434.00	63
124.00	2382	212.00	416	303.00	1903	435.00	84

Data File: \\Avogadro\Organics\S6.I\110713.B\S6A3290.d

Date : 13-JUL-2011 09:08

Client ID: DFTPP6K Instrument: S6.i

Sample Info: DFTPP6K,DFTPP6K

Volume Injected (uL): 2.0 Operator: SRC:

Column phase: RXi-5SILMS Column diameter: 0.25

Data File: S6A3290.d
Spectrum: Avg. Scans 139-141 (3.38), Background Scan 136
Location of Maximum: 198.00
Number of points: 344

m/z	Y	m/z	Y	m/z	Y	m/z	Y
125.00	2214	213.00	641	304.00	981	437.00	241
127.00	150272	214.00	218	305.00	102	438.00	498
128.00	11240	215.00	710	308.00	182	439.00	760
129.00	62144	216.00	2426	309.00	147	440.00	273
130.00	5435	217.00	17488	310.00	352	441.00	24640
131.00	774	218.00	2245	312.00	132	442.00	164288
132.00	397	219.00	563	313.00	281	443.00	32360
133.00	340	221.00	14642	314.00	1440	444.00	3627
134.00	2134	222.00	1245	315.00	2513	445.00	284
135.00	4753	223.00	4901	316.00	1045	457.00	56
136.00	2455	224.00	38288	317.00	256	478.00	113
137.00	2771	225.00	9463	318.00	147	526.00	56
138.00	314	226.00	1185	319.00	233	535.00	50
139.00	689	227.00	16162	320.00	144		
140.00	424	228.00	2861	321.00	686		
141.00	8198	229.00	3926	322.00	642		
142.00	3011	230.00	829	323.00	6282		

Figure 2

Data File: \\avogadro\organics\S6.I\110607.B\S6A2951B.d
Report Date: 08-Jun-2011 12:43

Spectrum Analytical, Inc. RI Division

SIM-PAH

Data file : \\avogadro\organics\S6.I\110607.B\S6A2951B.d
Lab Smp Id: SSTD0016F Client Smp ID: SSTD0016F
Inj Date : 07-JUN-2011 12:22
Operator : SRC: Inst ID: S6.i
Smp Info : SSTD0016F,SSTD0016F
Misc Info : 2,3
Comment   :
Method   : \\avogadro\organics\S6.I\110607.B\S6_pah_sim.m
Meth Date : 08-Jun-2011 12:43 S6.i Quant Type: ISTD
Cal Date : 11-MAY-2011 12:31 Cal File: S6A2693.d
Als bottle: 2 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: HP RTE Compound Sublist: PAH.sub
Target Version: 4.14
Processing Host: TARGET113

Concentration Formula: Amt * DF * Uf*(Vt/Vi)*(1/Vo) * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	GPC Factor
Vt	1000.000	Extract Volume (uL)
Vi	1.000	Injection Volume
Vo	1000.000	Sample Volume
Cpnd Variable		Local Compound Variable

	Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)
*	3 1,4-Dichlorobenzene-d4	152	2.869	2.869	(1.000)	74262	5.00000	
*	9 Naphthalene-d8	136	3.852	3.852	(1.000)	256303	5.00000	
	10 Naphthalene	128	3.864	3.864	(1.003)	55625	1.00000	0.84(Q)
	12 2-Methylnaphthalene	142	4.432	4.432	(1.151)	40232	1.00000	1.0
	15 Acenaphthylene	152	5.157	5.157	(0.977)	57463	1.00000	1.0
*	16 Acenaphthene-d10	164	5.278	5.278	(1.000)	130700	5.00000	
	17 Acenaphthene	154	5.295	5.295	(1.003)	35455	1.00000	0.99
	20 Fluorene	166	5.727	5.727	(1.085)	43988	1.00000	1.1
*	25 Phenanthrene-d10	188	6.495	6.495	(1.000)	423472	5.00000	
	26 Phenanthrene	178	6.514	6.514	(1.003)	117978	1.00000	0.92
	28 Anthracene	178	6.553	6.553	(1.009)	82119	1.00000	0.83
	29 Fluoranthene	202	7.499	7.499	(1.155)	133450	1.00000	1.2
	30 Pyrene	202	7.680	7.680	(1.183)	142453	1.00000	1.1
	32 Benzo(a)anthracene	228	8.718	8.718	(0.999)	127915	1.00000	1.0(Q)
*	33 Chrysene-d12	240	8.726	8.726	(1.000)	515735	5.00000	
	34 Chrysene	228	8.752	8.752	(1.003)	134634	1.00000	0.97
	35 Benzo(b)fluoranthene	252	9.651	9.651	(0.967)	123893	1.00000	1.2(T)
	36 Benzo(k)fluoranthene	252	9.677	9.677	(0.970)	147564	1.00000	1.2(T)
$	37 Benzo(e)pyrene-d12	264	9.859	9.859	(0.988)	149005	1.00000	1.1
	39 Benzo(a)pyrene	252	9.928	9.928	(0.995)	86856	1.00000	1.1

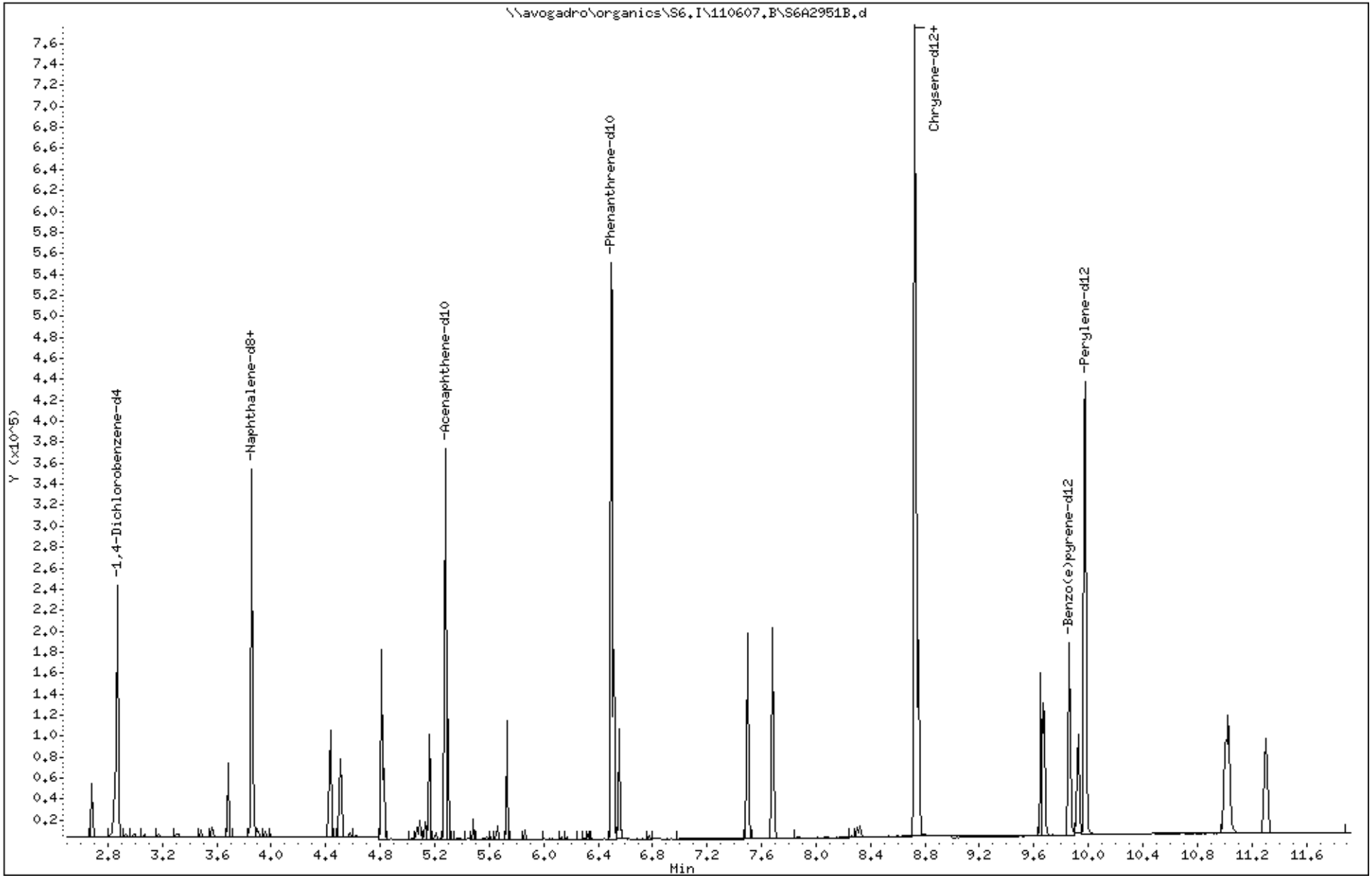
Data File: \\avogadro\organics\S6.I\110607.B\S6A2951B.d
Report Date: 08-Jun-2011 12:43

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)
* 40 Perylene-d12	264	9.980	9.980	(1.000)	401077	5.00000	
41 Indeno(1,2,3-cd)pyrene	276	11.008	11.008	(1.103)	127492	1.00000	1.3
42 Dibenzo(a,h)anthracene	278	11.034	11.034	(1.106)	104499	1.00000	1.2
43 Benzo(g,h,i)perylene	276	11.302	11.302	(1.133)	110224	1.00000	1.2

QC Flag Legend

T - Target compound detected outside RT window.
Q - Qualifier signal failed the ratio test.

Data File: \\avogadro\organics\S6.I\110607.B\S6A2951B.d
Date : 07-JUN-2011 12:22
Client ID: SSTD0016F
Instrument: S6.i
Sample Info: SSTD0016F,SSTD0016F
Volume Injected (uL): 1.0
Operator: SRC:
Column phase: DB-5MS
Column diameter: 0.25



Data File: \\Avogadro\Organics\S6.I\110819.B\S6A3873C.d
Report Date: 29-Aug-2011 14:53

Spectrum Analytical, Inc. RI Division

SIM-PAH

Data file : \\Avogadro\Organics\S6.I\110819.B\S6A3873C.d
Lab Smp Id: SSTD0016A Client Smp ID: SSTD0016A
Inj Date : 19-AUG-2011 11:51
Operator : SRC: Inst ID: S6.i
Smp Info : SSTD0016A,SSTD0016A
Misc Info : 2,3
Comment   :
Method   : \\Avogadro\Organics\S6.I\110819.B\S6_pah_sim.m
Meth Date : 29-Aug-2011 14:53 S6.i Quant Type: ISTD
Cal Date : 19-AUG-2011 12:59 Cal File: S6A3877.d
Als bottle: 2 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: HP RTE Compound Sublist: Dioxane.sub
Target Version: 4.14
Processing Host: TARGET104

Concentration Formula: Amt * DF * Uf*(Vt/Vi)*(1/Vo) * CpndVariable

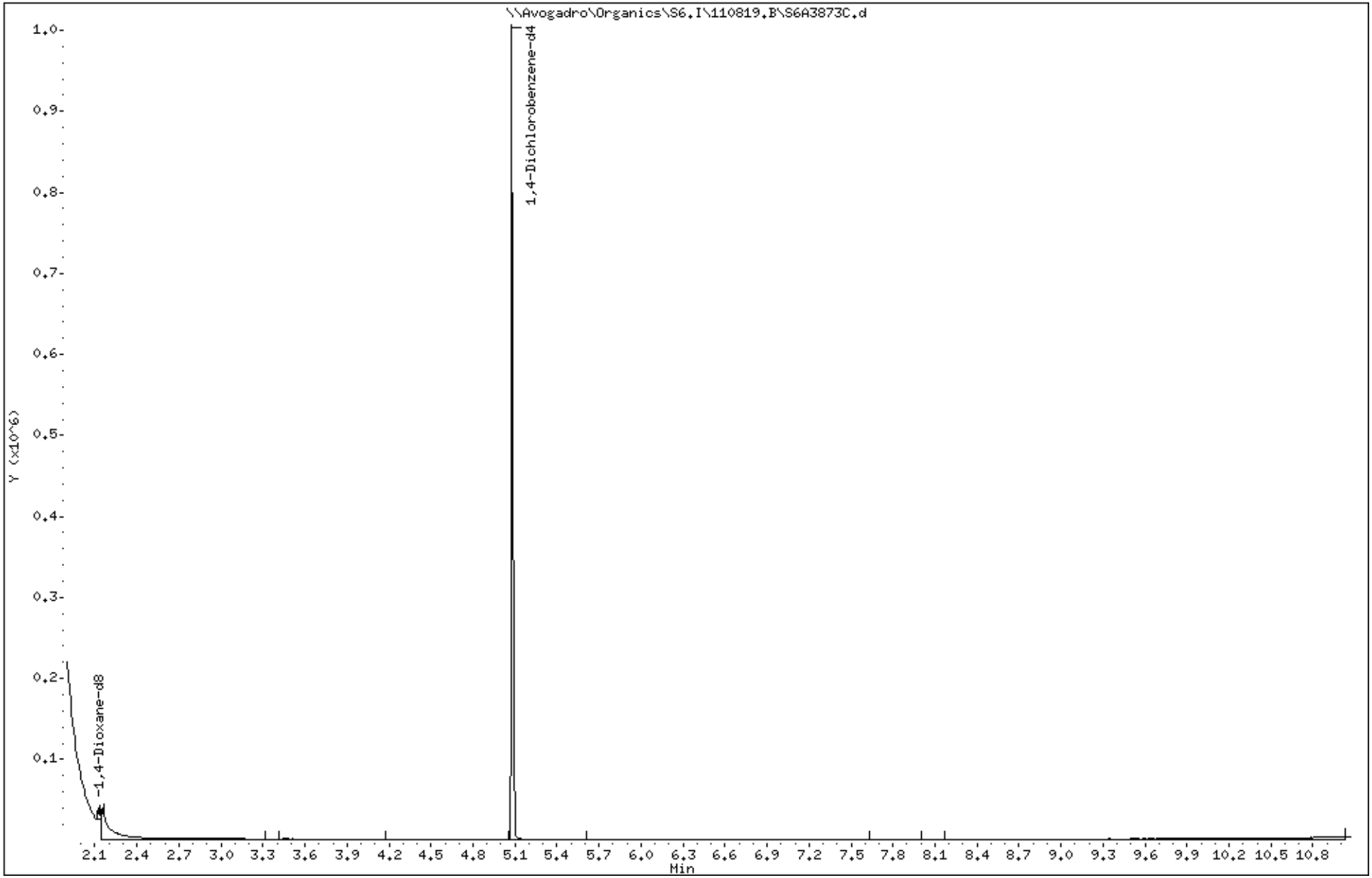
Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	GPC Factor
Vt	1000.000	Extract Volume (uL)
Vi	1.000	Injection Volume
Vo	1000.000	Sample Volume
Cpnd Variable		Local Compound Variable

		QUANT SIG					AMOUNTS CAL-AMT	AMOUNTS ON-COL
	Compounds	MASS	RT	EXP RT	REL RT	RESPONSE	(ng)	(ng)
$	145 1,4-Dioxane-d8	96	2.129	2.129	(0.419)	19954	1.00000	1.0(A)
	128 1,4-Dioxane	58	2.159	2.159	(0.425)	16370	1.00000	1.0(Q)
*	3 1,4-Dichlorobenzene-d4	152	5.076	5.076	(1.000)	267782	5.00000	

QC Flag Legend

A - Target compound detected but, quantitated amount
    exceeded maximum amount.
Q - Qualifier signal failed the ratio test.

Data File: \\Avogadro\Organics\S6.I\110819.B\S6A3873C.d
Date : 19-AUG-2011 11:51
Client ID: SSTD0016A
Instrument: S6.i
Sample Info: SSTD0016A,SSTD0016A
Volume Injected (uL): 1.0
Operator: SRC:
Column phase: DB-5MS
Column diameter: 0.25



Attachment 1: Example LIMS Standard log for 8270 SIM Intermediate Standard

Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division
Standard LOG

Standard ID: SI110607B

Standard Name: BNA SIM INTERMEDIATE | Type: Intermediate

Date Prepared: 6/7/2011 | Prepared By: 240

Date Expires: 3/12/2012

Date Validated: 8/18/2011 | Validated By: Catherine L Mosher

Department: MSSEMI | Status: New

Vendor: | Lot Number:

Comments:

Final Volume: 10 mL

Stock Source		Base Units	Amount Added
SI110513B	8270 INTERMEDIATE	µg/mL	0.5 mL
SP110505C	BENZO[E]PYRENE-D12	µg/mL	0.5 mL

	Analytes	CAS	Conc: µg/mL
	2-butoxyethanol		10
	2-ethyl-1-hexanol	104-76-7	10
	a-terpinol	10482-56-1	10
A	1,1´-Biphenyl	92-52-4	10
A	1,2,4,5-Tetrachlorobenzene	95-94-3	10
A	1,2,4-Trichlorobenzene	120-82-1	10
A	1,2-Dichlorobenzene	95-50-1	10
A	1,2-Dinitrobenzene	528-29-0	10
A	1,3-Dichlorobenzene	541-73-1	10
A	1,3-Dinitrobenzene	99-65-0	10
A	1,4-Dichlorobenzene	106-46-7	10
A	1,4-Dinitrobenzene	100-25-4	10
A	1,4-Dioxane	123-91-1	10
A	1-Methylnaphthalene	90-12-0	10
A	2,3,4,6-Tetrachlorophenol	58-90-2	10
A	2,3,5,6-Tetrachlorophenol	935-95-5	10
A	2,4,5-Trichlorophenol	95-95-4	10
A	2,4,6-Trichlorophenol	88-06-2	10
A	2,4-Dichlorophenol	120-83-2	10
A	2,4-Dinitrophenol	51-28-5	10
A	2,4-Dinitrotoluene	121-14-2	10
A	2,6-Dinitrotoluene	606-20-2	10
A	2.4-Dimethylphenol	105-67-9	10
A	2-Chloronaphthalene	91-58-7	10
A	2-Chlorophenol	95-57-8	10
A	2-Methylnaphthalene	91-57-6	10
A	2-Methylphenol (o-cresol)	95-48-7	10
A	2-Nitroaniline	88-74-4	10
A	2-Nitrophenol	88-75-5	10
A	3,3´-Dichlorobenzidine	91-94-1	10
A	3-Methylphenol (m-cresol)	108-39-4	5
A	3-Nitroaniline	99-09-2	10

Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division
Standard LOG

Standard ID: SI110607B

Standard Name: BNA SIM INTERMEDIATE | Type: Intermediate

Date Prepared: 6/7/2011 | Prepared By: 240

Date Expires: 3/12/2012

Date Validated: 8/18/2011 | Validated By: Catherine L Mosher

Department: MSSEMI | Status: New

Vendor: | Lot Number:

Comments:

A	4- Nitrophenol	100-02-7	10
A	4,6-Dinitro-2-methylphenol	534-52-1	10
A	4-Bromophenyl phenyl ether	101-55-3	10
A	4-Chloro-3-methylphenol	59-50-0	10
A	4-Chloroaniline	106-47-8	10
A	4-Chlorophenyl phenyl ether	7005-72-3	10
A	4-Methylphenol (p-cresol)	106-44-5	5
A	4-Nitroaniline	100-01-6	10
A	Acenaphthene	83-32-9	10
A	Acenaphthylene	208-96-8	10
A	Acetophenone	98-86-2	10
A	Aniline	62-53-3	10
A	Anthracene	120-12-7	10
A	Atrazine	1912-24-9	10
A	Azobenzene	103-33-3	10
A	Benzidine	92-87-5	10
A	Benzo(a)anthracene	56-55-3	10
A	Benzo(a)pyrene	50-32-8	10
A	Benzo(b)flouranthene	205-99-2	10
A	Benzo(g,h,i)perylene	191-24-2	10
A	Benzo(k)flouranthene	207-08-9	10
A	Benzoic acid	65-85-0	10
A	Benzyl alcohol	100-51-6	10
A	Benzyl butyl phthalate	85-68-7	10
A	Bis(2-chloroethoxy)methane	111-91-1	10
A	Bis(2-chloroethyl)ether	111-44-4	10
A	Bis(2-chloroisopropyl)ether	108-60-1	10
A	Bis(2-ethylhexyl)adipate	103-23-1	10
A	Bis(2-ethylhexyl)phthalate	117-81-7	10
A	Caprolactam	105-60-2	10
A	Carbazole	86-74-8	10
A	Chrysene	218-01-9	10
A	Dibenzo(a,h)anthracene	53-70-3	10
A	Dibenzofuran	132-64-9	10
A	Diethylphthalate	84-66-2	10
A	Dimethylphthalate	131-11-3	10
A	Di-n-butylphthalate	84-74-2	10
A	Di-n-octyl phthalate	117-84-0	10

Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division
Standard LOG

Standard ID:	SI110607B		
Standard Name:	BNA SIM INTERMEDIATE	Type:	Intermediate
Date Prepared:	6/7/2011	Prepared By:	240
Date Expires:	3/12/2012		
Date Validated:	8/18/2011	Validated By:	Catherine L Mosher
Department:	MSSEMI	Status:	New
Vendor:		Lot Number:	
Comments:			

A	Diphenylamine	122-39-4	10
A	Flouranthene	206-44-0	10
A	Flourene	86-73-7	10
A	Hexachlorobenzene	118-74-1	10
A	Hexachlorobutadiene	87-67-3	10
A	Hexachlorocyclopentadiene	77-47-4	10
A	Hexachloroethane	67-72-1	10
A	Indeno(1,2,3-cd)pyrene	193-39-5	10
A	Isophorone	78-59-1	10
A	Naphthalene	91-20-3	10
A	Nitrobenzene	98-95-3	10
A	N-Nitrosodimethylamine	62-75-9	10
A	N-Nitroso-di-n-propylamine	621-64-7	10
A	Pentachloronitrobenzene	82-68-8	10
A	Pentachlorophenol	87-86-5	10
A	Phenanthrene	85-01-8	10
A	Phenol	108-95-2	10
A	Pyrene	129-00-0	10
A	Pyridine	110-86-1	10
S	1,4-Dioxane-d8	17647-74-4	10
S	2,4,6-Tribromophenol	118-79-6	10
S	2-Fluorobiphenyl	321-60-8	10
S	2-Fluorophenol	367-12-4	10
s	Benzo(e)pyrene-d12	205440-82-0	10
S	Nitrobenzene-d5	4165-60-0	10
S	Phenol-d6	13127-88-3	10
S	Terphenyl-d14	1718-51-0	10

Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8260C

Contents SOP NO. 90.0012

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		9/7/12
Quality Assurance Director		9/7/12
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date

Revision Record

Revision Date	Revision Description	Comments	Initials
4/15/03	Added control page and renamed SOP	Was SOP O12A1	
5/7/03	Added section 8.6.2.6- adding surrogates to medium-level analysis	Per TRC audit	
9/29/06	Added 8260 low level details	Per NELAC	
11/2006	Updated to 8260C		
3/12/08	Added compliance criteria for storage blank, lab name change		SBL
4/28/08	Expanded state program disclaimer to include DoD.	Per Navy data audit 2008. Unable to use 8260C calibration criteria	SBL
10/6/08	Updated references to include SW-846 5035, storage blanks logged in.	Refered to LIMS for limits/MDL…	SBL
11/24/09	QSM4.1 added	Attachments revised	SBL
12/23/09	ICV 80-120 per QSM4.1		SBL
02/17/10	New revision with DoD required surrogates at multi levels in ICAL	Removed QSM3, added LIMS std option	SBL
11/3/10	Add new GC/MS V10, fix med soil calculation	On-line 11/2010	SBL
9/2011	Added more info about LCS outliers, SS, Zacq, section 12 edit, tuning V10 criteria to use CLP	Full Revision, Edited lab name.	SBL
3/8/12	Added correct trap info for V10. Instrument set up moved to sec 8. Calibrated vial for dilutions included.	Minor edit	SBL
5/25/12	Updated 5035 to 5035A, added new Dilution logbook reference.	Minor edit	SBL
9/7/12	Edit to calc for med soils (unit), SS rec criteria changed	Full Rev	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

Spectrum Analytical, Inc.

Featuring Hanibal Technology
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8260C

SOP No. 90.0012

Rev. 13

	Signature	Date
QA Director:		9/7/12
Lab Director:		9/7/12
Effective Date:	9/14/12	

Spectrum Analytical, Inc.
Featuring Hanibal Technology
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8260C

Rev. 13

1. Scope and Application

This SOP describes the analysis of volatile organic compounds in aqueous and soil samples using gas chromatography/mass spectrometry (GC/MS). The SOP covers the analyses according to protocols discussed in SW846 Update 8/2006 of Method 8260C. A modified version of Method 8260C for a low level aqueous analysis using a 25 mL aliquot is included as **Section 17**. This SOP meets all of the requirements specified in the method. To further familiarize oneself with the procedures, the analyst is encouraged to consult the following instrument manual:

- Hewlett Packard EnviroQuant GC/MS Manual

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol or unusual occurrences noted during analyses. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve reported results. The **Laboratory Director** and/or senior management evaluate laboratory reports for reasonableness of the results and sign the reports. The **QA Director** reviews the QC system and quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure/Instrumentation

3.1 The volatile compounds are introduced into the GC/MS by purge-and-trap system. Analytes are extracted from the sample by bubbling with helium. The analytes are trapped from the helium stream on an adsorbent trap. The analytes are desorbed at high temperature directly onto a narrow-bore capillary column after been split at 1:50 ratio via an EPC controlled injector for analysis. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer.

3.2 Analytes eluted from the capillary column are introduced into the mass spectrometer by direct interface to the ion source. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (primary) ion relative to an internal standard using a five-point calibration curve.

3.3 A list of abbreviations used in this SOP is included in **Table 1**.

3.4 The list of compounds to be analyzed and reported may vary from project to project. The SW-846 method contains several different lists of analytes, so there is no "official" EPA list of Method 8260 compounds. The lab typically analyzes samples for and reports a fairly extensive list of analytes. Certain projects also may have additional extra compounds not on the routine analytical target list. Alternatively certain projects may have a shorter list of target compounds. These project-specific lists of analytes are specified by the client through discussion with the Project Manager who discusses the list with the Laboratory Supervisor. The lists are managed in the lab by the use of "sublists" in the Target data reduction and reporting software. In addition, when utilizing the LIMS system, the sublist can be viewed using the SEL list option. SEL refers to the select list of target analytes requested by the client. It is used when this list differs from the "routine" analyte list. A list of the routine 8260 analytes used by the laboratory is shown in **Attachment 1**. Refer to the LIMS Test Information category/Test option/ limits of the test code, for the most current MDL values. Those listed in **Attachment 1** may not be the most up to date.

3.4.1 Several options exist for reporting extra compounds not on the normal Target list. The ideal approach includes purchasing a calibration standard solution and a second source calibration check solution, determination of method detection limit from 7 or more replicate analyses and addition of the compound to initial and continuing calibrations and laboratory control sample analyses. (See SOP No. 80.0005 for details on determination of the MDL). Depending on the needs of the client, alternative approaches may be appropriate, including single point calibration or searching for the compound as a Tentatively Identified Compound using the Target software's library search routines. The approach taken must be discussed with the client prior to analyses, and if needed, sufficient documentation is included in the analysis report to enable validating the data. The analyst will be instructed by the lab supervisor as to what documentation is needed and what is required to be sent to the data reporting area for inclusion in the final report.

3.4.2 The Quality Control requirements contained in this SOP apply to the specific list of analytes being reported. SOP criteria are to be evaluated for all project target analytes. While QC

issues with non-project target analytes should be investigated, they are not critical if that compound is not being reported. Calibration standards and LCS/MS solutions may contain compounds not being reported for a particular project.

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For volatile organic compound analysis by method 8260, water samples are collected in 40-milliliter (mL) glass vials, typically preserved with HCl. Solid samples may be collected in glass containers, EnCore™ samplers, pre-weighed 40-mL vials preserved by 5 mL of DI water to be frozen upon receipt or similar pre-weighed vials with sodium bisulfate solution for low-level analysis or preserved by 5 mL methanol for medium-level analysis. According to Method 5035, the low-level soil samples shipped in EnCore samplers need to be extruded into a pre-weighed DI water/stir bar vial or similar vial with sodium bisulfate solution as soon as received in the lab (within 48hours of collection). If needed, the soil samples received in EnCore samplers may be preserved by storage in a dedicated freezer until prior to the analysis. This freezer only contains samples and must not contain any analytical standards. ASTM method D6418-04 includes documentation of the ability of EnCore devices to contain volatile compounds without significant loss when frozen for up to 14 days. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples. Typical sample submittals are listed below:

- "normal aqueous samples: 2 X 40ml vials, HCl Preserved
- "low-level" aqueous samples: 3 X 40ml vials, HCl Preserved
- preserved soil samples: 2 X DI water/freeze preserved or
  sodium bisulfate preserved plus
  1 X methanol preserved plus
  4 ounce jar for percent solids determination.

Other sample submittals may be suitable for analysis depending on the needs of the specific project. The Project Manager, Supervisor or client should be contacted to determine the suitability of sample containers to meet SOP and project objectives.

4.2 All aqueous samples and soil samples received in glass jars are stored in the VOA lab at 4°C ± 2°C until analyzed. The soil samples received in 40mL vials preserved either by sodium bisulfate or methanol are also stored at 4°C ± 2°C in the VOA lab. Soil samples received in pre-weighed 5mL DI water vials are stored in a freezer at down to -20°C. For soil samples received in the EnCore type of device (typically in silver pouches), these are extruded into pre-weighed vials containing a stir bar and 5mL of DI water, then re-weigh the vials to obtain the final sample weights. The vials are to be placed into a freezer.

4.3 Storage areas used for samples for volatile organic analysis must be free of potential contaminants. To document storage conditions a storage blank consisting of a 40ml vial of organic-free water is

placed in every refrigerator used to store SW8260 VOC samples on a weekly basis. Storage Blanks (refrigerator and freezer where applicable) are logged in to the LIMS system and tracked using the reporting feature. These will be analyzed on a weekly basis. When a storage blank is removed for analysis, another blank will be placed in the refrigerator such that there will be a blank in each refrigerator on a 24/7 basis. Highly contaminated samples are stored in a specially marked refrigerator in the VOA lab.

4.4 Sample holding time for volatile organic compound analysis by method SW8260 are 14 days from the day of sample collection for both preserved aqueous and soil samples. The holding time for non-preserved aqueous samples is 7 days from the day of sample collection. Samples will be disposed after a minimum of 30 days after the submission of a complete data package.

5. Interference and Potential Problems

5.1 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 PTFE thread sealant, 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 value result in what the laboratory feels is a false positive result for a sample due to laboratory background contamination; the laboratory should fully explain this in text accompanying the uncorrected data. Compounds detected in method blanks and also detected in samples from the same batch are qualified with a “B” flag on data report forms, and listed on the data review checklist. The definition of the “B” qualifier is included in the data report.

5.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the purging apparatus and sample syringes must be rinsed with at least two portions of organic-free reagent water between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of one or more blanks to check for cross-contamination.

5.3 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 in the sample flow path including purging vessel, tubing, or sample valves may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is helpful to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished by analysis of an extra aliquot at a dilution beyond the 12 hour instrument tune time or comparison to any available previous results for the sample.

5.4 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PFTE 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. ***It is important for analysts to keep this in mind if they enter the organic preparation laboratory and plan to return to the volatiles lab. Their clothing may be a source of contamination in the volatiles lab***.

5.5 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 or other matrix and carried through the sampling, handling, and storage protocols serve as a check on such contamination. Trip blanks are typically sent with each shipment of VOA vials and the client is advised to have them analyzed. The client is also advised to prepare field blanks to be sent to the lab if appropriate for their sampling plan.

5.6 Use of sensitive mass spectrometers or larger sample sizes to achieve lower detection levels will increase the potential to detect laboratory contaminants as interference.

5.7 Direct injection (for BFB) – 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 case of extreme contamination.

6. Equipment and Apparatus

6.1 Equipment:

There are five GC/MS instruments in the Volatile Organic Analysis Lab. There are V1, V2, V5, V6 and V10. The GC/MS systems have similar configurations as follows: Hewlett Packard (HP) 5890 GC interfaced to a HP Model 5972A mass spectrometer connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V1); Hewlett Packard (HP) 5890 GC interfaced to a HP Model 5972A mass spectrometer connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V2); Hewlett Packard (HP) 6890 GC interfaced to a HP Model 5972A mass spectrometer connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V5); Hewlett Packard (HP) 6890 GC interfaced to a HP Model 5973 mass spectrometer connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V6), and an Agilent 7890A GC interfaced to an Agilent 5975C mass spectrometer connected to an Tekmar 3100 Purge and Trap Concentrator with a Tekmar SOLATek72 autosampler (V10); The laboratory maintains flexibility to interface various purge and traps to various autosamplers and subsequently to various GC or GC/MS systems. This flexibility is an important tool in trouble-shooting system problems or to minimize the impact of instrument down-time. EnviroQuant Software is used to handle data acquisition. Data files are

copied and transferred to the company file server via the computer network. Actual data quantitation and analyses are performed by the analyst using Target chromatographic software (Thru-Put systems, Inc.).

6.1.1 A 30m x 0.25mm id DB-624 capillary column is used for all GCs. The GC is directly interfaced to the MS. The GC injector operates under split mode (about 50:1) at all times.

6.1.2 OI 4552 autosamplers are fitted with heat function for low level soil analysis (V2, V5 and V1).

6.1.3 Supelco Trap K, VOCARB 3000 (containing 10 cm Carbopack B, 6 cm Carboxen 1000 and 1 cm Carboxen 1001) and OI Analytical Trap #10 (containing 8cm each of Tanax and silica gel and carbon molecular sieve).

6.1.4 High purity helium (99.999%) is used both as GC carrier gas and Purge and Trap purge gas.

6.1.5 The instruments scan from amu 35 to 300 at EM voltage similar to those of the tunes.

6.1.6 The BFB data acquisition method is V1TUNE, V2TUNE, V5TUNE, V6TUNE and V10TUNE for each of the five instruments.

6.1.7 The data acquisition methods for unheated purge (for aqueous and medium level soil samples) are V1VOA, V2VOA, V5VOA, V6VOA and V10VOA. The data acquisition methods for heated purge (for low-level soil samples) are named similarly.

6.1.8 Balance: A top-loading balance capable of weighing 200.0 ± 0.1 g.

6.2 Preventative Maintenance - The Purge and Trap GC/MS are maintained according to the manufacturers' recommendations. The lab analyst performs preventive maintenance as discussed below:

6.2.1 The Purge and Trap spargers are rinsed and cleaned automatically by the autosampler between each analysis. This cleaning procedure is sufficient for most sample analyses. Some samples exhibited target compounds or TIC at unusual high concentrations result in contaminating the Purge and Trap system that may require additional cleaning and baking out. Under those circumstances, any part on the flow path directly contacting with sample including transfer line, valves and sparger may need to be back-flushed with VOC-free water, then with methanol, and extra baking. Analyst performs this extensive cleaning procedure should record them into the LIMS maintenance logbook (see **Section 6.2.9**). The system must be demonstrated to be contaminant free by analyzing instrument blank before reuse for sample analysis. The trap will be replaced if tailing peak response and loss of gaseous compounds that can not be related to standard solution problems are observed.

6.2.2 The GC septum will be replaced monthly (this is done rarely as the septum is only penetrated whenever BFB tuning compound is analyzed.

6.2.3 The maintenance of GC injection liner will be performed as needed. If necessary, the liner will be replaced.

6.2.4 If needed, the analytical column will be replaced; this is usually indicated by excessive peak tailing and/or repeated failures of initial or continuing calibration verifications to meet SOP criteria.

6.2.5 The ion source will be cleaned when the system drifts out of BFB tune and/or repeated failure of initial and/or continuing calibrations to meet SOP-specified criteria.

6.2.6 If there is a second blown filament, the ion source will be vented to install two new filaments. Whenever the ion source is opened for maintenance, the analyst should make sure two good filaments are in place, or replace any filament blown since the last maintenance. This will minimize the times when both filaments are blown.

6.2.7 The pump oil will be replaced as needed.

6.2.8 Corrective maintenance is needed if the lab analyst or his/her supervisor fails to diagnose and/or correct the problem. The analyst or lab supervisor will promptly notify the manufacturer of the problem to schedule on-site diagnosis and repair.

6.2.9 All non-routine preventive and corrective maintenance shall be documented in the Instrument Maintenance log located in the LIMS system. This can be accessed using the category Analytical and option Instruments in the LIMS menus. All analysts have access to this function in LIMS. If help is needed, ask the Lab Supervisor for assistance.

6.3 Troubleshooting - Refer to troubleshooting section of the HP 5972A MSD and 5973 MSD hardware manuals.

6.4 Glassware:

6.4.1 Class “A” volumetric flasks: 10 mL and 100 mL.

6.4.2 Syringes: 2 uL, 5 uL, 10 uL, 25 uL, 50 uL, 100 uL, 500 uL, 2.5 mL, 5 mL, and 25 mL.

6.4.3 Syringe valve - Two-way, with Luer ends (three each) if applicable to the purging device.

6.4.4 40mL glass VOA vials with caps/septa (Sci/Spec Precleaned (2000 class) and QA Analyzed (3000 class) vials are washed to EPA Protocol “B”).

6.4.5 Assorted Mini-inert and Teflon-lined capped vials for standard storage.

7. Reagents and Standards

7.1 Analyte-Free Reagent Water (also referred-to as VOC-free water or DI water elsewhere in this SOP) - prepared by eluting tap water through a column of activated charcoal granules to remove traces of volatile organic compounds, or the Whirlpool Water Filter system.

7.2 Purge and trap grade methanol - from Fisher Scientific *or equivalent* quality of solvent from other vender will be used for standard preparation. Each new batch of solvent is checked by analyzing a 200µL aliquot of the methanol in a 5mL aliquot of pure water, or 1.6mL per 40ml vial of pure water. The new batch is acceptable if the analysis does not detect any contaminants that interfere with the measurement of target analyte compounds, or contain unacceptable levels of non-target compounds. While the criteria for method blank evaluation can be used for guidance, stricter criteria will be beneficial as potential contamination from various sources may add-up to impact the analysis.

7.3 The standards used for this SOP are discussed below. *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.* Stock solutions for calibration standards are:

Standards	Vendor	Cat. No.	Concentration
8260 Mix	Ultra	DWM-589N	2000 ug/mL
Gas Mix	Restek	30042	2000 ug/mL
Ketone Mix	Restek	30006	5000 ug/mL
Additional Mix	Ultra	CUS-6268	400-8000 ug/mL
Internal Standards	Restek	30241	2500 ug/mL
Surrogates	Restek	30240	2500 ug/mL

7.3.1 All of the primary standards are labeled as VPyymmddX,

where: VP = **V**olatile **P**rimary standard
yymmdd = date the standard is received
X = the order that the standard is logged into the Log Book on that date, in increasing alphabetical order

7.3.2 All unopened ampulated primary standards will be replaced either by following the expiration instructions from the manufacturer, or after two years from the date received if no expiration date was provided. For an opened stock standard ampule or prepared stock solution, replace after **6 months** from the date it was opened or sooner if the standards have degraded or evaporated.

7.3.3 Standards are stored separate from samples, at a temperature of ≤ 6º C or as recommended by the vendor. The lab stores standards in a separate freezer which is maintained between -10 and -20 º C.

7.4 Stock Solution for ICV (independent "second" source, *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to NIST reference materials.*):

Standards	Vendor	Cat. No.	Concentration
8260 Mix	Accu Standard	M-502A-R	200 ug/mL
Gas Mix	Accu Standard	M-502B	200 ug/mL
Additional Mix	Accu Standard	M-8260-ADD	200 ug/mL

7.4.1 The labeling and storage for all ICV primary standards should be handled following the similar procedures for primary calibration standards listed above in **Section 7.3.1 through 7.3.3.**

7.5 Working Standard solutions:

7.5.1 Working standard solution for calibration mix: place 2520 µL of methanol into a 4mL vial fitted with Teflon septum. Transfer 200 µL of 8260 Standard Mix (Cat. No. DWM589N), 200 uL of Gas Mix (Cat. No. 30042), 80 uL of Ketone Mix (Cat. No. 30006) and 1000 uL of Additional Mix (Cat. No. CUS-6268) into the vial to make a solution of all analytes at 100ug/mL.

Note: using the syringe, add the standards below the surface and into the methanol by pushing the syringe plunger smoothly to the end. Make sure all of the standard mix is transferred into the methanol before the syringe is removed.

Gently invert the 4mL vial several times to ensure proper mixing. Repeat this process for all mixtures. This working standard is then transferred into four 1 mL vials with mininert valve. Keep all four vials in freezer at -10 to -20°C. Only take one vial out and use it for calibration and QC samples. A new vial should be taken out and used weekly, or as needed based on standard degradation.

7.5.2 Working standard solution for ICV: place 700 uL of methanol into a 1mL vial fitted with Teflon septum. Transfer 100 uL each of Non-Gaseous standards (M-502A-R), 8260 Additional standards (M-8260-ADD) and Gaseous standards (M-502B) into the vial to make a solution of all analytes at 100ug/mL.

7.5.3 Surrogate Standard solution (SS): four surrogate compounds are used for analysis: dibromofluoromethane, 1,2-dichloromethane-d4, toluene-d8, and bromofluorobenzene. The working standard is prepared by transferring 160 uL of the stock surrogate standard solution

(Cat. No. 30240) into a 4 mL vial with 3840 uL of methanol to make a solution at 100 ug/mL.

7.5.4 Internal Standard solution (IS): three internal standards used for analysis: fluorobenzene, chlorobenzene-d5, and 1, 4-dichlorobenzene-d4. The working standard solution is prepared by transferring 160 uL of the stock internal standards (Cat. No. 30241) into a 4 mL vial with 3840 uL of methanol to make a solution at 100 ug/mL.

7.5.5 Internal Standard and Surrogate Standard Mix solution (IS/SS): The working standard of IS/SS solution is prepared by transferring 400 uL each of the IS stock solution (Cat. No. 30241) and SS stock solution (Cat. No. 30240) into a 4 mL vial with 3200 uL of methanol to make a 250ug/mL solution. Once prepared this solution is stored in the Standard Adding Module of either 4551A or 4552 autosamplers. 1uL of the solution is added to all Calibration Standards, ICV, blanks, LCS and samples. At a 5 mL purge volume, this yields a concentration of 50ug/L or ug/Kg.

7.5.6 All of the working standards are labeled as VWyymmddX,

where: VW = volatile working standard
yymmdd = date the standard is prepared
X = the order that the standard is logged into the Log Book on that date, in increasing alphabetical order

See **Table 2** for details on making working standard solutions. Working Standards are good for **one month**. All standards made from a primary standard must not exceed the primary standard's expiration date.

7.6 All of the standard information is recorded in the LIMS standard/spike Logbook upon receiving or preparation (see **Figures 1 and 2**). All vials containing working standards must be labeled according to the current version of SOP No. 80.0001 Standard Preparation, Equivalency and Traceability. Be sure the vial label is not worn or difficult to read. Any vial whose label becomes worn or difficult to read should be re-labeled.

8. Procedure

8.1 Instrument Set up.

8.1.1 The purge and trap systems operating conditions are as follows:

<u>Aqueous (including both 5mL and 25mL samples) and medium soil samples</u>

Purge	11 min at ambient temperature
Dry Purge	2 min
Desorb*	2 min at 190 °C

Bake*	8 min at 200 °C
Purge Flow	40 mL/min
Trap Type	OI Analytical Trap #10 or K type trap (instrument dependent).
Transfer Line Temp.	125 °C

Low level soil samples

Preheat	2 min
Purge	11 min at 40 °C
Dry Purge	2 min
Desorb*	2 min at 190 °C
Bake*	8 min at 200 °C
Purge Flow	40 mL/min
Trap Type	OI Analytical Trap #10 or K type trap (instrument dependent).
Transfer Line Temp.	125 °C

* Desorb time and Bake times may be varied to optimize the instrument performance, however these times must stay consistent between calibration and sample analysis.

8.1.2 Instrument operating conditions are as follows:

General Gas Chromatography Conditions

For V1 and V2:	
Carrier Gas	Helium (99.999%)
Column Flow	25mL/min
Initial Temperature	38 °C hold for 1.8 min
Temperature Program	10 °C/min to 120 °C, then 15 °C/min to 240 °C
Final Time	2 min
Injector Temperature	125 °C
Transfer Line Temperature	280 °C

For V5, V6, V10:	
Carrier Gas	Helium (99.999%)
Column Flow	25mL/min
Initial Temperature	38 °C hold for 1.8 min
Temperature Program	12 °C/min to 200 °C, then 20 °C/min to 240 °C
Final Time	2.7 min
Injector Temperature	125 °C
Transfer Line Temperature	280 °C

General Mass Spectrometry Conditions

For V1 and V2:	
Mass Range	35-300 amu
Scan Speed	1.6 scans/min
Ionization Mode	70 eV positive ion
EM Voltage	same as tune
For V5, V6, V10:	
Mass Range	35-300 amu
Scan Speed	0.97 scans/min
Ionization Mode	70 eV positive ion
EM Voltage	same as tune

In the event that these conditions are changed, Enviroquant Data Acquisition methods containing the actual GC operating conditions are copied and sent to the network along with all GC/MS raw data files. They are located in a folder in the sequence batch called “Zacq”.

8.2 Tuning:

8.2.1 The GC/MS must be tuned to meet 4-bromofluorobenzene (BFB) criteria every 12 hours when standards, blanks or samples are to be analyzed. All of the analysis information is to be recorded in the Instrument Run Logbook (**Figure 3**). The logbook is issued by the QA dept and will be returned for archiving when all pages are used.

8.2.1.1 Procedure for performing tune - Use the GC/MS conditions in **section 6.1.1.5** to perform the tune analysis.

Inject 2 µL of the working tune standard (50ng) directly into the GC/MS through the septum injection port using a 10uL syringe. Alternatively, the BFB can be introduced through the purge and trap system, as a sample is.

A typical BFB chromatogram is shown in **Attachment 2.**

8.2.1.2 Acceptance criteria for tune - The mass spectrum of BFB must be acquired across the peak. The primary mean for evaluating ion abundance is averaging three scans: the peak apex scan and the scans immediately proceeding and following the apex. One of following alternates may be used to evaluate the tune: averaging the entire BFB peak, the single scan of apex, the single scan before apex, or the single scan after apex. Background subtraction is required and accomplished by subtracting a single scan no more than 20 scans prior to the beginning of the elution of BFB. It is important that the analyst does not selectively add or subtract scans to meet the tune criteria.

A typical mass spectrum and mass spectral listing of the tune is listed in **Attachment 3**.

The acceptance criteria from SW8260C are as follows:

Mass	Ion Abundance
50	15.0 - 40.0% of mass 95
75	30.0% - 60.0% of mass 95
95	base peak, 100%
96	5.0 - 9.0% of mass 95
173	< 2.0% of mass 174
174	>50.0 % of mass 95
175	5.0 - 9.0% of mass 174
176	95.0 - 101.0% of mass 174
177	5.0 - 9.0% of mass 176

The acceptance criteria for Instrument V10 are taken from EPA SOM01.2 SOW*, and are as follows:

Mass	**Ion Abundance**
50	15.0 – 40.0% of mass 95
75	30.0 – 80.0% of mass 95
95	base peak, 100%
96	5.0 – 9.0% of mass 95
173	< 2.0% of mass 174
174	50.0 – 120.0% of mass 95
175	5.0 – 9.0% of mass 174
176	95.0 – 101.0% of mass 174
177	5.0 – 9.0% of mass 176

Once the mass spectrometer passes the BFB tune, all subsequent standards, samples, blanks and QC samples analyzed within the 12-hour shift must be analyzed using identical mass spectrometer instrument conditions.

*Alternate tuning criteria allowed per SW8260 Section 11.3.1.2. These criteria are better suited for use by the newer GC/MS systems such as the HP5975C.

8.3 Initial Calibration - Initial Calibration is performed after the instrument passes the tune requirements. Initial Calibration is required after major instrument maintenance including source cleaning and/or changing column. Initial Calibration will also be performed if Continuing Calibration analyses do not meet QA/QC criteria.

8.3.1 Five calibration standard solutions are required for all target compounds. Standard concentrations of 5, 20, 50, 100, 200 µg/L (or in µg/kg for soil samples) for typical 5 mL or 5 g sample analyses; 0.5, 4, 10, 20, 40 µg/L for 25 mL aqueous sample analyses are required.

(See Section 16 for more details on low level calibration) The lowest standard concentration is typically at or below the reporting limit for the analysis. This is the level closest to the method detection limit (MDL). There may be project-specific requests to calibrate the instrument to the 1 ug/L level using a 5ml purge. This procedure may involve the addition of a sixth level to the initial calibration at the 1 ug/L concentration, or replacement of one of the other concentrations to maintain a 5 level calibration. This is determined by the requirements of the specific project through discussions between the Project Manager and Supervisor. There also may be occasional requests to report results to a limit below the lowest initial standard concentration. These must be documented and discussed in the report narrative. Any request for non-routine calibration should be discussed with the laboratory Supervisor and Project Manager to insure the resulting data meets project and method requirements and the procedures used and the quality of the data are fully documented.

DoD– the ICAL range shall consist of a minimum of 5 contiguous calibration points for organics, for all target analytes and surrogates reported. The low-level standard must be less than or equal to the reporting limit.

Several state and government programs have specific QA/QC Requirements and Performance Standards for the Initial Calibration. Refer to the individual state/government documents for more details. In particular, Dept. of Defense requires the evaluation of SPCC/CCC compounds in both the ICAL and CCV. See **Attachment 5** for criteria.

Low level (25 mL) Calibration is documented in **Section 17**.

The calibrations for aqueous and medium level soil samples are performed at ambient temperature purge. The calibrations for low-level soil samples are performed at the same temperature used for sample analysis, using heated (40°C) purge condition.

8.3.2 Initial Calibration standards are made-up as follows:

Initial calibration standards are prepared by adding working standards into 40mL organic-free water using appropriate volume syringes. In aqueous analysis, 5mL of each 40mL solution are transferred into the purge chamber by the autosampler. In soil analysis, 5mL of each 40mL solution are transferred into vials with Teflon caps manually using gas tight syringes and these vials are heated and purged. The IS/SS may be added automatically by the autosampler.

DoD QSM: ICALs require surrogates to be added manually (to achieve multiple level calibration) according to **Table 2**.

Calculation for Initial Calibration - A typical chromatogram of a 50µg/L standard followed by the Quantitation Report is shown in the attachments to this document.

From the 5 level calibration the relative response factor (RRF) for each target compound is determined using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

where: A_x = area of the characteristic ion for the target compound to be measured
A_{is} = area of the characteristic ion for the associated internal standard
C_{is} = concentration of the internal standard
C_x = concentration of the compound to be measured

When using the DB624 column, note the following when determining the RRF:

- Cis- and trans-1, 2-dichloroethenes are separately calibrated.
- O-Xylene is calibrated by itself; the m- and p-isomers are summed together.

The mean relative response factor is determined by averaging the 5 level values.

The % Relative Standard Deviation (%RSD) of the RRF is also calculated using:

$$\% RSD = \frac{SD}{Mean} \times 100$$

where: SD = Standard Deviation, and

$$SD = \sqrt{\frac{(Xi - X)^2}{n-1}}$$

where: Xi = each individual value used to calculate the mean
X = the mean of n values
n = the total number of values = 5

8.3.3 Initial Calibration acceptance criteria for SW-846 8260C protocol is as follows:

- The relative retention time (RRT) for each of the target analyte including the surrogates at each calibration level must be within ± 0.06 RRT of the mean RRT for each compound.
- The area response for each internal standard at each calibration level must be within the inclusive range of –50% to +100% of the mean area response of the internal standard in all of the calibration levels.

- The retention time (RT) shift of the internal standards at each calibration level must be within ± 0.5 minutes compared to the mean retention time over the initial calibration range for each internal standard.
- If the RSD of any target analytes and/or surrogate compounds is less than 20%, then the RRF is assumed to be constant over the calibration range and the average RRF is used for quantitation. If the calibration is not linear, make sure whether the problem is related to calibration standards or instruments.
- A minimum RRF is suggested, see **Table 3**.
- No quantitation ion may saturate the detector.
- Given the large number of target analytes, it is likely that some analytes may exceed the acceptance limit. In those instances, the initial calibration is deemed acceptable if the following conditions are met (in order of preference):

(1) Ten percent (10%) of the compounds are allowed to be greater than 20% RSD with a maximum of 50% RSD. The number of outliers depends on the number of compounds of interest. Project specific compounds/common compounds are **not allowed** as one of the outliers. Initial Calibrations with over 10% of the compounds above the 20%RSD may be used for screening purposes.

(2) Linear calibration: a least squares regression may be used. The analyst may employ a regression equation for the analyte(s) that does not pass the earlier approach. The regression will produce the slope and intercept terms for the following linear equation:

$$y = mx + b$$

Where y = instrument response (peak area)
m = slope of the line
x = concentration of the calibration standard
b = intercept

It is important that the origin (0, 0) is not included as the sixth calibration point and that the above equation is not forced through the origin.

The linear regression is deemed acceptable if the correlation coefficient **$r \geq 0.995$**.

(3) Non linear calibration: The analyst may employ a non linear regression coefficient of determination (COD). The second order quadratic fit will have the following equation:

$$y = ax^2 + bx + c$$

Where y = instrument response (peak area or height)

a and b = slope of the curve
x = concentration of the calibration standard
c = intercept

In performing second order quadratic fit, the analyst should not force the curve to pass through the origin (0, 0). In addition, the origin should not be used as an additional calibration point. Quadratic fit (second order) requires 6 standard points.

From the quadratic fit, the "goodness of fit" is evaluated by calculating the coefficient of determination (COD). In order to be acceptable, the COD of the polynomial must be **≥0.99**.

8.3.4 Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. This is performed by analyzing the 50 ppb standard prepared in **Section 7.4.** The standard ID is documented in the run log. The acceptance criteria are as follows:

For routine SW 8260 analyses, the calculated value of the analyte in the ICV should be 70 – 130% of the expected value (35 – 65ng/uL).

For DoD analyses, the calculated value of the analyte in the ICV should be 80-120% of the expected value (40-60ng/uL), with no allowance for poor performing compounds.

If the above criteria are not met, the analyst has to evaluate the integrity of the primary and second source standards. First, reanalyze the ICV. Preparation and analysis of a new initial calibration may be required; however failure to meet the control limits does not in itself negate the Initial Calibration validity. Certain compounds may not meet the criteria under the best of circumstances. Some compounds will require a wider recovery limit. In some cases, the analysis of samples may be used for screening purposes when the ICV fails.

8.3.5 Corrective Action for Initial Calibration - Depending on which compound failed the criteria, corrective action included preparing fresh standards, source cleaning, reconditioning or changing the trap. Document the actions and resolution in the LIMS maintenance log.

8.3.6 Initial calibration acceptance criteria must be met before any sample, blanks or QC is to be analyzed. There may be circumstances where project-specific criteria allow the use of an initial calibration where one or more compound exceeds the acceptance criteria. For example, work performed under the Massachusetts Contingency Plan (MCP) allows up to 20% of the non-CCC analytes (calibration check compounds (CCC) are: vinyl chloride, 1, 1-dichloroethene, chloroform, ethyl benzene, toluene, and 1, 2-dichloropropane) to have %RSD > 30 or r < 0.99. This situation is to be discussed with the Technical Director or Project Manager for approval. Any compound not passing the calibration criteria will be flagged on Form 7 and the information included in the data report. This information will also

be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.

8.3.7 If necessary, the reference spectra in Target are updated from mid-point calibration (50 ppb standard), or from the continuing calibration standard.

8.3.8 Upon the successful completion of the initial calibration, the raw data are arranged in increasing concentration levels together with BFB tune. Raw data include chromatograms and quantitation reports plus any documentation of manual integrations. Refer to SOP No. 110.0008 for details on the need for and documentation of manual integration. A copy of the initial calibration summary listing the RRF and %RSD of each target analyte is also included. These raw data are to be filed separately for each of the instruments.

8.3.9 Initial calibration data must be archived in the company's organic analysis calibration (OCAL) database. The information in **Section 8.2.8** is brought to the Data Reporting area and left in the tray for filing OCAL data. The Data Reporting department will scan the calibration printouts into the optical filing database for long-term archiving. This may be done at anytime after the ICAL is deemed acceptable.

8.4 Continuing Calibration (CCV) - Continuing Calibration using standards containing all the target compounds at 50µg/L (or 50µg/kg) are performed every time samples are analyzed to ensure that the GC/MS continues to meet instrument sensitivity and linearity requirements.

8.4.1 Frequency of Continuing Calibration- A Continuing Calibration must be performed once every 12 hours. If time remains in the 12-hour time period after meeting the acceptance criteria for the Initial Calibration, samples may be analyzed using the mid-point ICAL standard as the continuing calibration verification. The Continuing Calibration is required whenever blanks, LCS and samples are analyzed.

8.4.2 Procedure for performing Continuing Calibration - The Continuing Calibration is performed at 50µg/L (µg/kg) injection. The IS/SS are added automatically by the autosampler. Calculate the % difference between the Continuing Calibration RRF and those from the most recent Initial Calibration.

The % difference is determined as follows:

$$\% \text{ Difference} = \frac{RRF_c - RRF_i}{RRF_i} \times 100$$

where: RRF_c = relative response factor from continuing calibration
RRF_i = mean relative response factor from the most recent initial calibration that meets acceptance criteria.

Use % Drift when using least squares or non-linear calibration.

$$\% \text{ Drift} = \frac{Conc_c - Conc_t}{Conc_t} \times 100$$

where: $Conc_c$ = concentration obtained from continuing calibration
$Conc_t$ = theoretical concentration of standard

8.4.3 Continuing Calibration acceptance criteria:

- The RRF for each compound should be greater than those listed in **Table 3**.
- Twenty percent (20%) of the compounds are allowed to be greater than 20 %D, with a maximum of 50 %D. The number of outliers depends on the number of compounds of interest. Project specific compounds/common compounds are not allowed as one of the outliers.
- The area response for the internal standards must be within the inclusive range of –50% to +100% of the area response of the internal standards in the mid-point ICAL standard level when run on the same day. Otherwise the CCV will be used for comparison of IS areas.
- The internal standard retention time of the calibration verification standard must be within 30 seconds from that of the mid-point calibration (50ug/mL) of the associated initial calibration when run on the same day. Otherwise the CCV will be used to set the day's RRT to account for potential changes due to GC column maintenance.
- No quantitation ion may saturate the detector.

Several states have specific QA/QC Requirements and Performance Standards for the Continuing Calibration. Refer to the individual state documents for more details.

8.4.4 Corrective Action for Continuing Calibration - Investigate the calibration to confirm that calculations have been performed correctly and that all integrations are correct. Depending on which compound(s) fail(s) the criteria, corrective action includes preparing fresh standards, source cleaning, reconditioning or changing trap. Repeated failure to pass continuing calibration may necessitate performing new initial calibration. See **Attachment 8** for specific QC criteria and corrective action.

Note—the following symptoms and corrective actions commonly occur in this analysis. If the gaseous compounds are low, this typically indicates too high purge flow rate, "blowing" these compounds through the trap. The gaseous compounds are also more sensitive to small leaks in the system between the purging chamber and the injection port. If the higher boiling point compounds are too low, this typically indicates too low purge flow, or too low desorb temperature. A cold spot in the transfer line could also cause loss of the higher boiling compounds. If the brominated compounds or 1,1,2,2-tetrachloroethane are low, this typically indicates active sites in the system causing break-down of these compounds. If

methylene chloride or acetone are too high or too low, this typically indicates contaminated "blank" water used to make the CCV or possibly in the ICAL (CCV too low).

8.4.5 Continuing calibration acceptance criteria must be met before any samples or QC is to be analyzed. There may be circumstances where project-specific criteria allow the use of a continuing calibration where one or more compound exceeds the acceptance criteria. For example, work performed under the Massachusetts Contingency Plan (MCP) allows up to 10% of the non-CCC analytes to have %D > 30. This is to be discussed with the Technical Director or Project Manager for approval. Any compound not passing the calibration criteria will be flagged on Form 7 and the information included in the data report. This information will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.

8.5 Sample Analysis - Samples are allowed to warm to ambient temperature before analysis.

8.5.1 Aqueous (method SW5030) - Samples are analyzed in 5mL or 25mL aliquots depending on desired reporting limits or by project specification. The sample is entered into the instrument run log, the bottle number documented, and the vial is placed in a location in the autosampler tray. Immediately prior to analysis, an aliquot of the sample is withdrawn from the VOA vial by the autosampler using a syringe. An aliquot of the combined internal standard/system monitoring standards (IS/SS) are added and the sample aliquot is transferred to the purge-and-trap sparger and injected into the sparger vessel. The sample is ready for analysis.

8.5.2 Soil samples are analyzed using heated purge for low level analysis and methanol extraction approach for medium level analysis.

8.5.2.1 Low level soil analysis (method SW5035A) – Samples are received preserved in DI water or sodium bisulfate (NaHSO4) solution. The vial also contains a small Teflon-coated stir bar. The "empty" vial/preservative solution/stir bar is weighed prior to shipment to the client/field. The vial is reweighed prior to analysis and the sample weight is determined by the difference in weight. The additional Spectrum RI sample ID label weight must also be accounted for in the final weight. The weight is recorded in the appropriate log book, and the sample and its jar number are logged into the instrument run log book. The vial is allowed to warm to room temperature and loaded into the autosampler. Prior to analysis the autosampler places the vial in a temperature controlled heating block to equilibrate to the analysis temperature 40 °C. For low level soil analysis, the instrument calibration and all QC analyses are to be performed at the same temperature (40 °C) as the sample analyses.

8.5.2.2 EnCore Samples – Samples collected into self-contained EnCore (or similar) devices are often collected. Samples are extruded from the EnCore into preservative solution (typically two EnCores into DI water and one into methanol). The aliquots are then analyzed by the low level soil or medium level soil procedure as appropriate.

8.5.2.3 Unpreserved Soil Samples – Samples for Method 8260 should be preserved per Method 5035. If soil samples are received unpreserved, but per discussion with the client they are still to be analyzed, the following procedure is used. Approximately 5.0-5.5g soil is weighed into a pre-weighed vial containing DI water and a stir bar. This should be done as soon as possible following sample receipt. Be sure to take the soil aliquot from below the soil surface in the sample jar to minimize headspace loss. The soil must be below the surface of the DI water in the vial. The sample is then batched up at the autosampler per the procedures listed under **section 8.4.2.1** above.

8.5.2.4 Medium level soil analysis – using field-preserved methanol sample aliquots. The customer collects approximately 5 g of soil sample into a pre-weighed 40mL vial containing 5mL methanol. At the laboratory the sample is weighed again to determine the soil weight by difference. The additional Spectrum RI sample ID label weight must also be accounted for in the final weight. A portion of the methanol extract is transferred into a 40 mL vial for analysis. The typical maximum methanol-water ratio is 100 µL of the methanol extract added to a total volume 5 mL sample, or 800 uL to a 40 mL vial. The prepared sample is analyzed using the aqueous sample procedure, using the water calibration to quantitate the medium level analysis.

8.5.2.5 Medium level soil analysis – if no field-preserved aliquot is submitted for analysis. Weigh 5.0-5.5g of soil sample into a 15 mL vial, and then quickly add 5mL of methanol. Be sure to take the soil aliquot from below the soil surface in the sample jar to minimize headspace loss. Cap and shake for 2 minutes. After phase separation, the methanol extract is transferred into a 4mL vial with no headspace for storage. When the extract is ready for analysis, up to 100 µL of the methanol extract is added to a 5 mL aliquot of analyte free water, or 800 uL to a 40 mL vial of DI water. Use the water calibration to quantitate the medium level analysis.

8.5.3 Sample Dilution - Sample dilution is performed to ensure that all of the target analytes are determined within the instrument calibration range. Based on the concentration determined in the initial sample analysis, if needed, the analyst will determine the dilution factor required to perform the diluted analysis such that the target compounds will be determined at or above the mid-point calibration. It is important to note that due to over-saturation (column or detector), the target compounds that were determined to exceed the calibration range are usually underestimated (detected concentration lower than actual) in the initial run.

8.5.3.1 Low level aqueous sample – Dilutions for aqueous samples are prepared in 40mL vials with Teflon septum/seal. An appropriate volume of analyte-free water is added to the vial. The proper volume of sample is measured in a gas tight volumetric syringe; 4mL for 10X dilution, 1mL for 40X dilution, 400uL for 100X dilution…etc. The unopened sample vial is used for the dilution. The sample is withdrawn by removing the cap; the septum seal is not punctured. The measured amount of sample is slowly injected into the vial below the surface of the analyte-free water. The vial is filled with analyte free water and closed. A calibrated 40mL vial is used to compare final volume and is kept

at the bench. The amount of sample used to prepare the dilution must be noted in the VOA Dilution logbook (#90.0230) along with the final dilution factor to allow for double-checking of dilution calculations. Any secondary dilution used must be clearly described in the logbook. If more space is necessary, the back of the logbook page is to be used, with a note on the front of the page to refer to the back of the page. If an unopened vial is not available for dilution analysis, the situation must be discussed with the Supervisor and Project Manager prior to using a previously opened vial. If a previously opened vial is approved for use, this must be noted on the run log book and on the data review checklist submitted with the data for review to allow discussion in the project narrative.

8.5.3.2 Low level soil sample - If a smaller volume preserved soil aliquot is provided by the client, a dilution analysis may be performed. Depending on the dilution factor, sample weight down to 0.5 gram or a 10X dilution may be used. Any dilution more than 10X, using less than 0.5 gram will necessitate using the medium level methanol preserved approach below.

8.5.3.3 Medium level approach -Depending on the dilution factor, reduce the methanol extract from the ratio of 100 µL/5mL to as low as 5 µL/5mL pure water, or from 800 uL to 40 uL per 40 mL vial. Further dilution will require secondary dilution of the 10mL methanol extract. The amount of methanol used per 40mL vial must be noted on the log book along with the final dilution factor to allow for double-checking of dilution calculations. Any secondary dilution used must be documented in the VOA Dilution logbook (#90.0230).

8.5.3.4 Criteria for reporting dilutions. The final dilution analysis is always reportable. This analysis should have the concentration of the highest compound near or above the mid range (100 ppb level for analytes using 5 to 200ppb range) of the initial calibration.

8.5.3.5 If an initial analysis is performed that meets all QC criteria with the exception of compounds exceeding the upper calibration limit, this analysis is generally also reported. The sample ID of the initial (less dilute) analysis is unchanged and the ID of the dilution analysis has the letters “DL” appended to the sample ID. Those compounds exceeding the calibration range are qualified with the “E” flag on the data sheet for the less dilute analysis, and, if reported on CLP-type forms, qualified with a “D” if detected and reported in the more dilute (DL) analysis. Diluted samples are uploaded using the Type of DL and RunNo of 2.

8.5.3.6 If the laboratory has prior information that a sample may contain concentrations of target or non-target compounds exceeding the calibration range of the instrument the initial analysis may be performed at dilution. This information may include project history, prior analyses, screening results, results of other (such as GRO or TPH) analyses, solvent or petroleum odors detected during other analyses or during sampling, etc. This information should be used to prevent overloading and

contamination of the autosampler/purge and trap system. If the initial analysis is performed at dilution, and the results of this analysis are acceptable (at or above the mid range calibration standard, or significant non-target compound concentrations), a less dilute analysis is not required. The sample ID is not changed by adding "DL", but the initial analysis at dilution is noted on the data review checklist included with the data submitted for review, to allow discussion in the project narrative.

8.5.3.7 If the initial analysis fails QC criteria, it is not typically reported. If only the dilution analysis is reported, the letters DL are not added to the sample ID, but the dilution is noted on the data review checklist submitted with the data for review. The letters "DL" indicate a second dilution, not an initial analysis at dilution.

8.5.3.8 If the initial and dilution analysis together demonstrate matrix interference, such as with surrogate/internal standard recoveries out of limit in both analyses, both runs are typically reported. Also if the initial analysis provides important information to the project, it should be reported, with the QC exceedences noted on the data sheets (flagged surrogates on Form 2, flagged internal standards on Form 8, "E" qualifiers on Form 1, etc) and fully described in the data review checklist included with the data submitted for review so they may be included in the project narrative.

8.5.4 Acceptance Criteria for Sample Analysis are as follows:

- The sample must meet analysis holding time.
- The sample has to have a compliant tune, initial calibration and continuing calibration.
- The sample has to have a compliant method blank.
- The sample has to have a compliant LCS.
- All surrogate recoveries are within control limits (See **section 10.4**) with the exception of one outlier, unless specified in client project. The outlier must have recovery value above 10%.
- The internal standard areas must meet the –50% to +100% criteria. If the criteria are not met, the sample should be rerun. In some circumstances (high TIC content) this is not necessary, see the corrective action tables, **Attachment 8** of this SOP.
- The relative retention time (RRT) of each of the IS must not shift more than ± 0.06 RRT units from the CCV or the mid-point standard of initial calibration.
- All of the target analyte concentration should be below the calibration range excluding the "solvent" front, no ion should saturate the detector
- If the previous run contains any target analyte above the calibration range, and the same target analyte is detected above the reporting limit in the subsequent run, the subsequent run has to be repeated to demonstrate that the compound is not due to carry-over.

9. Data Reduction and Calculations

9.1 Identification of Target Compounds - Two criteria are used to identify target compounds:

9.1.1 Relative retention time (RRT) - The sample component RRT must agree within ± 0.06 RRT units of the RRT of the component in the associated continuing calibration standard. The relative retention time is determined as follows:

$$RRT = \frac{\text{Retention of target compound}}{\text{Retention time of associated internal standard}}$$

9.1.2 Comparability of mass spectra - The requirements for qualitative verification by comparison of mass spectra is as follows:

- All ions present in the standard mass spectra at a relative abundance greater than 25% must be present in the sample spectrum.
- The relative intensities of ions specified above must agree within ± 20 % [method allows 30%] between the standard and sample spectra.
- Ions greater than 10.0% in the sample spectrum but not present in the standard spectrum must be considered; this may be due to potential co-eluting interference.
- The halogenated target analytes should contain the characteristic chlorine and bromine isotopic ratios.
- If the criteria above are not met but in the technical judgment of the analyst that the identification is correct, the lab will report the identification and proceed with the quantitation. Any suspect identification should be described on the data review checklist submitted with the data for review.

9.2 Identification of non-target compounds [tentatively identified compounds (TICs)] - Client may request the analysis of TICs. Non-target compounds will be searched using the NIST/EPA/NIH library. The non-target compound will be reported as part of the analysis requirement if client requested and:

9.2.1 The client requires a full data package deliverable, including CLP, Level 4 or New York ASP-B reporting format (exceptions are projects that have a short list of target analytes such as TCLP, BTEX, STAR list or projects that the client specified no TIC reporting).

The non-target compounds will be identified and reported if:

- Its response is greater than 10% of the closest eluting interference free internal standard.
- Its retention time is within the range of 30 seconds before the elution of the first target compounds, and 3 minutes after the elution of the last target compound.
- Unless specified, up to **10** TIC are to be reported.

9.2.2 Guidelines for making tentative identification :

- Ions greater than 10% in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%.
- Molecular ions present in reference spectrum should be present in sample spectrum.
- Ions present in sample spectrum but not in the reference spectrum should be reviewed for co-eluting interferences.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed with caution because of background contamination and/or co-eluting interferences.
- The lab shall not report semivolatile target compounds.
- The non-target compounds will be reported as "unknown" if no valid tentative identification can be made (as based on analysts' interpretation**)**.
- If the Quality (Qual) of the match as determined by the library search program is above 85%, it typically meets the criteria above, and is considered a tentative identification. If the Qual is less than 85%, the match typically does not meet the criteria above, and is usually identified as "unknown".

9.3 Quantitation of target compounds - The initial calibration is used to quantitate the target compounds. It is important to note that the concentrations of the target compounds not exceed the calibration range of 200 µg/L (µg/kg) for all compounds other than the m- and p-xylenes (at 400 µg/L or µg/kg) in the analyses of 5 mL water or 5 g soil samples. In the case of 25 mL water sample analyses, the concentrations for all the compounds should be less than or equal to 40 µg/L. Any target analyte concentration that exceeds the calibration range will be diluted and reanalyzed.

9.3.1 Manual integration will be performed if needed and documented according to the current revision of SOP 110.0008, Manual Integration of IC, GC and GC/MS Chromatographs. Manual integration is appropriate when sample-specific chromatographic conditions prevent the automatic integration routines from properly assigning baseline, resulting in improper quantitation. Manual integration is prohibited from use to achieve any specific numerical QC criteria, such as to reduce surrogate peak area in order to be within recovery limits. The use of manual integration to purposefully modify non-compliant data for this reason is prohibited, and will subject the analyst to immediate disciplinary action. Any questions should be referred to the QA Director or Technical Director. The analyst will initial and date the quantitation report (electronic entry acceptable)with the proper reason code per SOP 110.0008, Manual Integration of IC, GC and GC/MS Chromatographs.

9.3.2 Determining the concentration of Target Compounds - Target compounds identified are quantitated using the following equation:

9.3.2.1 Aqueous concentrations are calculated using the equation:

$$Conc = \frac{(Ax)(Is)(V_0)}{(Ais)(RRF)(V_s)}$$

where: *Conc* = sample concentration in µg/L
Ax = area of the characteristic ion for the compound to be measured
Ais = area of the characteristic ion of the associated internal standard
Is = concentration of internal standard in µg/L
V_0 = purge volume, 5 for 5 mL water sample and 25 for 25 mL water sample
V_s = sample volume analyzed in mL
RRF = relative response factor

9.3.2.2 Soil concentrations are calculated using the equation below:

Low Level:

$$Conc. = \frac{(Ax)(Is)(5)(1000)}{(Ais)(RRF)(S)(W)}$$

Medium Level:

$$Conc. = \frac{(Ax)(Is)(V_t)(5)(Df)(1000)}{(Ais)(RRF)(S)(W)(V_a)}$$

where: *Conc* = Sample concentration in µg/Kg.
Ax = area of the characteristic ion for the compound to be measured
Ais = area of the characteristic ion of the associated internal standard
Is = concentration of internal standard in µg/L
Df = dilution factor, typically is equal to 1; if there is a secondary dilution, the dilution factor refer to the dilution between the first and the secondary dilution
RRF = relative response factor
V_t = total volume of methanol extract, in mL*
V_a = volume of the aliquot of the sample methanol extract, in uL
S = solid content expressed in decimal value
W = sample weight added to purge tube or for extraction, in gram

Solid sample results will be reported at dry weight basis unless otherwise specified. To convert soil results to a dry weight basis, divide the sample concentration by the percent solids (see SOP 110.0038 Percent Solids Determination)

* Data Correction for Methanol Preservation Dilution Effect. Based on the requirements of SW-846 Method 8000C, Section 11.10.05, analytical results for soil/sediment samples must be corrected for the Methanol Preservation Dilution Effect. The potential for under reporting concentration is more pronounced as the “as received”% moisture content of the soil/sediment sample increases, if this correction is neglected. Target analyte concentrations in solid samples preserved with methanol are subject to a systematic negative bias if the potential increase of the total solvent

volume during the methanol extraction process is not considered. This increase in extraction solvent volume is a direct result of the solubility of the entrained sample moisture (water) in the methanol. The total solvent volume is the additive sum of the volume of methanol and the entrained sample moisture that partitions into the methanol during extraction. The volume of water partitioned is estimated from the % moisture determination (as well as the assumption that 1 g of water occupies a volume of 1 mL). This is a conservative correction regarding calculated concentrations because some fraction of the sample's % moisture may not partition into the methanol, due to various physiochemical binding forces. The total solvent/water volume (Vt) is calculated as follows:

mL solvent/water (Vt) = mL of methanol + ((% moisture/100) × g of sample)

This "corrected" Vt value should be substituted directly for the Vt value shown in Section 9.3.2.2.

9.4 Determining the concentration of non-target compounds - An estimated concentration for non-target compounds is determined using the closest eluting internal standard. The formula to calculate the concentration is the same as those for water and soil samples described above. Total area counts from the total ion chromatograms are to be used for both the compound to be measured and the associated internal standard. A RRF of one (1) is assumed. An estimated concentration must be calculated for all tentatively identified compounds as well as these identified as unknown.

9.5 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

% Recovery (%R) = 100 x (SSR-SR)/ (SA)

where: SSR = spiked sample result
SR = sample concentration

SA = spike added

9.6 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

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

where: *RPD* = relative percent difference
D1 = first sample value
D2 = second sample value

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results. All analysts must have read this SOP and asked questions and received explanation for any areas they are unsure of. This SOP should be referred to often, and used as a reference for this procedure. Details of the procedure for documenting analyst proficiency can be found in the current revision of SOP 80.0016.

10.2 Method Blanks - Method Blanks are analyzed to determine the level of contamination associated with the processing and analysis of samples.

10.2.1 Frequency of Method Blank

- The Method Blank must be analyzed after each initial calibration and during each 12-hour time period when the instrument is used for analysis.
- The Method Blank must be analyzed after the Continuing Calibration and before any samples are analyzed.

10.2.2 Procedure for Method Blank:

The Method Blank is analyzed using 5 mL of organic-free water that is spiked with 1 µL combined IS/SS (Internal Standard/Surrogate Standard) to give a final concentration of 50 µg/mL. For 25mL purge analysis, the sample is spiked with 5 µL of the IS/SS solution to yield a final concentration of 5 µg/L. Blanks are analyzed as ambient purge for aqueous/medium soil samples. For low soil analysis, 5.0g of VOA-free Ottawa sand will be weighed into a 40ml VOA vial. (This information should be written in the VOA soil extraction logbook). Add 5ml of organic-free water and analyze by the heated purge procedure. The auto-sampler adds the IS/SS solution automatically.

10.2.3 Acceptance criteria for Method Blank:

- Percent recovery of surrogate must be within the control limits listed in **Section 10. 5.**

- All internal standard response must be within the –50% to +100% criteria. If the criteria are not met, the blank should be rerun.

- The concentration of each target compound found in the Method Blank must be less than its reporting limit except for certain common laboratory contaminants which have expanded acceptance criteria. In the case of 5 mL water/5 g soil sample analyses, common contaminants must be less than 2 times their PQL (methylene chloride, acetone and 2-butanone must be less than 10 µg/L or 10 µg/Kg); in the case of 25 mL water sample analyses, common contaminants must be less than 2 times their PQL (methylene chloride must be less than 1 µg/L, and acetone and 2-butanone must be less than 10 µg/L).

For *DoD* projects, the concentration of the target compounds in the method blank must be less than one-half of the Method Reporting Limit; the concentration for common laboratory contaminants such as methylene chloride and ketones, must not exceed the Method Reporting Limit.

- Any Method Blank that fails to meet any of the above criteria must receive corrective action. First investigate the ISS integrations and subsequent quantitation of the analytes in question to verify concentration. Check calculations. If the analysis is valid, a common corrective action is to reanalyze the blank. There may be situations where other corrective actions are appropriate depending on project-specific criteria, such as when the sample analysis resulted in a non-detect for the compound that failed the blank acceptance criteria.

10.2.4 All compounds present in method blanks that are also present in samples will be qualified with a "B" flag on data sheets reported to the client. The meaning of this qualifier will be described in the report. This will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.

10.3 Storage Blanks- Storage Blanks are analyzed to determine the level of contamination associated with the storage of samples. They are analyzed as a sample at the end of the analytical sequence.

10.3.1 Acceptance criteria for Storage Blanks:

- The concentration of each target compound found in the Storage Blanks must be less than its reporting limit except for certain common laboratory contaminants which have expanded acceptance criteria. In the case of 5 mL water/5 g soil sample analyses, common contaminants must be less than 2 times their PQL (methylene chloride, acetone and 2-butanone must be less than 10 µg/L or 10 µg/Kg); in the case of 25 mL water sample analyses, common contaminants must be less than 2 times their PQL (methylene chloride must be less than 1 µg/L, and acetone and 2-butanone must be less than 10 µg/L).

For *DoD* projects, the concentration of the target compounds in the Storage Blank must be less than one-half of the Method Reporting Limit; the concentration for common laboratory contaminants such as methylene chloride and ketones, must not exceed the Method Reporting Limit.

- Any Storage Blank that fails to meet any of the above criteria must receive corrective action. First investigate the ISS integrations and subsequent quantitation of the analytes in question to verify concentration. Check calculations. If the analysis is valid, a common corrective action is to reanalyze another Storage Blank. If the reanalysis confirms the contamination, the situation must be investigated and the affected client(s) must be notified of the potential contamination issue in the applicable refrigerator.

10.4 Laboratory Control Sample (LCS) -One LCS is prepared with each batch of up to 20 samples of the same matrix. The LCS is spiked with all compounds being reported for the method. If a non-routine compound is being reported, but no LCS is available, this must be noted on the data review checklist submitted with the data for review for inclusion in the project narrative.

- For an aqueous LCS sample, mixed standards are spiked into a 40 mL vial of organic-free water, resulting in concentrations at the mid-level standard. See **Table 2** for details on spiking volumes and solutions.

- For a solid LCS sample, add 5.0g of VOA-free Ottawa sand to a 40ml VOA vial. (This information should be written in the VOA soil extraction logbook). Add 5ml of organic-free water to the vial. Then add the commercially prepared standards with known values of VOC concentrations by spiking standards into the vial and analyzing by the heated purge procedure. Where applicable, a Lab Control Sample Duplicate (LCSD) will also be performed to evaluate reproducibility.

10.4.1 Acceptance criteria for LCS:

- Percent recovery of surrogates must be within the control limits listed in **Section 10. 5.**
- For regular SW8260 projects, the recovery is evaluated against the DoD QC limits for those compounds listed in QSM, and established in-house limits for all other compounds. Non-DoD projects may be evaluated by in-house limits or those limits specified by the project. Refer to the LIMS Test Information category/Test option/ specs for the most current QC control limits. See **Attachment 4** for *DoD* QC limits.
- All internal standard response must be within the –50% to +100% criteria. If the criteria are not met, the LCS should be rerun.
- If target analytes are outside of the acceptance limits, corrective action is required. Project-specific requirements, if available, will dictate the corrective action performed. See **Attachment 8** for further guidance.
- Due to the large number of target analytes, some recoveries (up to 4 for full list) may be out. These outliers are to be a mixture of poor performing compounds and sporadic failures. Compounds that constantly fail to meet criteria and are not poor performing compounds are not considered sporadic and require corrective action and investigation. Poor performing compounds for method SW8260C are marked with an asterisk in **Attachment 1**.

Per *DoD* requirements, analyses of <11 analytes, no marginal exceedences (ME) are allowed. For the analysis of 11-30 analytes, one ME is allowed; for the analysis of 31-50 analytes, two ME are allowed; for 51-70 analytes, (typical 8260 analysis) three ME are allowed; for the analysis of 71-90 analytes, four ME are allowed; for the analysis of >90 analytes, five ME are allowed. See **Attachments 5 and 6** for further guidance.

- Reporting LCS Results – If any compounds are outside of the acceptance limits, their recoveries are qualified with the "*" flag on the LCS recovery summary report (Form 3)

for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

10.5 Surrogate recoveries - The recovery of the surrogate compounds (also called System Monitoring Compounds) in all samples, blanks and LCS will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.5.1 The percent recovery of each of the surrogate compounds in blanks, samples, duplicate matrix spikes and LCS must be within the QC or in-house acceptance windows with the exception of one surrogate allowed out. Outliers must have a minimum of 10% recovery. Refer to the LIMS Test Information category/Test option/ specs for the most current QC control limits.

- For *DoD* projects, values from QSM:

	Solid	Aqueous
• 1,2-dichloroethane-d4	no limits given	70-120
• dibromofluoromethane	no limits given	85-115
• toluene-d8	85-115	85-120
• 4-bromofluorobenzene	85-120	75-120

10.5.2 Corrective action - If the recovery of the system monitoring compound is out of the acceptable window in the method blank or LCS, corrective action must be implemented. Corrective action should include verification of the internal standard area integrations, checking for errors in calculations and confirming the use of appropriate standards. In addition, the blank and/or LCS may be re-analyzed. If the recovery of the system-monitoring compound is outside of the acceptance limit for a sample, the data will be evaluated and corrective action (commonly reanalysis) will be taken. See **Attachment 8** for corrective action guidelines.

10.5.3 Reporting – The Target data reduction and reporting programs will flag any surrogate recovery outside of the acceptance limits with a "*"; the LIMS Level 2 reporting will flag any surrogate recovery outside of the acceptance limits with a "S". If the sample is reanalyzed and the system monitoring compounds are within the acceptance criteria for the reanalysis, and the reanalysis is within holding time, report the results of the reanalysis only. If the same system monitoring compounds are out in the reanalysis, report both sets of

analysis results to demonstrate matrix-related problems. This should be noted on the data review checklist submitted with the data for review for inclusion in the project narrative.

10.6 Matrix spike/matrix spike duplicate samples (MS/MSD) are analyzed at a frequency of once per twenty samples of similar matrix and procedure. The duplicate matrix spikes are used to assess the effect of matrix on the analytical accuracy and precision for the batch of samples. Where the client has not provided sufficient sample aliquots for a MS/MSD to be included in every batch, a duplicate LCS should be performed so analytical precision can be demonstrated. The duplicate matrix spikes are typically spiked with all of the target analytes. There may be project-specific MS spiking lists and criteria which take precedence.

10.6.1 The percent recovery of each compound is compared to the in-house acceptance limits, project-specific limits or **Attachment 6**. These limits are the same as used for LCS, but are used as advisory guidelines.

10.6.2 The following factors could greatly affect the accuracy and precision of the matrix spikes and matrix spike duplicates: sample heterogeneity, much higher analyte concentration in the sample, and matrix effect. The best measurement is obtained if the spike concentration is two to four times the analyte concentration in the unspiked sample.

10.6.3 If target compound recoveries are outside of the MS acceptance limits corrective action is required. See **Attachment 8** of this SOP for corrective action guidelines. Evaluate the percent recovery for those compounds outside of the recovery limit to the same compound in the LCS. At a minimum the corrective action will involve flagging any MS value outside of the control limit with an "*" on the recovery summary report form (Form 3) or an "S" on the LIMS Level 2 report. This is also noted on the data review checklist submitted with the data for review to allow for inclusion in the report narrative. Other corrective actions may include reanalysis of the MS/MSD at a higher spiking concentration, reanalysis of the MS/MSD by dilution of the sample, discussion of the issue with the Project Manager and the client.

For *DoD* projects, the %RPD limits for the duplicate set is 30%.

10.7 MDL studies are conducted to establish the detection limits applicable to this method. MDL verification at approximately 1-4 x MDL is analyzed after the study which also sets the DoD QSM Limit of Detection (LOD). MDL verification must be analyzed quarterly on each instrument used for DoD program work and annually per method as the MDL verification for NELAC. Please refer to the SOP No. 80.0005 Determination of Method Detection Limits for more detail.

11. Data Validation and Reporting

All raw data, including calibrations, QC results, and samples results, are reviewed for technical accuracy and completeness. The guidelines and procedures taken to ensure the data quality is listed in Section 11 of Quality Assurance Plan.

12. Data Management and Records Management

12.1 Electronic data generated from the analysis of Volatile 8260 (calibrations, QC, samples) is saved and managed per SOP 110.0029 Electronic Data Management.

12.2 All analysis information is documented in the individual Instrument Run/Injection Logbook regardless of run acceptance. No injections are deleted from the sequence.

13. Corrective Action Procedures

13.1 All QC exceedences require a corrective action response and documentation. The proper corrective action depends on the specific situation. Many corrective actions are spelled-out in this SOP. A table describing common occurrences, corrective actions and documentation is attached as **Attachment 8**.

13.2 Further information on the company's corrective action policy and procedures are included in the current revision of SOP No. 80.0007.

14. Health and Safety

14.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel. MSDS sheets were kept in the lab. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Health and Safety Plan. In general, use gloves, a lab coat, and safety glasses when handling these reagents and work in a hood whenever possible.

14.2 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

15. Pollution Prevention, Waste Management, Acronyms and Definitions

See **Table 1** List of Abbreviations, and sections 19.0 (Waste Management) and 20.0 (Definitions, Acronyms, and Abbreviations) of the current Quality Assurance Plan.

16. References

1. Environmental Protection Agency. Gas Chromatography/Mass Spectrometry Method 8260C, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, Revision 3 August 2006.

2. Spectrum Analytical, Inc. RI Division Quality Assurance Plan (QAP), current revision.

3. "Quality Systems Manual for Environmental Laboratories" Department of Defense, Final Version 4.1 April 2009 or current version.

4. Environmental Protection Agency. Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples Method 5035A, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, Draft Revision 1, July 2002.

5. Environmental Protection Agency. Purge-and-Trap for Aqueous Samples Method 5030B, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, Revision 2, December 1996.

6. Hewlett Packard and OI Analytical Instrument instruction manuals.

7. ASTM D6418 - 04 Standard Practices for Using the Disposable EnCore Sampler for Sampling and Storing Soil for Volatile Organic Analysis.

17. Low Level Calibration and Analysis

1. A low level calibration procedure is used to achieve a low level reporting limit at 0.5 ug/L for most of the target analytes, except for ketones. The purge volume for all standards, QC samples and field samples are 25 mL instead of 5 mL. The 5 level calibration standards are 0.5, 4.0, 10, 20 and 40 ug/L, except for ketone compounds at 5.0, 40, 100, 200 and 400ug/L.

2. Internal Standard and Surrogate Standard Mix solution (IS/SS): The working standard of IS/SS solution is prepared by transferring 200 uL each of the IS stock solution (Cat. No. 30241) and SS stock solution (Cat. No. 30240) into a 4 mL vial with 3600 uL of methanol to make a 125 ug/mL solution. This solution once prepared is stored in the Standard Adding Module of either 4551A or 4552 autosamplers. 1uL of the solution is added to all Calibration Standards, ICV, blanks, LCS and samples. At a 25 mL purge volume, this yields a concentration of 5 ug/L.

3. The working standard for the calibration standards is prepared by diluting the 5 mL analysis calibration standard 5 times to a concentration of 20 ug/mL. Additional ketone standards are added at this step to result in a concentration of 100 ug/mL for the ketone compounds.

4. The 5 level initial calibration standards are prepared by adding 1, 8, 20, 40 and 80 uL of the calibration standard, 1, 8, 20, 40 and 80 uL of the surrogate standard and 10 uL of the internal standard to each 40 mL DI water. 25 mL of each solution are used for calibration analysis. Initial calibration criteria are the same as the 5 mL analysis.

5. A second source Initial Calibration Verification (ICV) is performed after the completion of the multi-level calibration, at 10ug/L. The calculated value of the analytes in the ICV should be 70 – 130% of the expected value (7.0-13.0 ng/uL). DoD limits are 80-120%.

6. The continuing calibration standard is prepared by adding 20 uL of the calibration standard, 20 uL of the surrogate standard and 10 uL of the internal standard to a 40 mL DI water. 25 mL of this solution is used for analysis. The frequency and criteria of continuing calibration are the same as the 5 mL analysis.

7. Method blank is prepared by adding 1uL of the IS/SS standard to a 25 mL DI water. The frequency and criteria of method blank are the same as 5 mL analysis. No target compound can be detected above one half of the required reporting limits, except for Methylene Chloride which must be less than 2 ug/L.

8. LCS is prepared by adding 20 uL of the calibration standard to a 40 mL DI water. 25 mL of this solution is used for analysis. The frequency of LCS is the same as 5 mL analysis. Recovery criteria are based on in-house limits and can be found in LIMS.

9. Samples are analyzed after all calibration and QC samples have been analyzed and passed their criteria. Each sample is spiked with 1uL of the IS/SS standard by the autosampler. 25 mL of each spiked sample is used for analysis. The criteria for sample analysis are the same as the 5 mL analysis.

10. MS/MSD samples are spiked with 20 uL of the calibration standard. 25 mL of each spiked sample is used for analysis and is spiked with 1uL of the IS/SS standard by the autosampler. The frequency and criteria of MS/MSD samples are the same as the 5 mL analysis. However, since the whole sample vial is spiked and used for each analysis, MS/MSD for 25 mL analysis can only be performed when there are three or more sample vials available for the designated sample.

Attachments:

Table 1: List of Abbreviations
Table 2: Working Standard / LCS Detail.
Table 3: Suggested minimum RFs (Table 4 from Published Method).
Figure 1: LIMS standard/spike Logbook, Main page**.**
Figure 2: LIMS standard/spike Logbook, Analyte page.
Figure 3: Instrument Run Logbook.
Attachment 1: SW8260 Target Analyte List
Attachment 2: BFB Tune Chromatogram and Mass Listing.
Attachment 3: BFB Tune Mass Spectrum and Ion Abundance Criteria.
Attachment 4: Chromatograph and Quantitation Report of 50ug/L Standard.
Attachment 5: DoD Specific QC Requirements: Table F-4.
Attachment 6: DoD Specific QC Control Limits, Tables G-4 and G-5.
Attachment 7: Additional QA/QC Requirements for MA_DEP
Attachment 8: Corrective Action and Documentation Examples.

Table 1
List of Abbreviations

BFB	Bromofluorobenzene
DoD	Department of Defense (includes Army, Navy, Air Force)
LCS	Lab control sample
LIMS	Laboratory Information Management System
LOD	Limit of Detection
LOQ	Limit of Quantitation
MB	Method Blank
MDL	Method detection limit
MQL	Method quantitation limit
ME	Marginal Exceedence
MS	Matrix spike
MSD	Matrix spike duplicate
QSM	Quality Systems Manual for DoD work
RL	Reporting Limit (occasionally referred to as PQL or Practical Quantitation Limit {in the LIMS}, or MRL or Method Reporting Limit)

Table 2

Working Standard / LCS Detail

Method SW8260

Aqueous uL spike amounts for 40mL Vials											
	QC				Sample	ICAL (Concentration)					
	CCV	Blank	LCS	MS/MSD		5	20	50	100	200	1
GAS	20	-	20	20	-	2	8	20	40	80	0.4
STD	20	-	20	20	-	2	8	20	40	80	0.4
APPIX	20	-	20	20	-	2	8	20	40	80	0.4
SS*	20	20	20	20	20	2	8	20	40	80	0.4
IS*	20	20	20	20	20	20	20	20	20	20	20

Soil uL spike amounts for 5mL H_2O										
	QC				Sample	ICAL (Concentration)				
	CCV	Blank	LCS	MS/MSD		5	20	50	100	200
GAS	2.5	-	2.5	2.5	-	0.25	1	2.5	5	10
STD	2.5	-	2.5	2.5	-	0.25	1	2.5	5	10
APPIX	2.5	-	2.5	2.5	-	0.25	1	2.5	5	10
SS*	2.5	2.5	2.5	2.5	2.5	0.25	1	2.5	5	10
IS*	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5

Blank and LCS vials should contain approx. 5g VOC-free soil preserved in 5mL D.I. Water

*These may be machined spiked for samples, CCV and some project ICALS

Table 3
Suggested minimum RFs
(Table 4 from Published Method)

TABLE 4

RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

[a] The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.

[b] Data provided by EPA Region III laboratory.

Figure 1

LIMS standard/spike Logbook
Main page

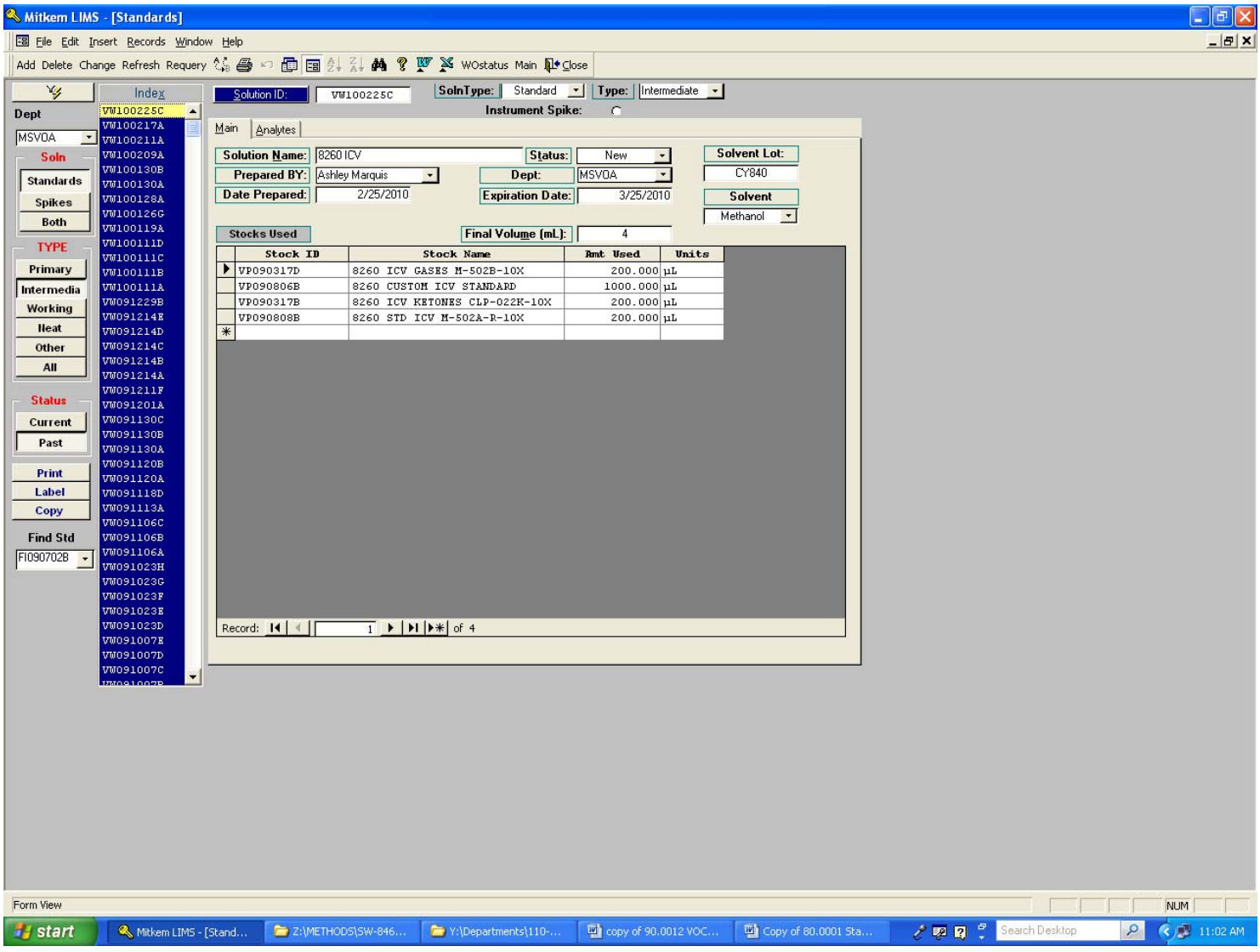


Figure 2

LIMS standard/spike Logbook
Analyte page

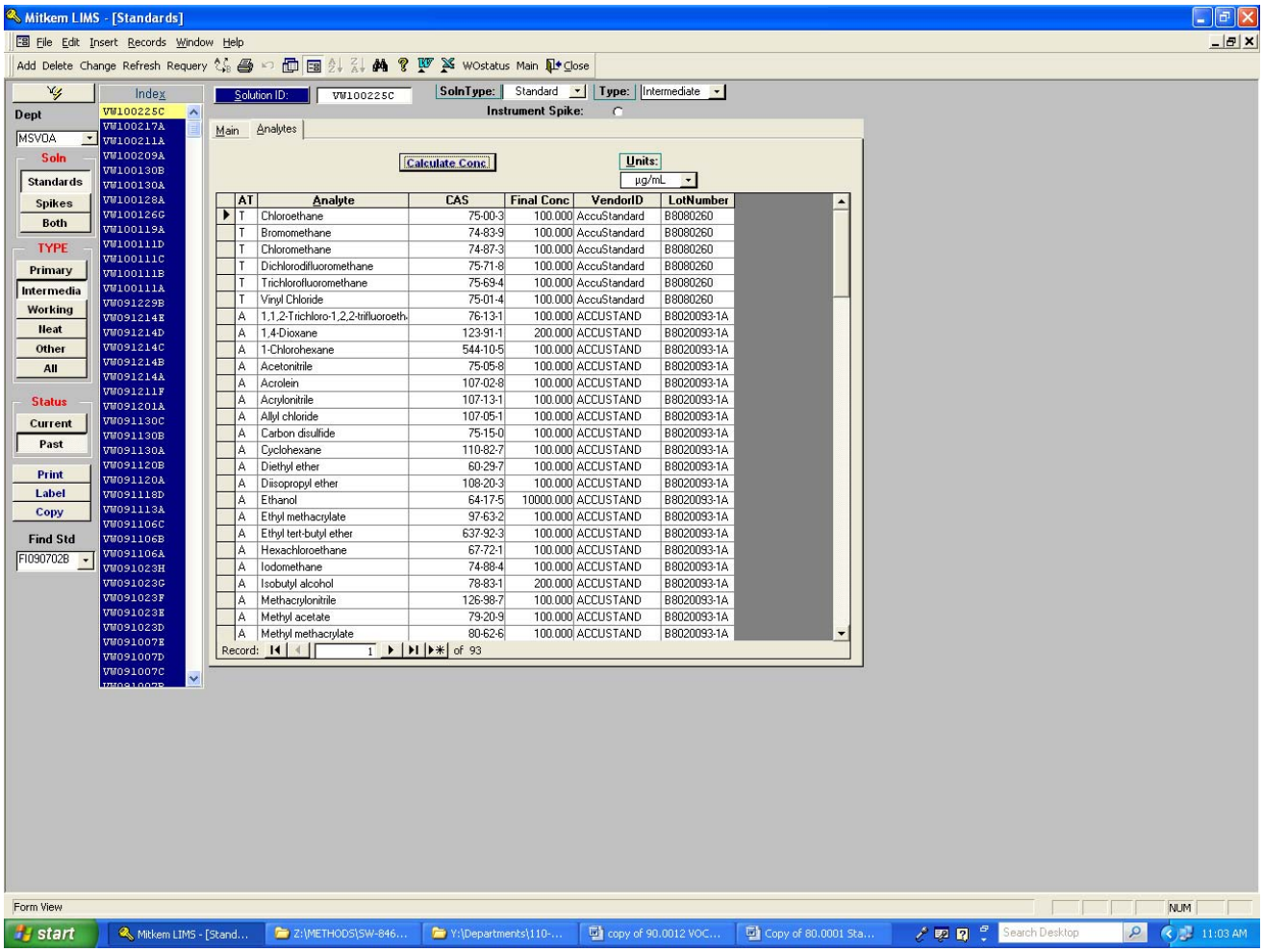


Figure 3

Instrument Run Logbook

METHOD: CAL ID: ANALYST:

INITIAL CAL: IS/SS ID: ARCHIVE:

COMMENTS: ICV ID:

AS #	FILE	LAB ID	CLIENT ID	SAMPLE SIZE	DIL	COMMENTS	IS	SS	pH

Attachment 1
Target Analyte List for 8260

SW8260 Aqueous Target list in ug/L (ppb)

GenericMDLPQL_qry 9/9/2011

AT	Analyte	CAS	MDL	~LOD	PQL
A	1,1,1,2-Tetrachloroethane	630-20-6	0.41	0.5	5
A	1,1,1-Trichloroethane	71-55-6	0.5	0.5	5
A	1,1,2,2-Tetrachloroethane	79-34-5	0.42	0.5	5
A	1,1,2-Trichloroethane	79-00-5	0.38	1	5
A	1,1-Dichloroethane	75-34-3	0.25	0.5	5
A	1,1-Dichloroethene	75-35-4	0.39	0.5	5
A	1,1-Dichloropropene	563-58-6	0.5	0.5	5
A	1,2,3-Trichlorobenzene	87-61-6	0.33	0.5	5
A	1,2,3-Trichloropropane	96-18-4	0.82	1	5
A	1,2,4-Trichlorobenzene	120-82-1	0.26	0.5	5
A	1,2,4-Trimethylbenzene	95-63-6	0.4	0.5	5
A	1,2-Dibromo-3-chloropropane	96-12-8	0.75	1	5
A	1,2-Dibromoethane	106-93-4	0.5	0.5	5
A	1,2-Dichlorobenzene	95-50-1	0.33	0.5	5
A	1,2-Dichloroethane	107-06-2	0.41	0.5	5
A	1,2-Dichloropropane	78-87-5	0.61	1	5
A	1,3,5-Trimethylbenzene	108-67-8	0.45	0.5	5
A	1,3-Dichlorobenzene	541-73-1	0.29	0.5	5
A	1,3-Dichloropropane	142-28-9	0.22	0.5	5
A	1,4-Dichlorobenzene	106-46-7	0.4	0.5	5
A	2,2-Dichloropropane	594-20-7	0.3	0.5	5
A	2-Butanone	78-93-3	2.1	2.5	5
A	2-Chlorotoluene	95-49-8	0.54	1	5
A	2-Hexanone	591-78-6	1.7	2.5	5
A	4-Chlorotoluene	106-43-4	0.45	0.5	5
A	4-Isopropyltoluene	99-87-6	0.46	0.5	5
A	4-Methyl-2-pentanone	108-10-1	0.82	1	5
A	Acetone	67-64-1	2.2	2.5	5
A	Benzene	71-43-2	0.33	0.5	5
A	Bromobenzene	108-86-1	0.36	0.5	5
A	Bromochloromethane	74-97-5	0.43	0.5	5
A	Bromodichloromethane	75-27-4	0.26	0.5	5
A	Bromoform	75-25-2	0.77	1	5
A	Bromomethane	74-83-9	0.8	1	5
A	Carbon disulfide	75-15-0	0.34	0.5	5
A	Carbon tetrachloride	56-23-5	0.54	1	5
A	Chlorobenzene	108-90-7	0.26	0.5	5
A	Chloroethane	75-00-3	0.48	0.5	5
A	Chloroform	67-66-3	0.33	0.5	5
A	Chloromethane	74-87-3	0.26	0.5	5
A	cis-1,2-Dichloroethene	156-59-2	0.48	0.5	5
A	cis-1,3-Dichloropropene	10061-01-5	0.45	0.5	5
A	Dibromochloromethane	124-48-1	0.57	1	5
A	Dibromomethane	74-95-3	0.49	0.5	5
A	Dichlorodifluoromethane	75-71-8	0.66	1	5
A	Ethylbenzene	100-41-4	0.35	0.5	5
A	Hexachlorobutadiene	87-68-3	0.41	0.5	5

AT	Analyte	CAS	MDL	~LOD	PQL
A	Iodomethane	74-88-4	0.63	1	5
A	Isopropylbenzene	98-82-8	0.38	0.5	5
A	m,p-Xylene	1330-20-7	0.77	1	5
A	Methyl tert-butyl ether	1634-04-4	0.24	0.5	5
A	Methylene chloride	75-09-2	0.41	0.5	5
A	n-Butylbenzene	104-51-8	0.33	0.5	5
A	n-Propylbenzene	103-65-1	0.64	1	5
A	Naphthalene	91-20-3	0.8	1	5
A	o-Xylene	95-47-6	0.36	0.5	5
A	sec-Butylbenzene	135-98-8	0.28	0.5	5
A	Styrene	100-42-5	0.5	0.5	5
A	tert-Butylbenzene	98-06-6	0.37	0.5	5
A	Tetrachloroethene	127-18-4	0.65	1	5
A	Toluene	108-88-3	0.32	0.5	5
A	trans-1,2-Dichloroethene	156-60-5	0.65	1	5
A	trans-1,3-Dichloropropene	10061-02-6	0.48	0.5	5
A	Trichloroethene	79-01-6	0.36	0.5	5
A	Trichlorofluoromethane	75-69-4	0.54	1	5
A	Vinyl acetate	108-05-4	0.35	0.5	5
A	Vinyl chloride	75-01-4	0.5	0.5	5
M	Xylene (Total)	1330-20-7	0.36	1	5
S	1,2-Dichloroethane-d4	17060-07-0	1		5
S	Bromofluorobenzene	460-00-4	0.69		5
S	Dibromofluoromethane	1868-53-7	0.83		5
S	Toluene-d8	2037-26-5	0.81		5
X	1,1,2-Trichloro-1,2,2-trifluoroethan	76-13-1	0.82	1	5
X	1,2-Dichloroethene, Total	540-59-0	0.65		5
X	1,2-Dichlorotetrafluoroethane	76-14-2	0.68	1	5
X	1,3,5-Trichlorobenzene	108-70-3	0.81	1	5
X	1,4-Dioxane	123-91-1	34	50	100
X	2,3,6-Trichlorotoluene	2077-46-5	0.4		1
X	2,3/3,4-Dichlorotoluene	23/34-29797408	0.83		1
X	2,4,5-Trichlorotoluene	6639-30-1	0.33		1
X	2,4-Dichlorobenzotrifluoride	320-60-5	0.39		1
X	2,4/2,5-Dichlorotoluene	24/25-29797408	0.93		1
X	2,6-Dichlorotoluene	118-69-4	0.58		1
X	2-Chloro-1,3-butadiene	126-99-8	0.51	1	5
X	2-Chlorobenzotrifluoride	88-16-4	0.48		1
X	2-Chloroethyl vinyl ether	110-75-8	0.24	0.5	5
X	3,4-Dichlorobenzotrifluoride	328-84-7	0.39		1
X	3-Chlorobenzotrifluoride	98-15-7	0.72		1
X	3-Chlorotoluene	108-41-8	0.45		1
X	4-Chlorobenzotrifluoride	98-56-6	0.38		1
X	Acetonitrile	75-05-8	3.5	5	50
X	Acrolein	107-02-8	3.8	5	25
X	Acrylonitrile	107-13-1	2.1	2.5	5
X	Allyl chloride	107-05-1	0.45	0.5	5

AT	Analyte	CAS	MDL	~LOD	PQL
X	Cyclohexane	110-82-7	0.71	1	5
X	Diethyl ether	60-29-7	0.25	0.5	5
X	Diisopropyl ether	108-20-3	0.2	0.5	5
X	Ethanol	64-17-5	240	250	500
X	Ethyl methacrylate	97-63-2	0.77	1	5
X	Ethyl tert-butyl ether	637-92-3	0.42	0.5	5
X	Freon-113	76-13-1	0.82		5
X	Hexachloroethane	67-72-1	0.33	0.5	5
X	Isobutyl alcohol	78-83-1	13	20	100
X	Methacrylonitrile	126-98-7	3.5	5	10
X	Methyl acetate	79-20-9	0.29	1	5
X	Methyl methacrylate	80-62-6	1.5	2.5	5
X	Methylcyclohexane	108-87-2	0.76	1	5
X	Propionitrile	107-12-0	4.7	5	50
X	tert-Amyl Methyl ether	994-05-8	0.18	0.5	5
X	tert-Butyl Alcohol	75-65-0	3.2	5	10
X	Tetrahydrofuran	109-99-9	2.1	5	10
X	trans-1,4-Dichloro-2-butene	110-57-6	1.5	2.5	5
X	Xylenes (Total)	1330-20-7	0.36	1	5

SW8260 Soil Target list in ug/kg (ppb)

GenericMDLPQL_qry 9/19/2011

AT	Analyte	CAS	MDL	~LOD	PQL
A	1,1,1,2-Tetrachloroethane	630-20-6	0.77	2	5
A	1,1,1-Trichloroethane	71-55-6	0.53	2	5
A	1,1,2,2-Tetrachloroethane	79-34-5	0.68	2	5
A	1,1,2-Trichloroethane	79-00-5	0.48	2	5
A	1,1-Dichloroethane	75-34-3	0.67	2	5
A	1,1-Dichloroethene	75-35-4	0.95	2	5
A	1,1-Dichloropropene	563-58-6	0.81	2	5
A	1,2,3-Trichlorobenzene	87-61-6	0.64	2	5
A	1,2,3-Trichloropropane	96-18-4	0.87	2	5
A	1,2,4-Trichlorobenzene	120-82-1	0.63	2	5
A	1,2,4-Trimethylbenzene	95-63-6	0.57	2	5
A	1,2-Dibromo-3-chloropropane	96-12-8	1.3	2	5
A	1,2-Dibromoethane	106-93-4	0.74	2	5
A	1,2-Dichlorobenzene	95-50-1	0.62	2	5
A	1,2-Dichloroethane	107-06-2	0.54	2	5
A	1,2-Dichloropropane	78-87-5	0.69	2	5
A	1,3,5-Trimethylbenzene	108-67-8	0.61	2	5
A	1,3-Dichlorobenzene	541-73-1	0.7	2	5
A	1,3-Dichloropropane	142-28-9	0.87	2	5
A	1,4-Dichlorobenzene	106-46-7	0.8	2	5
A	2,2-Dichloropropane	594-20-7	0.29	2	5
A	2-Butanone	78-93-3	2	4	5
A	2-Chlorotoluene	95-49-8	0.74	2	5
A	2-Hexanone	591-78-6	0.83	4	5
A	4-Chlorotoluene	106-43-4	0.84	2	5
A	4-Isopropyltoluene	99-87-6	0.71	2	5
A	4-Methyl-2-pentanone	108-10-1	0.73	4	5
A	Acetone	67-64-1	1.6	4	5
A	Benzene	71-43-2	0.61	2	5
A	Bromobenzene	108-86-1	0.58	2	5
A	Bromochloromethane	74-97-5	0.76	2	5
A	Bromodichloromethane	75-27-4	0.97	2	5
A	Bromoform	75-25-2	2	2	5
A	Bromomethane	74-83-9	1.1	2	5
A	Carbon disulfide	75-15-0	0.3	2	5
A	Carbon tetrachloride	56-23-5	0.33	2	5
A	Chlorobenzene	108-90-7	0.51	2	5
A	Chloroethane	75-00-3	1	2	5
A	Chloroform	67-66-3	0.64	2	5
A	Chloromethane	74-87-3	0.8	2	5
A	cis-1,2-Dichloroethene	156-59-2	0.75	2	5
A	cis-1,3-Dichloropropene	10061-01-5	0.67	2	5
A	Dibromochloromethane	124-48-1	0.65	2	5
A	Dibromomethane	74-95-3	0.58	2	5
A	Dichlorodifluoromethane	75-71-8	0.98	2	5
A	Ethylbenzene	100-41-4	0.5	2	5
A	Hexachlorobutadiene	87-68-3	0.62	2	5

AT	Analyte	CAS	MDL	~LOD	PQL
A	Iodomethane	74-88-4	0.69	2	5
A	Isopropylbenzene	98-82-8	0.58	2	5
A	m,p-Xylene	1330-20-7	1.6	4	5
A	Methyl tert-butyl ether	1634-04-4	0.61	2	5
A	Methylene chloride	75-09-2	1.3	2	5
A	n-Butylbenzene	104-51-8	0.67	2	5
A	n-Propylbenzene	103-65-1	0.44	2	5
A	Naphthalene	91-20-3	0.78	2	5
A	o-Xylene	95-47-6	0.47	2	5
A	sec-Butylbenzene	135-98-8	0.62	2	5
A	Styrene	100-42-5	0.52	2	5
A	tert-Butylbenzene	98-06-6	0.52	2	5
A	Tetrachloroethene	127-18-4	0.62	2	5
A	Toluene	108-88-3	0.47	2	5
A	trans-1,2-Dichloroethene	156-60-5	0.53	2	5
A	trans-1,3-Dichloropropene	10061-02-6	0.68	2	5
A	Trichloroethene	79-01-6	0.62	2	5
A	Trichlorofluoromethane	75-69-4	0.42	2	5
A	Vinyl acetate	108-05-4	0.37	2	5
A	Vinyl chloride	75-01-4	0.63	2	5
I	1,4-Dichlorobenzene-d4	3855-82-1	5		5
I	Chlorobenzene-d5	3114-55-4	5		5
I	Fluorobenzene	462-06-6	5		5
M	Xylene (Total)	1330-20-7	0.47	2	5
X	1,1,2-Trichloro-1,2,2-trifluoroethan	76-13-1	3	4	5
X	1,2-Dichloroethene, Total	540-59-0	0.75		5
X	1,2-Dichlorotetrafluoroethane	76-14-2	5		5
X	1,3,5-Trichlorobenzene	108-70-3	5		5
X	1,4-Difluorobenzene	540-36-3	5		5
X	1,4-Dioxane	123-91-1	61	100	100
X	1-Chlorohexane	544-10-5	1.9	2	5
X	1-Chloropropane	540-54-5	5		5
X	2,3-Dibromopropene	513-31-5	5		5
X	2,3-Dichloropropene	78-88-6	5		5
X	2-Bromo-1-chloropropane	3017-95-6	5		5
X	2-Chloro-1,3-butadiene	126-99-8	5		5
X	2-Chloroethyl vinyl ether	110-75-8	1	4	5
X	2-Chloropropane	75-29-6	5		5
X	2-Hexanone-d5	4840-82-8	5		5
X	Acetonitrile	75-05-8	10	40	50
X	Acrolein	107-02-8	5.1	20	25
X	Acrylonitrile	107-13-1	1.2	2	5
X	Alkylbenzenes, Total		5		5
X	Allyl chloride	107-05-1	5		5
X	bis(2-Chloroethyl)ether	111-44-4	5		5
X	Cyclohexane	110-82-7	1.7	2	5
X	Diethyl ether	60-29-7	1.3	2	5

AT	Analyte	CAS	MDL	~LOD	PQL
X	Diisopropyl ether	108-20-3	0.61	2	5
X	Ethanol	64-17-5	260	400	500
X	Ethyl methacrylate	97-63-2	5		5
X	Ethyl tert-butyl ether	637-92-3	0.85	2	5
X	Freon-113	76-13-1	3		5
X	Hexachloroethane	67-72-1	5		5
X	Isobutyl alcohol	78-83-1	100		100
X	Isopropyl alcohol	67-63-0	5		5
X	Methacrylonitrile	126-98-7	10		10
X	Methyl acetate	79-20-9	1.4	2	5
X	Methyl methacrylate	80-62-6	5		5
X	Methylcyclohexane	108-87-2	1.8	2	5
X	Pentachloroethane	76-01-7	5		5
X	Pentafluorobenzene	363-72-4	5		5
X	Propionitrile	107-12-0	50		50
X	tert-Amyl Methyl ether	994-05-8	1.1	2	5
X	tert-Butyl Alcohol	75-65-0	5	8	10
X	Tetrahydrofuran	109-99-9	4.6	8	10
X	trans-1,4-Dichloro-2-butene	110-57-6	0.98	2	5
X	Vinyl bromide	593-60-2	5		5
X	Xylenes (Total)	1330-20-7	0.47	2	5

Attachment 2
BFB Tune Chromatogram

Data File: \\avogadro\organics\V6.i\120814.B\V6I9000.D

Date : 14-AUG-2012 08:36

Client ID: BFB6P

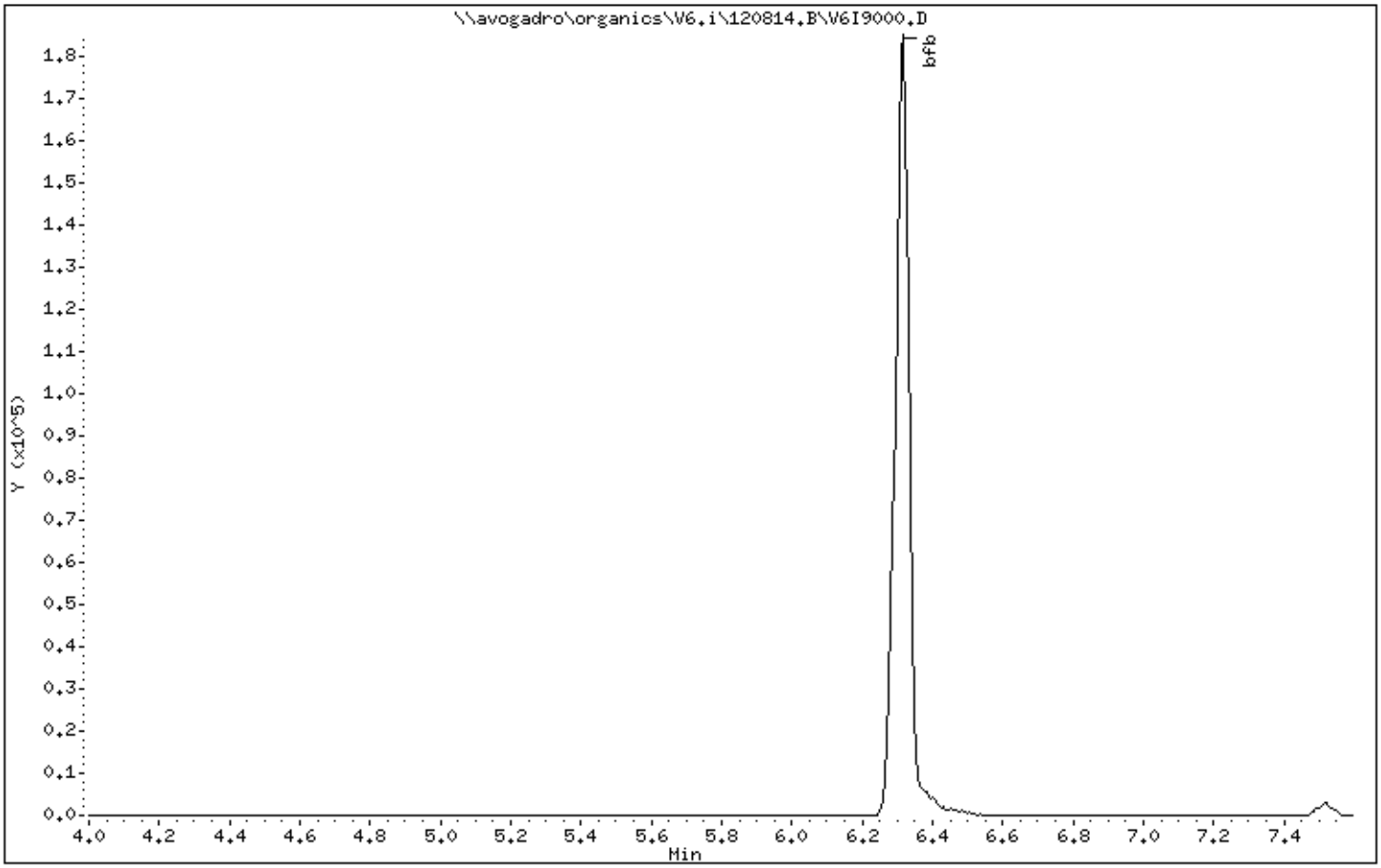
Instrument: V6.i

Sample Info: 5ML,BFB6P,BFB6P

Operator: AM SRC: AM

Column phase: DB-624

Column diameter: 0.25



Attachment 3
BFB Tune Mass Spectrum and Ion Abundance Criteria

Data File: \\avogadro\organics\V6.i\120814.B\V6I9000.D

Date : 14-AUG-2012 08:36

Client ID: BFB6P

Instrument: V6.i

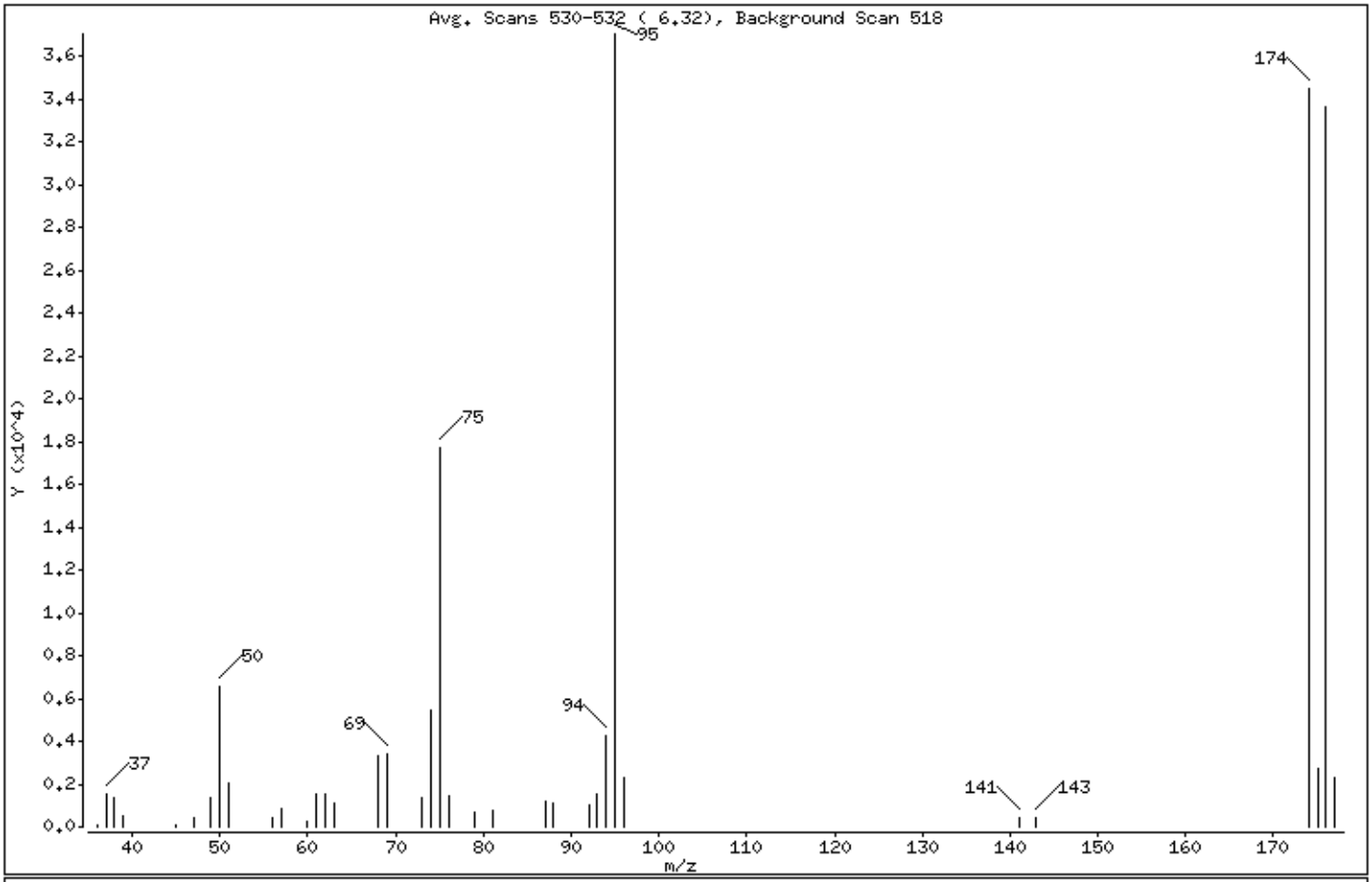
Sample Info: 5ML,BFB6P,BFB6P

Operator: AM SRC: AM

Column phase: DB-624

Column diameter: 0.25

1 bfb



m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
95	Base Peak, 100% relative abundance	100.00
50	15.00 - 40.00% of mass 95	17.72
75	30.00 - 60.00% of mass 95	47.72
96	5.00 - 9.00% of mass 95	6.30
173	Less than 2.00% of mass 174	0.00 (0.00)
174	50.00 - 100.00% of mass 95	93.18
175	5.00 - 9.00% of mass 174	7.27 (7.80)
176	95.00 - 101.00% of mass 174	90.74 (97.38)
177	5.00 - 9.00% of mass 176	6.14 (6.77)

Date : 14-AUG-2012 08:36

Client ID: BFB6P Instrument: V6.i

Sample Info: 5ML,BFB6P,BFB6P

Operator: AM SRC: AM

Column phase: DB-624 Column diameter: 0.25

Data File: V6I9000.D
Spectrum: Avg. Scans 530-532 (6.32), Background Scan 518
Location of Maximum: 95.00
Number of points: 36

m/z	Y	m/z	Y	m/z	Y	m/z	Y
36.00	107	57.00	865	76.00	1427	141.00	466
37.00	1564	60.00	224	79.00	699	143.00	416
38.00	1338	61.00	1561	81.00	730	174.00	34520
39.00	546	62.00	1537	87.00	1232	175.00	2693
45.00	102	63.00	1116	88.00	1101	176.00	33616
47.00	458	68.00	3283	92.00	1058	177.00	2276
49.00	1362	69.00	3389	93.00	1494		
50.00	6564	73.00	1404	94.00	4275		
51.00	2075	74.00	5444	95.00	37048		
56.00	460	75.00	17680	96.00	2333		

Attachment 4
Chromatogram and Quantitation Report of 50 µg/L Standard

Data File: \\avogadro\organics\V6.i\120814.B\V6I9001.D
Report Date: 15-Aug-2012 10:04

Spectrum Analytical, Inc. RI Division

Method 8260 Water and Medium Soil

Data file : \\avogadro\organics\V6.i\120814.B\V6I9001.D
Lab Smp Id: VSTD0506P Client Smp ID: VSTD0506P
Inj Date : 14-AUG-2012 09:01
Operator : AM SRC: AM Inst ID: V6.i
Smp Info : 5ML,VSTD0506P,VSTD0506P
Misc Info :
Comment   :
Method   : \\avogadro\organics\V6.i\120814.B\v68260Gadd-6lvl.m
Meth Date : 15-Aug-2012 10:02 adatta Quant Type: ISTD
Cal Date : 08-AUG-2012 14:11 Cal File: V6I8869.D
Als bottle: 2 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: HP RTE Compound Sublist: all.sub
Target Version: 4.14

Concentration Formula: Amt * DF * Uf * 5/Vo * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	ng unit correction factor
Vo	5.000	Sample Volume purged (mL)
Cpnd Variable		Local Compound Variable

	Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ug/L)	ON-COL (ug/L)
1	Dichlorodifluoromethane	85	1.602	1.601	(0.312)	71886	50.0000	44
2	Freon114	85	1.708	1.708	(0.333)	106862	50.0000	43
3	Chloromethane	50	1.767	1.779	(0.345)	112836	50.0000	42
4	Vinyl Chloride	62	1.862	1.861	(0.363)	89329	50.0000	47
5	Bromomethane	94	2.134	2.146	(0.416)	67212	50.0000	52
6	Chloroethane	64	2.229	2.228	(0.435)	51969	50.0000	48
7	Trichlorofluoromethane	101	2.406	2.406	(0.469)	180301	50.0000	54
126	Ethanol	46	2.536	2.536	(0.495)	18971	5000.00	7500(A)
8	Ether	59	2.607	2.607	(0.509)	62866	50.0000	47
9	Acrolein	56	2.726	2.725	(0.532)	55558	250.000	490(A)
10	1,1-Dichloroethene	96	2.809	2.808	(0.548)	104198	50.0000	47
11	1,1,2-Trichloro-1,2,2-Trifluo	101	2.809	2.808	(0.548)	105506	50.0000	51
12	Acetone	58	2.832	2.844	(0.552)	11234	50.0000	51
13	Iodomethane	142	2.951	2.962	(0.575)	202870	50.0000	46
14	Carbon Disulfide	76	2.986	2.986	(0.582)	248991	50.0000	34
15	Acetonitrile	41	3.069	3.068	(0.599)	231325	500.000	410(A)
16	Allyl Chloride	39	3.069	3.068	(0.599)	126886	50.0000	50
17	Methyl Acetate	43	3.081	3.080	(0.601)	81986	50.0000	39
18	Methylene Chloride	84	3.164	3.175	(0.617)	96208	50.0000	36
19	tert-Butanol	59	3.235	3.246	(0.631)	23351	100.000	100
20	Acrylonitrile	53	3.365	3.364	(0.656)	34988	50.0000	40
21	trans-1,2-Dichloroethene	96	3.377	3.376	(0.659)	93028	50.0000	48
22	Methyl tert-butyl ether	73	3.365	3.364	(0.656)	272856	50.0000	47

Data File: \\avogadro\organics\V6.i\120814.B\V6I9001.D
Report Date: 15-Aug-2012 10:04

			QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ug/L)	AMOUNTS ON-COL (ug/L)
	23	1,1-Dichloroethane	63	3.708	3.707	(0.723)	163754	50.0000	47
	24	Vinyl acetate	43	3.732	3.731	(0.728)	302765	50.0000	45
	25	Diisopropyl Ether	45	3.732	3.731	(0.728)	291996	50.0000	45
	26	2-Chloro-1,3-Butadiene	53	3.767	3.767	(0.735)	155668	50.0000	49
	27	Ethyl tert-butyl ether	59	4.016	4.015	(0.783)	286816	50.0000	47
	29	2,2-Dichloropropane	77	4.169	4.169	(0.813)	91726	50.0000	55
	28	cis-1,2-Dichloroethene	96	4.157	4.169	(0.811)	96778	50.0000	48
	30	2-Butanone	72	4.169	4.169	(0.813)	11143	50.0000	45
	32	Propionitrile	54	4.228	4.228	(0.825)	130447	500.000	400(A)
	33	Methacrylonitrile	41	4.347	4.346	(0.848)	106713	100.000	85
	34	Bromochloromethane	128	4.359	4.358	(0.850)	56156	50.0000	49
	31	Tetrahydrofuran	72	4.406	4.406	(0.859)	20250	100.000	78
	35	Chloroform	83	4.418	4.417	(0.862)	173470	50.0000	50
$	36	Dibromofluoromethane	113	4.548	4.547	(0.887)	102655	50.0000	53
	37	1,1,1-Trichloroethane	97	4.583	4.583	(0.894)	163222	50.0000	51
	38	Cyclohexane	56	4.631	4.630	(0.903)	155358	50.0000	47
	39	1,1-Dichloropropene	110	4.714	4.713	(0.919)	47959	50.0000	48
	40	Carbon Tetrachloride	117	4.714	4.713	(0.919)	176170	50.0000	54
	41	Isobutyl Alcohol	43	4.773	4.772	(0.931)	83833	1000.00	870(A)
$	42	1,2-Dichloroethane-d4	102	4.844	4.843	(0.945)	21359	50.0000	53
	43	Benzene	78	4.891	4.891	(0.954)	317722	50.0000	47
	44	1,2-Dichloroethane	62	4.903	4.902	(0.956)	150722	50.0000	52
	45	tert-Amyl methyl ether	73	4.962	4.962	(0.968)	255914	50.0000	46
M	50	1,2-Dichloroethene (Total)	96				189806	100.000	(a)
*	46	Fluorobenzene	96	5.128	5.127	(1.000)	316212	50.0000	
	47	Trichloroethene	130	5.447	5.447	(1.062)	114982	50.0000	49
	48	Methylcyclohexane	83	5.625	5.624	(1.097)	128230	50.0000	46
	49	1,2-Dichloropropane	63	5.660	5.660	(1.104)	84592	50.0000	46
	51	Methyl Methacrylate	69	5.731	5.731	(1.118)	66741	50.0000	42
	52	Dibromomethane	93	5.767	5.766	(1.125)	58482	50.0000	46
	53	1,4-Dioxane	88	5.779	5.778	(1.127)	18280	1000.00	960(A)
	54	Bromodichloromethane	83	5.909	5.908	(1.152)	139316	50.0000	51
	55	2-Chloroethyl vinyl ether	63	6.654	6.654	(1.298)	32080	50.0000	50
	56	cis-1,3-Dichloropropene	75	6.323	6.322	(1.233)	151187	50.0000	50
	57	4-Methyl-2-pentanone	43	6.465	6.464	(1.261)	91019	50.0000	41
$	58	Toluene-d8	98	6.595	6.594	(0.814)	332642	50.0000	50
	59	Toluene	91	6.654	6.654	(1.298)	369499	50.0000	46
	60	trans-1,3-Dichloropropene	75	6.879	6.879	(1.341)	147250	50.0000	52
	61	Ethyl Methacrylate	69	6.950	6.950	(1.355)	95703	50.0000	43
	62	1,1,2-Trichloroethane	97	7.068	7.068	(1.378)	80677	50.0000	47
	63	Tetrachloroethene	164	7.210	7.210	(0.890)	94041	50.0000	46
	64	1,3-Dichloropropane	76	7.234	7.234	(0.893)	133228	50.0000	45
	65	2-Hexanone	43	7.305	7.305	(0.902)	69201	50.0000	40
	66	Dibromochloromethane	129	7.483	7.482	(0.924)	132279	50.0000	48
	67	1,2-Dibromoethane	107	7.613	7.612	(0.940)	101148	50.0000	46
	69	1-Chlorohexane	91	8.086	8.086	(0.999)	120525	50.0000	43
*	68	Chlorobenzene-d5	117	8.098	8.097	(1.000)	305368	50.0000	
	70	Chlorobenzene	112	8.121	8.121	(1.003)	273792	50.0000	46
	71	1,1,1,2-Tetrachloroethane	131	8.216	8.216	(1.015)	126908	50.0000	48
	72	Ethylbenzene	106	8.240	8.239	(1.018)	142508	50.0000	46
	73	m,p-Xylene	106	8.370	8.370	(1.034)	349682	100.000	91
	74	o-Xylene	106	8.808	8.807	(1.088)	172338	50.0000	46
	75	Styrene	104	8.831	8.831	(1.091)	297759	50.0000	45
	76	Bromoform	173	9.044	9.044	(1.117)	95592	50.0000	47

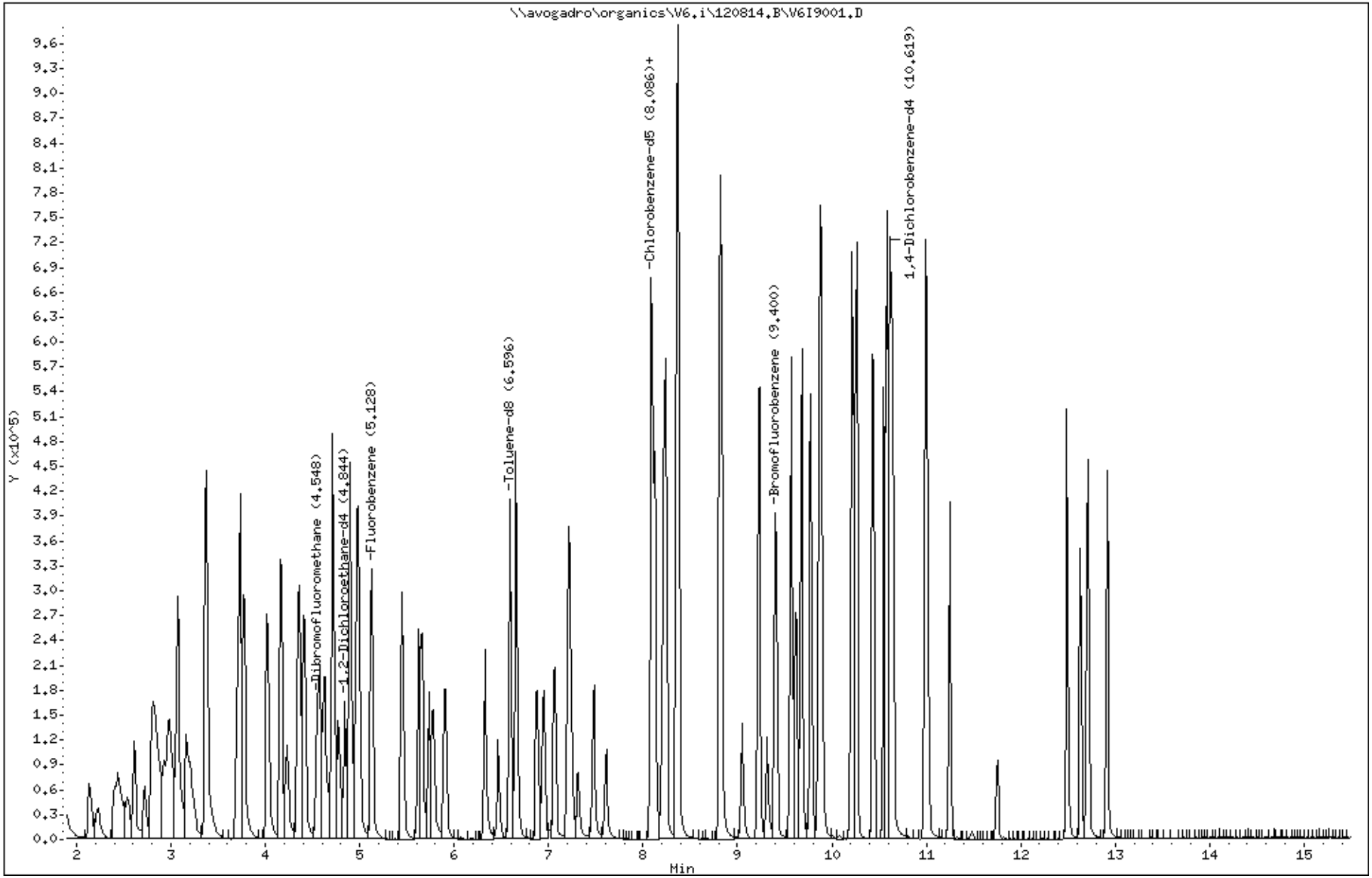
Data File: \\avogadro\organics\V6.i\120814.B\V6I9001.D
Report Date: 15-Aug-2012 10:04

			QUANT SIG					AMOUNTS	
		Compounds	MASS	RT	EXP RT	REL RT	RESPONSE	CAL-AMT (ug/L)	ON-COL (ug/L)
	77	Isopropylbenzene	105	9.234	9.233	(1.140)	466841	50.0000	47
	78	trans-1,4-Dichloro-2-butene	75	9.317	9.316	(1.150)	34357	50.0000	43
$	79	Bromofluorobenzene	95	9.399	9.399	(1.161)	157602	50.0000	51
	80	1,1,2,2-Tetrachloroethane	77	9.565	9.565	(0.901)	213621	50.0000	42
	81	Bromobenzene	156	9.577	9.577	(0.902)	137548	50.0000	44
	82	1,2,3-Trichloropropane	75	9.612	9.612	(0.905)	142254	50.0000	39
	83	n-Propylbenzene	120	9.683	9.683	(0.912)	129172	50.0000	45
	84	2-Chlorotoluene	126	9.778	9.778	(0.921)	125153	50.0000	43
	85	1,3,5-Trimethylbenzene	105	9.873	9.872	(0.930)	406589	50.0000	45
	86	4-Chlorotoluene	126	9.896	9.896	(0.932)	130909	50.0000	44
M	94	Xylene (Total)	106				522020	150.000	(a)
	87	tert-Butylbenzene	119	10.583	10.582	(0.997)	436118	50.0000	45
	88	1,2,4-Trimethylbenzene	105	10.263	10.263	(0.967)	417441	50.0000	45
	89	sec-Butylbenzene	105	10.441	10.428	(0.983)	473298	50.0000	45
	90	1,3-Dichlorobenzene	146	10.547	10.547	(0.993)	252605	50.0000	44
	91	4-Isopropyltoluene	119	10.583	10.582	(0.997)	436118	50.0000	45
*	92	1,4-Dichlorobenzene-d4	152	10.618	10.618	(1.000)	192256	50.0000	
	93	1,4-Dichlorobenzene	146	10.642	10.641	(1.002)	269825	50.0000	43
	95	n-Butylbenzene	91	10.985	10.985	(1.035)	353293	50.0000	45
	96	1,2-Dichlorobenzene	146	11.009	11.008	(1.037)	258066	50.0000	45
	97	Hexachloroethane	117	11.245	11.245	(1.059)	90576	50.0000	45
	98	1,2-Dibromo-3-chloropropane	75	11.742	11.742	(1.106)	27935	50.0000	42
	141	1,3,5-Trichlorobenzene	182	12.488	12.487	(2.435)	161696	50.0000	46(A)
	99	1,2,4-Trichlorobenzene	180	12.488	12.487	(1.176)	171974	50.0000	43
	100	Hexachlorobutadiene	225	12.630	12.629	(1.189)	66159	50.0000	46
	101	Naphthalene	128	12.713	12.712	(1.197)	407877	50.0000	39
	102	1,2,3-Trichlorobenzene	180	12.914	12.913	(1.216)	144311	50.0000	41

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).
A - Target compound detected but, quantitated amount
exceeded maximum amount.

Data File: \\avogadro\organics\V6.i\120814.B\V6I9001.D
Date : 14-AUG-2012 09:01
Client ID: VSTD0506P
Instrument: V6.i
Sample Info: 5ML,VSTD0506P,VSTD0506P
Purge Volume: 5.0
Operator: AM SRC: AM
Column phase: DB-624
Column diameter: 0.25



Attachment 5
DoD-Specific QC Requirements
QSM Table F-4

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050. 2. RSD for RFs for CCCs: VOCs and SVOCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression–coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Attachment 6
DoD Specific QC Control Limits
QSM Tables G-4 and G-5

DoD strongly believes that it is important for laboratories to maintain their own in-house LCS limits. These in-house limits must be consistent with (i.e., within) the DoD limits (project-specific, if available; otherwise the following LCS-CLs). The laboratory in-house limits shall be calculated from the laboratory's historical LCS data in accordance with a documented procedure (e.g., SOP) that is consistent with good laboratory practice. That document must describe the process for establishing and maintaining LCS limits and the use of control charts.

The laboratory in-house limits are to be used for several purposes:

- Laboratories are expected to utilize their in-house limits as part of their quality control system, and to evaluate trends and monitor and improve performance.
- When a laboratory's in-house limits are outside the DoD control limits (upper and/or lower), they must report their in-house limits in the laboratory report (see Appendix E) even if the LCS associated with the batch fell within the DoD limits. Using this information, DoD will be able to determine how laboratory performance affects the quality of the environmental data.
- DoD may review the laboratory in-house limits and associated trends, as reflected in control charts, to determine whether the laboratory's overall performance is acceptable. If deemed unacceptable, this can allow DoD to decide not to use the laboratory again until substantial improvement has occurred.

Table G-4. LCS Control Limits for Volatile Organic Compounds SW-846 Method 8260 Water Matrix[2]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
1,1,1,2-Tetrachloroethane	105	8	80	130	75	135
1,1,1-Trichloroethane	100	11	65	130	55	145
1,1,2,2-Tetrachloroethane	96	11	65	130	55	140
1,1,2-Trichloroethane	100	8	75	125	65	135
1,1-Dichloroethane	101	11	70	135	60	145
1,1-Dichloroethene	99	10	70	130	55	140
1,1-Dichloropropene	102	10	75	130	65	140
1,2,3-Trichlorobenzene	99	14	55	140	45	155
1,2,3-Trichloropropane	98	9	75	125	65	130
1,2,4-Trichlorobenzene	100	11	65	135	55	145
1,2,4-Trimethylbenzene	103	10	75	130	65	140
1,2-Dibromo-3-chloropropane	91	14	50	130	35	145
1,2-Dibromoethane	100	7	80	120	75	125
1,2-Dichlorobenzene	96	9	70	120	60	130
1,2-Dichloroethane	100	10	70	130	60	140
1,2-Dichloropropane	100	8	75	125	65	135
1,3,5-Trimethylbenzene	102	10	75	130	65	140
1,3-Dichlorobenzene	100	8	75	125	65	130
1,3-Dichloropropane	100	9	75	125	65	135
1,4-Dichlorobenzene	99	8	75	125	65	130
2,2-Dichloropropane	103	11	70	135	60	150
2-Butanone	91	20	30	150	10	170

[2] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Total Xylene. Xylene may be reported on a project-specific basis as a total number; however, for the purposes of the DoD QSM, it will be analyzed and reported as m,p-Xylene and o-Xylene. Additional limits for poor performing compounds can be found in section G.5 and for surrogate compounds in section G.6.

Table G-4. LCS Control Limits for Volatile Organic Compounds SW-846 Method 8260 Water Matrix[2] (continued)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
2-Chlorotoluene	100	9	75	125	65	135
2-Hexanone	92	12	55	130	45	140
4-Chlorotoluene	101	9	75	130	65	135
4-Methyl-2-pentanone	96	13	60	135	45	145
Acetone	91	17	40	140	20	160
Benzene	102	7	80	120	75	130
Bromobenzene	100	8	75	125	70	130
Bromochloromethane	97	11	65	130	55	140
Bromodichloromethane	98	8	75	120	70	130
Bromoform	99	10	70	130	60	140
Bromomethane	88	19	30	145	10	165
Carbon disulfide	100	21	35	160	15	185
Carbon tetrachloride	102	12	65	140	55	150
Chlorobenzene	102	7	80	120	75	130
Chlorodibromomethane	96	13	60	135	45	145
Chloroethane	99	12	60	135	50	145
Chloroform	100	12	65	135	50	150
Chloromethane	83	15	40	125	25	140
cis-1,2-Dichloroethene	99	9	70	125	60	135
cis-1,3-Dichloropropene	100	10	70	130	60	140
Dibromomethane	101	8	75	125	65	135
Dichlorodifluoromethane	93	21	30	155	10	175
Ethylbenzene	100	9	75	125	65	135
Hexachlorobutadiene	97	15	50	140	35	160
Isopropylbenzene	101	9	75	125	65	135
m,p-Xylene	102	9	75	130	65	135
Methyl tert-butyl ether	94	10	65	125	55	135
Methylene chloride	96	14	55	140	40	155
Naphthalene	96	14	55	140	40	150
n-Butylbenzene	103	11	70	135	55	150
n-Propylbenzene	101	9	70	130	65	140
o-Xylene	100	7	80	120	75	130
p-Isopropyltoluene	102	10	75	130	65	140
sec-Butylbenzene	100	9	70	125	65	135
Styrene	100	11	65	135	55	145
tert-Butylbenzene	99	10	70	130	60	140
Tetrachloroethene	96	18	45	150	25	165
Toluene	100	7	75	120	70	130
trans-1,2-Dichloroethene	99	13	60	140	45	150
trans-1,3-Dichloropropene	98	15	55	140	40	155
Trichloroethene	99	9	70	125	60	135
Trichlorofluoromethane	103	15	60	145	45	160
Vinyl chloride	99	16	50	145	35	165

Table G-5. LCS Control Limits for Volatile Organic Compounds SW-846 Method 8260 Solid Matrix[3]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
1,1,1,2-Tetrachloroethane	100	9	75	125	65	135
1,1,1-Trichloroethane	101	11	70	135	55	145
1,1,2,2-Tetrachloroethane	93	13	55	130	40	145
1,1,2-Trichloroethane	95	11	60	125	50	140
1,1-Dichloroethane	99	9	75	125	65	135
1,1-Dichloroethene	100	12	65	135	55	150
1,1-Dichloropropene	102	11	70	135	60	145
1,2,3-Trichlorobenzene	97	12	60	135	50	145
1,2,3-Trichloropropane	97	11	65	130	50	140
1,2,4-Trichlorobenzene	98	11	65	130	55	140
1,2,4-Trimethylbenzene	100	12	65	135	55	145
1,2-Dibromo-3-chloropropane	87	16	40	135	25	150
1,2-Dibromoethane	97	9	70	125	60	135
1,2-Dichlorobenzene	97	7	75	120	65	125
1,2-Dichloroethane	104	11	70	135	60	145
1,2-Dichloropropane	95	8	70	120	65	125
1,3,5-Trimethylbenzene	99	11	65	135	55	145
1,3-Dichlorobenzene	98	9	70	125	65	135
1,3-Dichloropropane	100	8	75	125	70	130
1,4-Dichlorobenzene	98	9	70	125	65	135
2,2-Dichloropropane	101	11	65	135	55	145
2-Butanone	94	22	30	160	10	180
2-Chlorotoluene	98	10	70	130	60	140
2-Hexanone	97	16	45	145	30	160
4-Chlorotoluene	100	9	75	125	65	135
4-Methyl-2-pentanone	97	17	45	145	30	165
Acetone	88	23	20	160	10	180
Benzene	99	9	75	125	65	135
Bromobenzene[4]	93	9	65	120	55	130
Bromochloromethane	99	9	70	125	60	135
Bromodichloromethane	100	9	70	130	60	135
Bromoform	96	13	55	135	45	150
Bromomethane	95	21	30	160	10	180
Carbon disulfide	103	19	45	160	30	180
Carbon tetrachloride	100	11	65	135	55	145
Chlorobenzene	99	8	75	125	65	130
Chlorodibromomethane	98	11	65	130	55	140
Chloroethane	98	20	40	155	20	175

[3] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Methyl tert-butyl ether and Total Xylene. Sufficient data to perform statistically significant analyses were not received for MTBE during the LCS study. Xylene may be reported on a project-specific basis as a total number; however, for the purposes of the DoD QSM, it will be analyzed and reported as m,p-Xylene and o-Xylene. Additional limits for poor performing compounds can be found in section G.5 and for surrogate compounds in section G.6.

[4] Provisional limits – outlier analyses during the LCS study resulted in LCS-CLs generated with data from fewer than four laboratories. Limits may be adjusted in the future as additional data become available.

Table G-5. LCS Control Limits for Volatile Organic Compounds SW-846 Method 8260 Solid Matrix[3] (continued)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Chloroform	98	9	70	125	65	135
Chloromethane	90	13	50	130	40	140
cis-1,2-Dichloroethene	96	10	65	125	55	135
cis-1,3-Dichloropropene	99	9	70	125	65	135
Dibromomethane	100	9	75	130	65	135
Dichlorodifluoromethane[4]	85	17	35	135	15	155
Ethylbenzene	101	9	75	125	65	135
Hexachlorobutadiene	98	15	55	140	40	155
Isopropylbenzene	103	9	75	130	70	140
m,p-Xylene	102	8	80	125	70	135
Methylene chloride	97	14	55	140	40	155
Naphthalene	84	14	40	125	25	140
n-Butylbenzene	101	12	65	140	50	150
n-Propylbenzene	99	12	65	135	50	145
o-Xylene	101	8	75	125	70	135
p-Isopropyltoluene	104	10	75	135	65	140
sec-Butylbenzene	97	11	65	130	50	145
Styrene	101	9	75	125	65	135
tert-Butylbenzene	99	11	65	130	55	145
Tetrachloroethene	103	12	65	140	55	150
Toluene	99	9	70	125	60	135
trans-1,2-Dichloroethene	100	11	65	135	55	145
trans-1,3-Dichloropropene	96	10	65	125	55	140
Trichloroethene	101	8	75	125	70	130
Trichlorofluoromethane	106	27	25	185	10	215
Vinyl chloride	92	11	60	125	45	140

Table G-6. LCS Control Limits for Semivolatile Organic Compounds SW-846 Method 8270 Water Matrix[5]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	75.0	9.5	45	105	35	115
Acenaphthene	77.6	10.1	45	110	35	120
Acenaphthylene	78.5	9.4	50	105	40	115
Anthracene	83.0	9.7	55	110	45	120
Benz[a]anthracene	82.7	8.9	55	110	45	120
Benzo[a]pyrene	81.3	9.5	55	110	45	120

[5] A number of sporadic marginal exceedances of the control limits are allowed depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, and N-nitrosopyrrolidine. Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section G.5.

Attachment 7
Additional QA/QC Requirements for MA-DEP

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
GC/MS Tunes with BFB	Inter-laboratory consistency and comparability	(1) Criteria listed in Table 4 of SW-846 Method 8260B (the same criteria must be used for all analyses) (2) Every 12 hours	No	Perform instrument maintenance as necessary; retune instrument	Suspend all analyses until tuning non-compliance is rectified
Initial Calibration	Laboratory Analytical Accuracy	(1) Minimum of 5 standards (2) Low standard must be ≤ Reporting Limit (RL) (3) %RSD should be ≤15 or "r" should be ≥0.99 for all compounds except CCCs which must be ≤30 %RSD or "r" ≥0.99 (4) Must contain all target analytes (5) If regression analysis is used, the curve must not be forced through the origin.	No	Recalibrate as required by method (1) if any of CCC % RSDs >30 or any of CCC "r" <0.99 or (2) if >20% of remaining analytes have % RSDs >30 or "r" <0.99.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds in Environmental Laboratory case narrative. If the average response factor or linear regression are not used for analyte quantitation (e.g., use of a quadratic equation), this must be noted in the Environmental Laboratory case narrative with a list of the affected analytes.
Continuing Calibration (CCAL)	Laboratory Analytical Accuracy	(1) Every 12 hours prior to the analysis of samples (2) Concentration level near midpoint of curve (3) Must contain all target analytes (4) Percent difference or percent drift must be ≤20 for CCCs and should be ≤30 for other compounds	No	Recalibrate as required by method (1) if %D of any of CCCs >20, or (2) if %D of >10% of other analytes >30.	Report non-conforming compounds in Environmental Laboratory case narrative.
Method Blanks	Laboratory Method Sensitivity (contamination evaluation)	(1) Every 20 samples prior to running samples and after calibration standards (2) Matrix and preservative-specific (e.g., water, MeOH, $NaHSO_4$) (3) Target analytes must be <RL except for common laboratory contaminants (such as acetone, methylene chloride, and MEK which must be <5x the RL)	Yes	Locate source of contamination; correct problem; reanalyze method blank.	(1) Report non-conformance in Environmental Laboratory case narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" flag or some other convention, should qualify the sample results. Blank contamination should also be documented in the Environmental Laboratory case narrative.
Laboratory Control Spikes (LCSs)	Laboratory Method Accuracy	(1) Every 20 samples or for each new tune clock, whichever is more frequent. (2) Prepared using standard source different than used for initial calibration (3) Concentration level must be at or near the mid-level (50%) standard (4) Must contain all target analytes (5) Matrix and preservative-specific (e.g., water, MeOH, $NaHSO_4$) (6) Laboratory–determined percent recoveries must be between 70 – 130 for target compounds. (7) Can also be used as CCAL	Yes	Recalculate the percent recoveries; Reanalyze the LCS; Locate source of problem; reanalyze associated samples.	(1) Report non-conformances in Environmental Laboratory case narrative. (2) Individual laboratories must identify and document "difficult" (**) analytes for which laboratory-determined recovery ranges routinely exceed the 100 ± 30% criterion. Exceedances for these "difficult" analytes must be qualified in Environmental Laboratory case narrative. Analytical data to support the "difficult" analyte classification must be available for review during an audit.

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
LCS Duplicate	Laboratory Method Precision	(1) Every 20 samples or for each new tune clock, whichever is more frequent. (2) Prepared using same standard source and concentration as LCS. (3) Must contain all target analytes. (4) Recommended to be run immediately after LCS in analytical sequence. (5) Laboratory–determined percent recoveries must be between 70 – 130 for target compounds (6) Matrix and preservative-specific (e.g., water, MeOH, $NaHSO_4$). (7) Laboratory–determined Relative Percent Difference (RPD) must be ≤ 25 except for "difficult" (**) analytes which must be ≤ 50.	Yes	Recalculate RPD; Locate source of problem; Narrate non-conformances	(1) Locate and rectify source of non-conformance before proceeding with the analyses of subsequent sample batches. (2) Individual laboratories must identify and document "difficult" (**) analytes for which laboratory-determined RPDs routinely exceed the ≤ 25 criterion. (3) Exceedances for these "difficult" analytes must be qualified in Environmental Laboratory case narrative. Analytical data to support the "difficult" analyte classification must be available for review during an audit. (4) Narrate non-conformances
MS/MSDs	Method Accuracy in Sample Matrix Method Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data-user) (2) Matrix-specific (3) Prepared by fortifying field sample with standard from source different than source used for initial calibration (4) Concentration level - between low (RL) and mid-level (50%) standard (5) Must contain all target analytes. (6) Percent recoveries - between 70 – 130 (7) RPDs should be ≤30 for waters and solids	Yes Only when requested by the data-user	Check LCS; if recoveries acceptable in LCS, narrate non-conformance.	Note exceedances in Environmental Laboratory case narrative.
Surrogates	Accuracy in Sample Matrix	(1) Evaluate surrogate recovery from individual field samples. (2) Minimum of 3 surrogates, at retention times across GC run (3) Percent recoveries must be between 70-130 for individual surrogate compounds. Laboratory-determined surrogate recovery limits that exceed ± 30% are acceptable for some difficult matrices (wastes, sludges, etc.) with appropriate analytical documentation.	Yes	If one or more surrogates are outside limits, reanalyze sample unless one of the following exceptions applies: (1) obvious interference present (e.g., UCM). (2) for methanol-preserved samples, re-analysis is not required if % moisture >25 and recovery is >10%. (3) if one surrogate exhibits high recovery and target analytes are not detected in sample.	(1) Note exceedances in Environmental Laboratory case narrative. (2) If re-analysis yields similar surrogate non-conformances, the laboratory should report results of both analyses. (3) If re-analysis is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time and yields acceptable surrogate recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Internal Standards (IS)	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 3 at retention times across GC run (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard (Section 5.10 of 8260B) (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard	No	If one or more internal standards are outside limits, reanalyze sample unless obvious interference present (e.g., UCM)	(1) Note exceedances in Environmental Laboratory case narrative. (2) if re-analysis yields similar internal standard non-conformances, the laboratory should report both results. (3) If re-analysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on IS calibration. (2) The laboratory must use the average response factor or linear regression curve generated from the associated initial calibration for quantitation of each analyte The IS used for quantitation must be the one nearest the retention time of the subject analyte.	NA	NA	(1) If the average response factor or linear regression are not used for analyte quantitation (e.g. quadratic equation), this must be noted in the Environmental Laboratory case narrative with a list of the affected analytes. (2) It is essential that the laboratory clearly document the calculation of analyte concentrations when non-linear calibrations are employed.
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc) for each analysis must be "reported". (3) Refer to Section 3.3, TIC Compounds by GC/MS for guidance	NA	NA	(1) Qualification of the data is required if reporting values below the sample-specific reporting limit. (2) Complete analytical documentation for diluted and undiluted analyses is to be available for review during an audit. (3) TICs will be evaluated at the discretion of the LSP consistent with the guidelines presented in Appendix II A–4. (4) The performance of dilutions must be documented in the Environmental Laboratory case narrative.

GC/MS = Gas Chromatography/Mass Spectrometry
BFB = 4-Bromofluorobenzene
MS/MSDs = Matrix Spikes/Matrix Spike Duplicates
%RSD = Percent Relative Standard Deviation
UCM = Unresolved Complex Mixture
"r" = Correlation Coefficient
CCC = Calibration Check Compounds
RPDs = Relative Percent Differences
TIC = Tentatively Identified Compound
NA = Not Applicable

**** Potentially "difficult" analytes include: acetone, bromomethane, chloroethane, dichlorodifluoromethane, diethyl ether, dibromochloromethane, hexachlorobutadiene, MEK, 4-methyl-2-pentanone, 1,4-dioxane and trichlorofluoromethane**

Attachment 8
Corrective Action and Documentation Examples

OCCURRENCE	ACTION	DOCUMENTATION
1. Initial calibration does not meet QC criteria.	1. Investigate source of problem, determine if source is an instrument problem or a standard solution problem. If problem is with a single point of the ICAL, reanalyze the bad standard and reevaluate. Depending on extent of problem, major maintenance or invoking manufacturer service contract for instrument repair will be performed.	1. Notation in instrument run log book, and if necessary notation in instrument maintenance log book.
2. Initial calibration verification check does not meet QC criteria.	2. Investigate source of problem, determine if source is with ICAL or ICV, is it an instrument problem or a standard solution problem, reanalyze ICV or perform new ICAL.	2. Notation in instrument run log book, and if necessary notation in instrument maintenance log book. If source determined to be bad standard solution, formal corrective action form must be initiated.
3. Continuing calibration verification check does not meet QC criteria.	3. Investigate source of problem. If source is instrument, perform instrument maintenance and reanalyze CCV. If CCV still will not pass, repeat the above, or perform new initial calibration. Depending on extent of problem, major maintenance or invoking manufacturer service contract for instrument repair will be performed.	3. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
4. GC/MS tune does not meet method criteria.	4. Investigate source of problem, evaluate instrument response to cal gas (PFTBA), when instrument response to PFTBA is improved, re-inject BFB tune.	4. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
5. Method blank contains target		

compound above reporting limit.	5. Investigate source of problem. Reanalyze all effected samples. If reanalysis is within holding time, report only these analyses. If they are beyond holding time, report both sets and notify project manager. If contaminant is not present in samples, data may be released with commentary.	5. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
6. Surrogate standard outside of acceptable range.	6. Investigate source of problem. If it is determined to be an instrument problem, reanalyze sample. If it is determined to be a preparation problem, analyze another aliquot of the sample. If it can be determined to be an obvious matrix problem (masking of surrogate by target or non-target compound at significantly greater concentration, excessive hydrocarbons in sample, other knowledge of sample matrix, etc.) the sample may be reanalyzed at dilution to reduce interference or reported with notation in narrative, depending on project objectives.	6. If only the reanalysis is reported make a notation in the instrument run log. If both sets of data are to be reported, notation in instrument run log, preparation logbooks, commentary on data review checklist to be included in project narrative, flagging all non-compliant values on Form 2 of data report. If source of problem found to be systematic (bad spike solution, etc), a formal corrective action form must be initiated.
7. Compound out of acceptance range in laboratory control sample.	7. Investigate source of problem. If LCS is acceptable per method/SOP specifications, associated sample data can be reported. If LCS recovery is above upper QC limit, and if analyte is not detected in associated samples, data may be flagged and reported. If LCS is not acceptable per method/SOP requirements, reanalyze all associated samples. If insufficient sample volume, notify project manager to discuss with client, report initial data if no other sample can be provided.	7. If LCS is acceptable per method/SOP, flag all compounds out of range on Form 3 of data report, if samples are reanalyzed within holding times, note in instrument run logbook. If samples are beyond holding time and both sets of data are to be reported, note in instrument run logbook and commentary in data review checklist to be included in project narrative. If reanalysis cannot be performed due to insufficient sample, commentary in data review checklist to be included in project narrative. If source of problem found to be systematic (bad spike solution, etc), a formal corrective action form must be initiated.
8. Compound in sample exceeds upper calibration standard concentration.		

	8. Reanalyze sample at dilution. If calibration limit exceedence is the only QC problem, report both initial and dilution analyses. If initial analysis has multiple QC problems, evaluate further to determine if initial run is to be reported (often this cannot be determined until the results of the dilution are evaluated). Instrument must be shown to be free of carryover contamination prior to acceptable analysis of next sample. If running instrument using autosampler, evaluate following sample. If following sample contains less than reporting limit of compound, the analysis is valid, and no instrument blank is required. If following sample(s) contain compound (typically in decreasing concentration—carryover typically occurs at 1% of concentration of high sample in following analysis, with effect more pronounced for later-eluting compounds. Effected samples must be reanalyzed if sufficient volume exists.	8. Notation in instrument run log book. If initial analysis is reported, flag compound exceeding calibration limit with "E" on data report and commentary on data review checklist to be included in project narrative. If only diluted analysis is to be reported, commentary in data review checklist to be included in project narrative. If both initial and dilution are to be reported, all of the above.
9. Instrument blank (GC) contains contamination above QC criteria.	9. Investigate source of problem, decontaminate purge and trap instrument, reanalyze all effected samples.	9. Notation in instrument run logbook, and if instrument maintenance performed, in instrument maintenance logbook.
10. Matrix spike recovery out of QC range.	10. Evaluate problem. If duplicate spike (MSD) shows same effect, it is generally matrix interference. If concentration of spike analyte is significantly (approx. 4 times) greater in unspiked sample, this is matrix interference masking quantitation of spike concentration. If source cannot be determined, reanalyze spike sample.	10. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.
11. Duplicate (or MSD) relative percent difference exceeds QC limit.	11. Evaluate problem. If concentration of analyte is	11. Flag RPD on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.
12. Internal Standard areas exceed QC criteria. (-50% to +100%)		

A. CCV. B. QC (blank, LCS) C. Samples and MS/MSD.	close to reporting limit, variation of analysis is acceptable. If sample is soil or other heterogeneous matrix, high RPD is typical. If sample is a typically homogeneous matrix, reanalyze duplicate sample. 12. A. Evaluate problem. Re-analyze CCV. If the CCV does not meet criteria re-analyze the initial calibration and proceed with CCV/QC/samples. B. Evaluate CCV and QC. As blank and LCS are "interference-free matrix" the IS areas should be within the same limits as the CCV. Evaluate for potential problems. If time allows (data not required on a rush basis), reanalyze QC prior to sample analysis. If insufficient time due to client deadline, data may be reported (as they meet method requirements) but the issue should be noted for the data reviewer and for the client. C. Evaluate CCV and QC. The IS areas may indicate a potential problem, or matrix interference. If the CCV and batch QC meet criteria, document and report results as matrix interference. In particular, if the recovery of the surrogate standard associated with the IS compound is within the recovery range, then the internal standard method is effectively quantifying the compounds. If the associated surrogate is outside of the recovery criteria, the IS issue is impacting quantitation. Evaluate whether this indicates a potential high or low bias for the associated compound results (low IS=high surrogate=high bias; high IS=low surrogate=low bias) This requires notation and	12. A. Document in the analytical run log. B. If the criteria are met after re-analysis, document in run log. If the criteria have not been met and the results of the sample batch are reported, document in the run log and the Corrective Action Logbook. Have the supervisor review situation, initial/date, and include a comment on the data review checklist for the data reviewer and for inclusion in the narrative information to the client. C. If the CCV and QC meet criteria, document in the run log and on the package checklist for inclusion in the narrative submitted to the client. Note that certain compounds may be potentially high bias or potentially low bias due to IS recoveries outside of range. If QC and samples do not meet criteria and the results are reported, document in the run log, document in the LIMS Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer and for inclusion in the narrative.

	communication of the effect to the data reviewer and the client. Based on the severity of the problem, discuss with supervisor, technical director and /or reanalyze effected samples. If results are reported as is, document per 12C.	

Sample Preparation of Aqueous Samples by Acid Digestion Using SW 846 Methods SW3010/SW3005 for Analysis by ICP/AES or ICP/MS

Contents SOP NO. 100.0003

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		2/22/10
Quality Assurance Director		2/20/10
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	2/27/11				
	Supervisor	12/6/11			
	Supervisor	01/14/13			

Revision Record

Revision Date	Revision Description	Comments	Initials
3/13/08	Clarified initial pH test and LCS/MS target list, lab name edit		SBL
6/23/08	Volume of LCS/MS edit	450ul to 455ul, 45.0 ul to 45.5 ul	SBL
3/04/09	Add ICP/MS preparation		SBL
1/11/10	Revision of SW method not included	3005A and 3010A from 7/1992	SBL
02/16/10	Updated spiking section		SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample Preparation of Aqueous Samples by Acid Digestion
Using SW 846 Methods SW3010/SW3005
for Analysis by ICP/AES or ICP/MS

SOP No. 100.0003

Rev. 8

	Signature	Date
QA Director:	Maryn B Sanders	2/20/10
Lab Director:		2/20/10
Effective Date:	2/27/10	

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A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

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Sample Preparation of Aqueous Samples by Acid Digestion
Using SW 846 Methods SW3010/SW3005
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Rev. 8

1. Scope and Applications

This Standard Operating Procedure (SOP) deals with the preparation of aqueous samples for analysis by USEPA SW846 methods 6010 and 6020A. Discussion includes sample extraction and sample concentration technique and analysis of dissolved metals or total/ total recoverable metals in aqueous samples using methods SW-846 SW3005/3010.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. Supervisors/lab managers are responsible for ensuring that this SOP is accurate and up to date, and that it is implemented appropriately. Supervisors/lab managers review the logbooks and data generated for this procedure and approve all reported results.

3. Summary of Procedure

A 50mL aliquot of an aqueous sample is digested with acid prior to metals analysis by Inductively Coupled Argon Plasma (ICAP). This method has been modified from the original 100mL sample volume to reduce reagent use and acid waste. All reagents have been adjusted accordingly. ICP-MS preparation has been modified to utilize a lower volume of HCL to minimize molecular-ion interferences. Refer to Chapter 2 of SW-846 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.

4. Sample Preservation, Containers, Handling, and Storage

4.1 The hold time for ICAP analysis is 180 days from the date of sample collection.

4.2 Aqueous samples for Total recoverable metals analysis must be preserved with concentrated HNO_3 to a pH of less than 2 and stored in an amber glass or polyethylene bottle.

4.3 Aqueous samples for dissolved metals must be filtered through 0.45um filter paper prior to preservation with concentrated HNO_3 to a pH <2.

5. Interferences and Potential Problems

Possible contamination:

5.1 Hood fall-out.

5.2 Acid bath for glassware contaminated.

5.3 Acid dispensers.

5.4 DI water rinsing bottles contaminated.

5.5 Poor lab technique.

5.6 Cross-contamination from high-level samples.

5.7 Sample matrix effects: Extreme organic samples.

6. Equipment and Apparatus

Equipment used in this preparation method includes:

6.1 10% HNO_3 acid bath, reagent grade acid.

6.2 50 mL Polyethylene centrifuge/digestion tubes with caps.

6.3 Polyethylene ribbed Watch Glasses.

6.4 Whatman 41 filter paper (if necessary).

6.5 50 ml Plastic funnels (if necessary).

6.6 Thermometers- NIST Calibrated (0-100°C)

6.7 Hot plates with graphite tube holders for even heating.

6.8 Wide range pH paper, 0-14, Whatman ColorPHast.

6.9 Narrow range pH paper 0-2.5, Whatman ColorPHast.

6.10 Eppendorf and/or Wheaton pipettes.

6.11 Beckman GPR Centrifuge.

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

7.1 Concentrated HNO_3, ACS trace metals grade.

7.1.1 1:1 (v/v) HNO_3 ACS trace metals grade.

7.2 Concentrated HCl, ACS trace metals grade.

7.2.1 1:1 (v/v) HCl, ACS trace metals grade.

7.3 Spiking solutions, High Purity Standards.

8. Procedure

8.1. Digestion of highly colored total water samples for ICAP Analysis **(Method 3010)**:

Enter Lab ID #'s of all the samples to be digested into the proper column in the Sample Digestion Logbook. All volumes of reagents added as well as the initial and final volumes must be entered in this Logbook.

8.1.1. The sample pH should have been checked at the time of receipt in sample Log-in. Prior to analysis, re-check the pH for aqueous samples using the wide range pH paper (pH should be <2). If the pH strip color is more red than magenta, or there is any doubt of the pH being less than 2, validate the pH with the narrow range pH paper which measures pH 0 to 2.5. Enter observations in the appropriate column in the Logbook and adjust and note the pH if not less than 2. If pH adjustment needs to be done, be sure to notify the client first. Acidify the sample with concentrated HNO_3 to a pH <2. Document all pH adjustments. Shake sample well and let it sit for 24 hours before digestion.

8.1.2. Transfer 50 mL of sample into a plastic digestion tube using the graduated marking on the tube to reach volume.

8.1.3. Add 1.5mL conc. HNO_3 to the sample.

8.1.4. Cover with a ribbed watch glass.

8.1.5. Place the tubes in the graphite holders, on a hotplate, inside the inorganic hood (PVC ducting).

8.1.6. Heat at 95^{o}C (90°C – 95°C is an acceptable temperature range) until the sample volume has been reduced to approx. 2.5 –5.0mL. Do not boil the sample or let it go dry.

8.1.7. Take tubes off the hotplate and allow them to cool to room temperature.

8.1.8. Add another 1.5mL of conc. HNO_3 to each sample.

8.1.9. Heat the sample again and reduce to 1.5 –3.0 mL, ensuring a reflux action occurs.

8.1.10. Continue to add HNO_3 in 1.5mL aliquots until digestate appears pale in color. (Add no more than 5mL conc. HNO_3.)

8.1.11. Cool the tube and add 5mL of 1:1 (v/v) HCl.

8.1.12. Heat for an additional 15 minutes.

8.1.13. Rinse the walls of tube with DI water.

8.1.13.1. If the sample is not to be filtered, volumize the sample back to 50 ml with DI water using the graduation on the tube to obtain the proper volume.

8.1.13.2. If sample needs to be filtered, volumize the sample to 50 ml and filter through Whatman 41 filter paper. Do not rinse filter.

8.1.14. Assign the samples to a preparation batch using Mitkem's LIMS system.

8.1.15. Transfer logbook and digestates to the Metals Instrument Lab.

8.2. Digestion of Dissolved or Total/Total Recoverable Aqueous Samples and Leachates for ICAP Analysis **(Method 3005)**:

Enter Lab ID# of all samples to be digested into the proper column in the Sample Digestion Logbook. All volumes of reagents added as well as the initial and final volumes must be entered in the Logbook.

8.2.1 Dissolved samples must be filtered first through 0.45um filter paper and preserved with conc. $HNO_{3.}$ Typically this is done in the field however, if samples arrive at Mitkem requiring filtration, it should be done ASAP. Our general goal is within 24 hours of receipt.

8.2.2 Transfer a 50mL aliquot of the sample to a plastic digestion tube using the graduated marking on the tube to reach volume.

8.2.2.1 Add 1.0mL of conc. HNO_3 and 2.5mL of conc. HCL for **ICP-AES** samples.

8.2.2.2 Add 1.0mL 1:1 HNO_3 and 0.5mL of 1:1 HCL for **ICP-MS** samples.

8.2.3 Heat to 90-95^{o}C in graphite holders on hotplates in the hood. Cover each tube with a ribbed watch glass and evaporate until the sample volume equals 7.5-10 mL.

8.2.4 Remove tube and allow to cool.

8.2.5 Rinse the walls of the beaker with DI water and volumize to 50mL with DI water.

8.2.6 If insoluble material remains, volumize sample to 50 mL and filter through Whatman 41 filter paper, or centrifuge.

8.2.7 Record all digestion information in the Aqueous Metal Prep Logbook (**figure 1)**.

8.2.7.1 ICP-MS samples may be recorded in the Method E200.7 E200.8 Aqueous Metal Prep Logbook (**figure 2**) as the concentration of the acids are the same. Make sure to circle the method SW3005A ICP/MS. Initial and date all crossouts.

8.2.8 Assign the samples to a preparation batch using Mitkem's LIMS system.

8.2.9 Transfer logbook and digestates to the Metals Instrument Lab.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are established to ensure generation of data of known quality. QA/QC procedures associated with the Inorganic Prep Lab include preparation of Method Blanks, Lab Control Samples, matrix spikes and sample duplicates.

10.1. Method blanks:

A method blank must be run with every discrete batch of samples that is prepped. The number of samples per batch may not exceed 20.

10.1.1 Use 50mL of DI water as the method blank for aqueous sample preparation. Prep this sample as described in **Section 8**. Label appropriately.

10.2. Laboratory Control Samples:

A laboratory control sample containing all target analytes must be prepped and analyzed for each batch of samples. The number of samples per batch may not exceed 20.

10.2.1 For **ICP-AES** samples: measure approximately 40 ml of DI water as if it were a sample and add High Purity Standard CLP-CV-1 at 455µL; CLP-CV-2 and CLP-CV-3 at 45.5 µL each. Prep this sample as described in **Section 8**. Label appropriately.

10.2.2 For **ICP-MS** samples: measure approximately 40 ml of DI water as if it were a sample and add 0.5 mL of an intermediate standard containing Mg, Ca, Na, and K, 50 µL of an intermediate standard containing Sb, As, Cd, Pb, Se, and Tl, 50 µL of CLP Spike 1A and CLP Spike 1B. Prep this sample as described in **Section 8**. Label appropriately.

10.3. Matrix Spikes and Duplicate Samples:

10.3.1 With at least every 20 samples a sample duplicate and sample spike (containing all target analytes) must be prepped and analyzed by the laboratory. Prepare 3 digestion tubes to be used for the same aqueous sample. Designate one tube as the sample, one as the duplicate of that sample, and the other as a spiked portion of the sample. Into each tube, measure 50mL of sample (the same sample).

10.3.1.1 Spike Sample:

- For **ICP-AES** samples: add three spiking standards; High Purity Standard CLP-CV-1 at 455µL; CLP-CV-2 and CLP-CV-3 at 45.5µL each to the sample. Label this sample: "LabID"'MS'.
- For **ICP-MS** samples: add 0.5 mL of an intermediate standard containing Mg, Ca, Na, and K, 50 µL of an intermediate standard containing Sb, As, Cd, Pb, Se, and Tl, 50 µL of CLP Spike 1A and CLP Spike 1B. Label this sample: "LabID"'MS'.

10.3.1.2 Sample Duplicate:

Label the other additional 50mL of sample as "LabID"'D'. This is an exact duplicate of the original sample. Digest the sample duplicate as described in **Section 8**.

10.4 Standard Preparation

10.4.1 All standards made from a primary standard expire on or before the primary standard's expiration date. For standards without manufacturer-designated expiration dates, the expiration date will be 1 year from receipt.

10.5 Digestion Tubes and Pipettes.

10.5.1 All lots of digestion tubes must be tested for conformance. A group of 10 tubes are measured for volume by weight. The weights are recorded and the average weight is calculated. See SOP 80.0030 Labware Volume Verification for acceptance criteria.

10.5.2 Pipettes: Two sizes of pipettes are used in the method; 0 to 1000 µL and 0 to 100 µL. Five replicate weights are recorded. The average of the five replicates is calculated. See SOP 80.0030 Labware Volume Verification for acceptance criteria.

11. Data Validation and Reporting

Data generated in the inorganic preparation laboratory will be reviewed and signed by a peer, the supervisor or the manager.

12. Corrective Action Procedures

12.1 If the method blank concentration for any analyte is greater than the established method criteria, the samples will be scheduled for re-prep by the metals analyst/supervisor.

12.2 If the LCS is outside of control limits, the samples will be scheduled for re-prep by the metals analyst/supervisor for those elements that failed.

12.3 If a Spike sample was not spiked, the sample will be scheduled for re-prep by the metals analyst/supervisor.

13. Health and Safety

Health and safety hazards in the Inorganic Preparation Laboratory (prep lab) include exposure to concentrated acids, their fumes and toxic metals standards. Labcoats, gloves and safety glasses must be worn in the prep lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. Reference

1. U.S. Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Update 3 Method 3005A, Revision 1 July 1992.

2. U.S. Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Update 3 Method 3010 A, Revision 1 July 1992.

3. U.S. Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Chapter Three: Inorganic Analytes. Revision 4, February 2007.

Attachments

Figure 1: Aqueous Metal Prep Logbook
Figure 2: Method E200.7/200.8/SW3005MS Aqueous Metal Prep Logbook

Figure 1
Aqueous Metal Prep Logbook

Start time: **MITKEM LABORATORIES: Aqueous Metals Preparation Logbook**

Date	Sample ID		Client ID	Sample Vol (ml)	pH	Sample Color Before	Sample Clarity Before	Conc. HNO_3 (ml)	Conc.H Cl (ml)	1:1 HCl (ml)	Sample Color After	Sample Clarity After	Final Volume (ml)	Comments

HCl Lot# _____

HNO3 Lot# _____

Analyst:_____

Method#:_____

Hotblock ID: _____

Digestion Temp:_____**oC/ MT-**____

LCS/Spike Lot # _____

_____

_____

Digestate Relinquished to:_____

Narrative Notes on Reverse Side

Yes / No

Logbook ID 100.0125 -xx/10

Reviewed By: _____

Figure 2

Method E200.7/E200.8/SW3005ICP/MS Aqueous Metal Prep Logbook

Start time: **MITKEM LABORATORIES: Aqueous Metals Preparation Logbook**

Date	Sample ID		Sample Vol (ml)	Sample pH	Sample Color Before	Sample Clarity Before	1:1 HNO_3 (ml)	1:1 HCl (ml)	Sample Color After	Sample Clarity After	Final Volume (ml)	Comments

HCl Lot# _____

HNO3 Lot# _____

Analyst:_____

Method#(circle one) E200.7 E200.8

SW3005 ICP/MS

Hotblock ID: _____

Digestion Temp:_____oC/ MT-_____

LCS/Spike Lot # _____

_____

_____

Narrative Notes on Reverse Side

Yes / No

Digestate Relinquished to:_____

Logbook ID 100.0130 -01/10

Reviewed By: _____

Total Cyanide by Automated Colorimetric with Midi-distillation SW846 9012B

Document Control Page

Contents SOP NO. 100.0004 **1. Procedure Document**	
2. Training Document	**N/A**
3. Process Overview	
4. Validation Document	**N/A**

Procedure Signatures

Title:	**Signature**	**Date**
Laboratory Director/Technical Director		
Quality Assurance Director		
Laboratory/Quality Designee		

Procedure Reviews

Signature	**Title**	**Date**	**Signature**	**Title**	**Date**

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Revision Record

Revision Date	Revision Description	Comments	Initials
10/24/07	Added information for working LCS and check std solutions, fixed some typos. Added section 4.6	Section 8	SBL
12/1/07	Lab name change		SBL
3/11/08	Removed degassing requirement for reagent preparation	Was not providing any benefit to procedure	SBL
4/22/09	Stopped distilling ICV. Changed distilled std to 0.3ppm	Working stds prepared day of use	SBL/NJ
7/22/10	Revised ICV, midrange std, CRA req	Removed references to distilled calib check.	HZA
11/15/10	More detail added to sulfide removal	EHGS MICE recommendation, NELAC. Full rev.	SBL
1/24/12	Software information	Minor rev	SBL
3/12/12	Added hi/lo distilled std req for DoD only	Lab name change Full rev	SBL
05/03/13	Calibration range edited	Full	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

SPECTRUM ANALYTICAL, INC.
Featuring Hanibal Technology,
Rhode Island Division

STANDARD OPERATING PROCEDURE

Total Cyanide by Automated Colorimetric with Midi-distillation

SW846 9012B

SOP 100.0004

Rev. 9

	Signature	Date
QA Director:	_____	_____
Lab Director:	_____	_____

Effective Date: _____

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SPECTRUM ANALYTICAL, INC.
Featuring Hanibal Technology,
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Total Cyanide by Automated Colorimetric with Midi-distillation

SW846 9012B

Rev. 9

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of and determination of the concentration of inorganic cyanide (CAS Registry Number 57-12-5) in solid and aqueous wastes and leachates by US EPA SW846 Method 9012B. The method detects inorganic cyanides that are present as either soluble or complex salts. It is used to determine values for both total cyanide and cyanide amenable to chlorination. The "reactive" cyanide content of a waste, that is, the cyanide content that could generate toxic fumes when exposed to mild acidic conditions, is not distilled by Method 9012. Method 9012 may be used however to quantify the concentration of cyanide from the reactivity test.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts and technicians are responsible for performing analyses in accordance with the SOP and documenting and variations in the protocol. Supervisors/Managers are responsible for ensuring that SOPs are accurate and up to date, and that they are implemented appropriately. Supervisors review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

3.1 The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by automated UV colorimetry.

3.2 In the automated colorimetric measurement, the cyanide is converted to cyanogen chloride (CNCl) by reaction with Chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed by the addition of pyridine-barbituric acid agent. The concentration of NaOH must be the same as in the standards, the scrubber solutions, and any dilution of the original scrubber solution to obtain colors of comparable intensity.

4. Sample Preservation, Containers, Handling and Storage

4.1 Holding time for cyanide analysis is 14 days from date sampled.

4.2 Samples should be collected in pre-cleaned glass or plastic containers.

4.3 Samples are kept at 4°C.

4.4 When properly preserved, cyanide samples can be stored for up to 14 days prior to sample preparation steps.

4.5 Aqueous samples must be preserved by adding sodium hydroxide until the pH is greater than or equal to 12.

4.6 If fatty acids, detergents, and surfactants are a problem, they may be extracted using the following procedure. Acidify the sample with acetic acid (1.6M) to pH 6.0 – 7.0.

CAUTION: This procedure can produce lethal HCN gas.

Extract with isooctane, hexane, or chloroform (preference in order named) with solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the compounds below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When the extraction is completed, immediately raise the pH of the sample to above 12 with 50% NaOH solution.

5. Interferences and Potential Problems

5.1 Interferences are eliminated or reduced by using the distillation procedure. Chlorine and sulfide are interferences.

5.2 Oxidizing agents such as chlorine decompose most cyanide. To determine whether oxidizing agents are present, test a drop of the sample with potassium iodide-starch test paper. A blue color indicates the need for treatment. Add 0.1N sodium arsenite solution a few mL at a time until a drop of sample produces no color on the indicator paper. Add an additional 5mL of sodium arsenite solution for each liter of sample. Ascorbic acid can be used as an alternative although it is not as effective as arsenite.

Add a few crystals of ascorbic acid at a time until a drop of sample produces no color on the indicator paper. Then add an additional 0.6g of ascorbic acid for each liter of sample.

5.3 Sulfide interferences can be removed by adding an excess of cadmium carbonate to the waste extract (to precipitate the sulfide) after distillation followed by filtration. Samples that contain hydrogen sulfide, metal sulfides or other compounds that may produce sulfide during the distillation should be treated by the addition of bismuth nitrate or cadmium carbonate.

5.3.1 Bismuth nitrate is added to the sample prior to distillation if sulfide is detected. If bismuth nitrate is used, the method requires the calibration curve be distilled with this added as well. See **section 8.4.3**.

5.3.2 Alternately cadmium carbonate can be used to remove sulfides. Avoid adding a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Because of this potential loss, it is recommended that at least two check standards per distillation batch are prepared using the same contact time and maximum amount of cadmium carbonate employed for any field sample in order to verify acceptable cyanide recovery. See section 8.2.3 of SOP No. 100.0033 EPA Method E335.4.

5.4 High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds, once formed, will decompose under these conditions to generate HCN. The possibility of interference of nitrate and nitrite are eliminated by pretreatment with sulfamic acid just before distillation. Nitrate and nitrite are interferences when present at levels higher than 10mg/L and in conjunction with certain organic compounds.

5.5 Thiocyanate is reported to be an interferent when present at very high levels. Levels of 10mg/L were not found to interfere in Method 9012.

5.6 Fatty acids, detergents, surfactants, and other compounds may cause foaming during the distillation when they are present in large concentrations and will make the endpoint of the titration difficult to detect. A few drops of an anti-foaming agent may be added to help alleviate foaming upon distillation.

6. Equipment and Apparatus

Equipment and instrumentation used in this preparation and analysis method include:

6.1 Fisher Midi-distillation apparatus.

6.2 Vacuum pump.

6.3 Graduated cylinder – 50mL.

6.4 Volumetric flasks – 50mL, 100mL, 1L.

6.5 Top loading balance.

6.6 Boiling stones – Teflon.

6.7 Refrigerator.

6.8 Adjustable pipettes.

6.9 Automated continuous flow analytical instrument – Lachat Quick Chem-8000 with:

6.9.1 Autosampler.

6.9.2 Manifold/peristaltic pump.

6.9.3 Heating coil.

6.9.4 Colorimeter equipped with a 15mm flow cell and 570nm filter.

6.9.5 Computer automation/recorder.

6.9.6 Data system; Omnion Data System Version 2.0

6.10 100mL polyethylene bottles.

6.11 Repeating Eppendorf-type pipettes.

7. Reagents and Standards

Reagent grade chemicals shall be used in all tests. All reagents shall be ACS approved. *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.* The following reagents are used extensively in the lab:

7.1 Reagents for distillation:

7.1.1 0.25N Sodium hydroxide: Dissolve 10g to 1L of DI H_2O.

7.1.2 Bismuth nitrate (0.062M), Dissolve 30g of Bi(NO)3 C 5H2O in 100mL of water. While stirring, add 250mL of glacial acetic acid. Stir until dissolved and dilute to 1 liter with water.

7.1.3 Cadmium Carbonate powder (alternate for treating samples with excess sulfide).

7.1.4 4.0N Sulfamic acid, H_2NSO_3H: Dissolve 40g in 100mL of DI H_2O.

7.1.5 50% (18N) Sulfuric acid: Slowly and carefully add 500mL of concentrated H_2SO_4 to 500mL of DI H_2O while submerged in a controlled ice bath. Caution: solution will become excessively hot.

7.1.6 2.5M Magnesium chloride solution: Dissolve 510g of $MgCl_2 \bullet 6H_2O$ in 1L of DI H_2O.

7.1.7 Lead acetate paper.

7.1.8 0.1N Sodium arsenite: Dissolve 3.2g $NaAsO_2$ into 250mL of DI H_2O and mix thoroughly.

7.1.9 Potassium-iodide starch paper.

7.1.10 Reagent water (ASTM Type I water). Our water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. Reagent water is also referred to as DI water.

7.2 Reagents and standards for automated colorimetric determination: *Please note that standards and reagents from other vendors could be used as long as the standards are of high purity (>96%) and traceable to reference materials.*

7.2.1 Pyridine-barbituric acid reagent: Place 60g of barbituric acid into a 4L volumetric flask that has a small quantity of DI H_2O, and then add just enough reagent water to wash the sides of the flask, and wet the barbituric acid. Add 300mL of pyridine and mix with the use of a stir bar and stir plate. Add 60mL of concentrated HCl, mix and cool to room temperature. Dilute to 4L with DI H_2O and mix. This reagent can be stored up to 6 months in a cool, dark place.

7.2.2 Chloramine-T solution: Dissolve 4.0g of white water-soluble Chloramine-T in 1L of DI H_2O. Refrigerate until ready to use. Prepare fresh daily. For short expiration times, add 1.0g to 250mL.

7.2.3 0.25N Sodium hydroxide: Dissolve 10g NaOH in DI H_2O and dilute to 1L.

7.2.4 0.71M Potassium Phosphate Monobasic (buffer agent for Lachat): Dissolve 97.0g into 1L of DI H_2O. Prepare fresh every 28 days.

7.2.5 1000mg/L commercially available standard; Primary source ERA. Second source SPEX Certiprep.

All working standards should be prepared in the same concentration NaOH as samples and QC samples.

8. Procedure

8.1 Calibration Curve:

8.1.1 Intermediate standard – Pipette 1mL of 1000mg/L commercially available Cyanide standard into a 100mL Class A volumetric flask. Bring to volume with 0.25N NaOH solution to prepare a 10ug CN^-/mL standard. Label as IZyymmdd# where,

II = Inorganic Intermediate
yy = year of preparation
mm = month of preparation
dd = date of preparation and
# = the sequential letter of intermediate standards prepped on this day (A,B,C…)

8.1.1.1 Prepare working curve from the Intermediate Calibration Standard. The working standards are prepared by taking the following volumes and diluting them to 50mL with 0.25N NaOH in a volumetric flask to get the following concentrations:

Volume (mL) (1mL = 0.01mg CN^-)	Standard ID (mg CN^-/L)
0.0	S0.0
0.05	S0.010
0.125	S0.025*
0.25	S0.050
0.50	S0.100
1.0	S0.200
1.5	S0.300*

*The DoD high and low distilled standards are prepared in this same manner/concentration.

8.1.1.2 Prepare the working solution CCV at 0.15mg CN^-/L from the Intermediate standard above, by pipetting 0.75mL of the intermediate standard into a 50mL volumetric flask and bring to volume.

8.2 ICV Standard Preparation:

8.2.1 Prepare a second source intermediate ICV solution by pipetting 1mL of the second source 1000mg/L commercially available standard into a 100mL volumetric flask containing 0.25N NaOH. This will yield a concentration of 10mg/L.

8.2.2 Prepare the Working ICV solution at 0.20mg CN^-/L from the intermediate ICV solution in section 8.1.3. Pipette 1.0mL of the intermediate standard into a 50mL volumetric flask and bring to volume. Prepare fresh on day of use. Label the working standard IWyymmdd# where:

IW = Inorganic Working
yy = year of preparation
mm = month of preparation
dd = Date of preparation
# = the sequential letter of intermediate standards prepped on this day (A,B,C…)

8.3 LCS Standard:

Prepare the LCS working solution at 0.1mg CN^-/L from the Intermediate ICV solution in section 8.1.3. Pipette 0.5mL of the intermediate standard into a 50mL volumetric flask and bring to volume.

NOTE:

- All standards are prepared in 0.25N NaOH.
- All standards prepared from a primary standard expire on or before the primary standard's expiration date.
- The intermediate standard expires 28 days from date prepared.
- Working standards are prepared fresh on the day of use.

8.4 Distillation Procedure:

See SOP 110.0039 for soil sub-sampling procedures.

8.4.1 Place 50mL of 0.25N NaOH into the gas absorbing impinger.

8.4.2 For aqueous samples, pour 50mL aliquot of sample into the back sample tube along with at least 3 Teflon boiling stones.

8.4.2.1 To the matrix spike, add 0.5mL of the intermediate standard prepared in section 8.1.1.

8.4.3 Test for the presence of sulfides with lead acetate paper. If the paper turns blue to blue-black then the sample contains sulfides. Add 5mL of 0.062M bismuth nitrate solution through the air inlet tube. Allow to mix for at least three minutes.

Prepare a calibration curve with the addition of the bismuth nitrate to all standards as well.

8.4.4 Test the sample for the presence of excess chlorine with potassium-iodide starch paper. A blue color is a positive result. If oxidizing agents are present, add 0.1N sodium arsenite a few drops at a time until a drop of sample produces no color change of the sample. Then add an additional 5mL of sodium arsenite for every liter of sample.

8.4.5 For soil samples, calibrate the balance and then weigh out 1.00 to 2.00g of sample. Add 50mL of DI water and proceed with the distillation procedure. Be sure to check all soil extracts with lead acetate paper after distillation for the presence of sulfides.

8.4.6 Connect the sample tube and the gas scrubber and the vacuum source in the train.

8.4.7 Start a slow steady stream of air bubbles in the sample tube by adjusting the vacuum inlet valve. 2-3 bubbles per second is acceptable.

8.4.8 Turn on the pre-adjusted chiller: adjust the poundage of the water flow according to the number of samples you are distilling.

8.4.9 Add 0.5mL of 4 N sulfamic acid to every sample to remove any nitrate/nitrite interferences.

8.4.10 After 5 minutes of vacuum flow, pipette 5mL of 50% H_2SO_4 with the Mopet pipette aide through the top air inlet tube of the distillation head and into the reaction vessel. Allow to mix for 5 minutes.

8.4.11 Add 2mL of magnesium chloride solution through the top air inlet tube of the distillation head into the reaction flask by using the Mopet pipette aide. Excess foaming can be controlled by adding another 2mL of magnesium chloride at this point. Adjust vacuum if necessary, allow mixing for 5 minutes.

8.4.12 Turn on the heating block and adjust temperature control to 125°C. Set the timer for 120 minutes. Heat the solution to boiling. Be careful to prevent solution backup by periodic adjustment of the vacuum flow.

8.4.13 After approximately 90 minutes of refluxing, turn off the heat and continue the vacuum for an additional 15 – 30 minutes. Leave the chiller on. The sample tubes should be cooled within this time.

8.4.14 After samples have cooled, turn off the vacuum and chiller. Place sample distillate into a 100mL bottle and store until analysis. All distillation information is documented in the Cyanide Distillation Logbook, **figure 1**.

8.4.15 Assign the samples to a preparation batch using the LIMS system.

8.5 Analysis:

Turn on the master switch for the Lachat to allow warming to 60°C.

8.5.1 Set up the cyanide manifold per the Lachat Instruments Auto Analyzer Model 8000 instructions ensuring the proper tubing is connected to the appropriate reagent. See section 17 of Lachat Methods Manual Quick Chem 8000 in **figure 2,** for more details.

8.5.2 Set up your run on the computer in the order you will run it on the instrument. Name your run and the analysis parameters, including method name, tray name. Print out tray worksheet.

The analytical sequence will be run with the following setup:

1. S0 (Calibration Blank)
2. S 0.01mgL
3. S 0.025mgL
4. S 0.05mg/L
5. S 0.10mg/L
6. S 0.2mg/L
7. S 0.3mg/L
8. DoD low distilled standard*
9. DoD high distilled standard*
10. ICV
11. ICB
12. CCV
13. CCB
14. Method Blank
15. LCS
16. Sample
17. Sample
18. Sample
19. Sample (Maximum of 10 samples)
20. CCV
21. CCB
22. Sample
23. Sample
24. Sample

25. Sample
26. Sample
27. Sample
28. Sample
29. Sample
30. Sample
31. Sample (Maximum of 10 samples)
32. CCV
33. CCB

*DoD QSM projects only: Each calibration must be verified by the analysis of a distilled low and high standard, run after the ICAL and before any samples. The standard must meet within ±15% of its true value.

8.5.3 Place your standards and samples on the auto-sampler tray in the following order: "S" 0, 0.01, 0.025, 0.05, 0.1, 0.2, 0.3 mg/L, then ICV, ICB, CCV, CCB, method blank, LCS(W/S) and samples.

8.5.4 Place the carrier and reagent lines in the solutions; allow solution to run through for at least 10 minutes to remove any/all air bubbles in the lines.

8.5.5 Begin the analysis by requesting "RUN TRAY" on the icons. The computer will instruct the analyzer to begin with the standard's injections. After the instrument has finished sampling the standards, it will automatically calculate the Initial calibration curve to a minimum required 0.995 correlation in order to proceed, or will stop if correlation fails the 0.995 requirement.

8.5.6 If the calibration does not meet the requirement criteria, further action is needed. See the data reduction and calculations section for further guidance and corrective action.

8.5.7 When analysis is complete, clean all the lines with DI water for a period of 5 – 10 minutes. The analysis information is documented in the Lachat Run Logbook, **figure 3**. Download the data file to the network server for uploading into the LIMS system.

9. Data Reduction and Calculations

The Lachat Omnion Data System will calculate the concentration from the plotted curve. This is based on the instrument response (absorbance) against the standard concentrations.

9.1 A linear correlation of ≥0.995 must be established before sample analysis can occur. A minimum of 5 standards and a blank establishes the calibration curve.

9.2 Document the calibration curve by pressing the **analyze** icon. Clear any old calibration information and press the **analyze** icon again. Print the current calibration curve information.

9.3 Go to the icons at the bottom of the screen, and click on the **FIA** analysis icon. Go to the **custom** icon at the top of the page. Customize your report and print the data. Make sure to save your report before exiting this part of the program.

9.4 Exit the program; exit the FIA analysis program to main menu screen. At this point, you may shut off all the mechanical parts as well as the computer using the main power switch.

9.5 All calculations are performed internally by the computer. Concentration is measured against the calibration curve and printed in units of mg/L CN.

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Inorganic Laboratory include preparation of a Method Blank, lab control sample, matrix spike, sample duplicates, balance checks and pipette calibrations.

10.1 Distillation – Every distillation batch of up to 20 samples contains the following:

- A method blank: 50mL of DI water (Aqueous batch), or 1g of Ottawa sand (Soil batch).
- An LCSW for aqueous samples; add 50mL of prepared 0.1 mg/L that is made from the ICV Intermediate in 0.25N NaOH (second source).
- An LCSS for soils; add 50mL of prepared 0.1 mg/L that is made from the ICV Intermediate in 0.25N NaOH into a sample tube containing 1g of Ottawa sand.
- A duplicate sample and a spike sample are performed every 20 samples. The Matrix Spike sample is prepared by adding 0.5mL of the Intermediate calibration standard to a third aliquot of a field sample.
- A high and a low standard must be distilled to verify each DoD calibration. The standards must meet within ±15% of true value or the calibration is not valid.

10.2 The correlation coefficient of the curve must be ≥0.995.

10.3 The ICV must meet within ±15% of the true value.

10.4 The method blank analyzed with every batch should not exceed 20µg/L (MRL) of CN^-. Low level blank contamination is acceptable where sample concentration is **at least 10 times** the concentration of the method blank.

DoD QSM; the method blank concentration can not exceed one half the RL, or 10ug/L CN^-.

10.5 CCV's are analyzed at least every 10 field samples with the recovery requirements of 85 – 115%. All ICVs and CCVs are followed by an ICB and CCB, respectively. The ICB and CCBs should not exceed 20µg/L CN (MRL).

10.6 Matrix spike and duplicate samples are distilled and analyzed at a minimum of one set per 20 samples or every distillation batch, whichever occurs first. Post spikes are analyzed if the spike recovery is not within 75 – 125%. The post-digestion spike level = 2 x the MRL or 2 x the sample concentration, whichever is greater. Duplicates can be repeated if the RPD is greater than 20%.

DoD QSM; Matrix spike recovery use the same control limits as LCS, 80-120%.

10.7 LCS Recovery is evaluated based on in-house QC limits, or 80-120%.

10.8 All standards made from a primary standard expire on or before the primary standard's expiration date.

10.9 Repeating pipettes used in this procedure are checked for accuracy by weighing 5 pipette volumes on the analytical balance with a percent error of less than 2% (0.45ul pipette±1%). If adjustments are necessary, the manufacturer's instructions are followed.

10.10 MDL studies are performed to establish the detection limit and quarterly LOD (MDL verification check) are run thereafter. See SOP No. 80.0005 for instructions concerning MDL studies. LOQ checks (distilled at RL) are also analyzed quarterly.

10.11 Deviations are approved/noted by the Department Supervisor for inclusion in the project narrative.

11. Data Validation and Reporting

Results are checked first by the analyst and then again by the supervisor. A project review worksheet is used to document the reviews. LCS and ICV recoveries are validated to ensure the distillation was acceptable. All data are uploaded to the LIMS and reported to the client on LIMS Level 2 forms. The Inorganic Laboratory Manager or the Wet Chemistry Supervisor reviews the data prior to release to the client.

12. Data Management and Records Management

12.1 Electronic data generated from the analysis of cyanide distillates (calibrations, QC, samples) is saved and managed per SOP 110.0029 Electronic Data Management.

12.2 All analysis information is documented in the individual Instrument Run/Injection Logbook regardless of run acceptance. No analyses are deleted from the sequence.

13. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP fail:

13.1 If the LCS is outside the control limits, the samples are to be re-prepped and then re-analyzed.

13.2 If the linear correlation of 0.995 is not established, or the ICV/ DoD high and low distilled standards do not meet ±15% criteria, check/perform the following:

13.2.1 Check standard S0 for possible contamination.

13.2.2 Check for proper integration in instrument.

13.2.3 Remake the standards and run a new calibration curve.

13.2.4 Remake the intermediate standard and run a new calibration curve.

13.2.5 To ensure reagents are not contaminated, run as samples.

13.3 Duplicates with RPD>20% may require re-distillation or re-analysis of the sample and duplicate if the sample and duplicate concentrations both exceed the MDL by a factor of 10. No action is required when the sample concentrations are less than 10 X MDL.

DoD QSM requires precision data in every batch therefore DUPs with ND results will not meet this requirement. For DoD projects always add an LCSD.

13.4 Matrix Spike recoveries outside the control limit requires a post-distillation spike. The sample is spiked at 2X the MRL (0.04mg/L) or 2 times the sample concentration, whichever is greater.

13.5 It is possible that there may be mechanical problems or software problems with the Lachat analysis instrument itself:

13.5.1 Check all pump tubing to assure that there are no clogs or flattening of the tubing itself. If the tubing is flattened, replace immediately. A green dye may be run through the lines to find any clogs. Change tubing as needed. Record all maintenance in the run logbook.

13.5.2 Check to make sure that the data is being properly integrated. Improper integration will cause the results to be skewed. See the Lachat Methods Manual for assistance in this area.

13.5.3 Make sure that all reagents have not passed their respective expiration dates.

13.5.4 Air bubbles in any of the lines or reagents will cause air spikes and poor peak integration.

13.5.5 There is a maintenance schedule in the Lachat Methods manual. It should be followed as closely as possible. This will ensure proper analysis and results.

13.6 Corrective Action reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for initiating a report for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

14. Health and Safety

Precautions to protect analysts include the nature of toxicity or carcinogenicity of analytes of reagents used in the method. Basic good housekeeping practices such as the wiping up of spills immediately and regular cleaning of counters and hoods will help reduce the potential for cross-contamination and create a safe working environment. Whenever possible, work under a well ventilated hood. Lab coats, gloves, and safety glasses must be worn at all times in the lab.

15. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 (Waste Management) and 20.0 (Definitions, Acronyms, and Abbreviations) of the current Quality Assurance Plan.

16. References

U. S. Environmental Protection Agency. Test Methods for Evaluating Solid Wastes, SW846 Method 9012B, Revision 2.0, November 2004.

Lachat Methods Manual Quick Chem Method 10-204-00-1-A, Revised June 1996.

Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2 Oct 2010 or current version.

Attachments:

1. **Figure 1**: Cyanide Distillation Logbook
2. **Figure 2**: Lachat Methods Manual Quick Chem 8000
3. **Figure 3**: Lachat Instrument Logbook

Figure 1
Cyanide Distillation Logbook

.

Spectrum Analytical, Inc. RI Div: CYANIDE DISTILLATION LOGBOOK

Method # :E335.4 / SW9012B (circle one)

Date:_____ **Time On:**_____ **Time Off:**_____ **Analyst:**_____

Place #	Lab ID		Sample Vol (ml) Weight (g)	Sample pH	Pb Ac Positive (Y/N)	O.1N NaAsO2 (ml)	KI Test (Y/N)	4N Sulfamic Acid (ml)	50% H2SO4 (ml)	2.5M MgCl2 (ml)	Final Volume (ml)
1											50
2											50
3											50
4											50
5											50
6											50
7											50
8											50
9											50
10											50
1											50
2											50
3											50
4											50
5											50
6											50
7											50
8											50
9											50
10											50

LCS ID: _____

Sulfamic Acid: _____ **$MgCl_2$:** _____ **LCS volume:**_____

Bismuth Nitrate: _____ **Spike ID:** _____

Na_2AsO_2: _____ **Cd. Carbonate:** _____ **Spike volume:**_____

ICV ID: _____

H_2SO_4: _____ **Temp:** _____

Reviewed By:_____

Logbook ID: 100.0169-04/13

Figure 2
Lachat Methods Manual Quick Chem 8000

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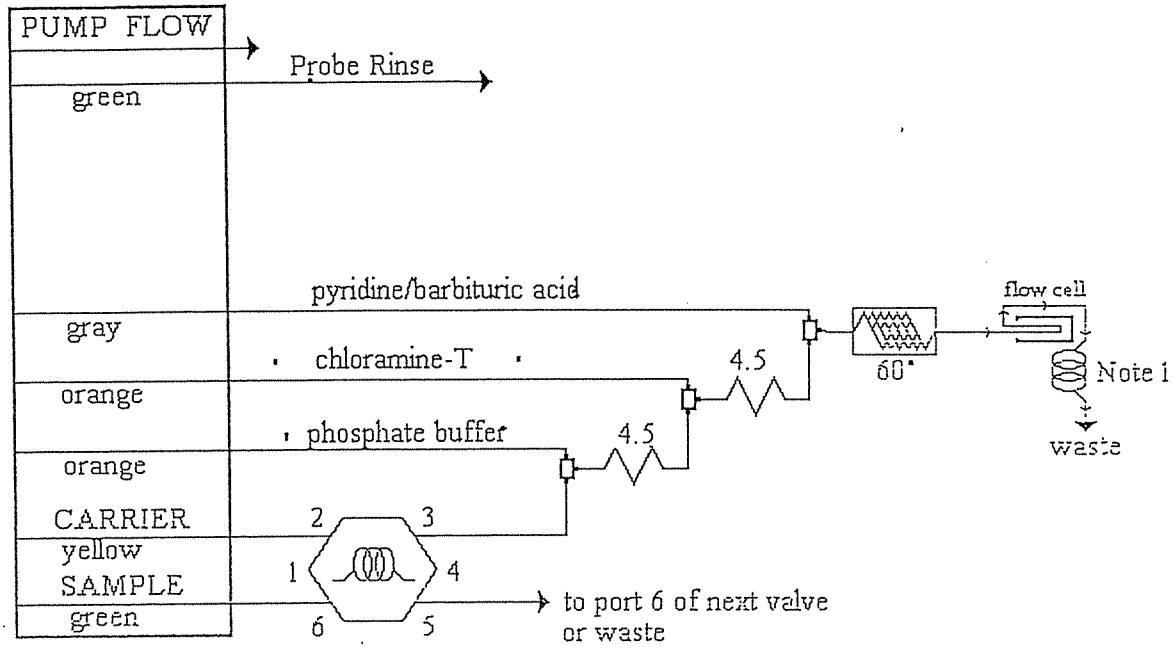
17. TABLE, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1. CYANIDE MANIFOLD DIAGRAM

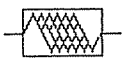


Sample loop = 150 cm x 0.8 mm i.d. per method Interference Filter = 570 nm
QC8000 Sample loop = 150 cm x 0.8 mm i.d.

CARRIER is 0.25 M sodium hydroxide solution (Reagent 1).

All manifold tubing is 0.8 mm (0.030 in) i.d. This is 5.2 uL/cm.

4.5 is 70 cm of tubing on a 4.5 cm coil support

APPARATUS: An injection valve, a 10 mm path length flow cell, and a colorimetric detector module are required. The  shows 650 cm of tubing wrapped around the heater block at the specified temperature.

Note 1: 2 meter back pressure loop, 0.52 mm i.d.

Figure 3
Lachat Instrument Logbook

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Spectrum Analytical, Inc. RI Division LACHAT RUN LOGBOOK (LAB ID: LACHAT1) Date: Analyst:

Method # (circle one) : E335.4 / SW9012B /ISM Analyses: Channel 1: Channel 2:

AS POS	Lab ID		AS POS	Lab ID		AS POS	Lab ID		AS POS	Lab ID		AS POS	Lab ID	
S1			12			32			52			72		
S2			13			33			53			73		
S3			14			34			54			74		
S4			15			35			55			75		
S5			16			36			56			76		
S6			17			37			57			77		
S7			18			38			58			78		
S8			19			39			59			79		
S9			20			40			60			80		
1			21			41			61			81		
2			22			42			62			82		
3			23			43			63			83		
4			24			44			64			84		
5			25			45			65			85		
6			26			46			66			86		
7			27			47			67			87		
8			28			48			68			88		
9			29			49			69			89		
10			30			50			70			90		
11			31			51			71			91		

*Report all results in mg/L

DATA FILE NAME _____

METHOD FILE NAME _____

TRAY FILE NAME _____

REPORT FILE NAME _____

Reagent Lots

Pyridine_____

NaOH_____

KH2PO4_____

Barbituric Acid_____

Chloramine-T_____

Other

_____

_____

_____

_____

_____

Curve on _____

m = _____

b = _____

r = _____

Logbook ID 100.0144-10/12

Reviewed by _____

Mercury Analysis in Aqueous and Soil Samples by Flow Injection Mercury System (FIMS) for Cold Vapor Atomic Analysis by SW846 Method 7470A/7471B

Contents SOP NO. 100.0012

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		6/15/10
Quality Assurance Director		6/15/10
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	Supervisor	12/6/11			
	Supervisor	01/14/13			

Revision Record

Revision Date	Revision Description	Comments	Initials
5/15/07	Added clarification to aq stds, serial dil for DoD.		SBL
2/27/08	Soil MS recovery, lab name change, method update to 7471B	Did not adopt the 0.5 to 0.6g suggested wt. Using 0.6 to 0.75g	SBL
11/4/08	0.5-0.6g required. Corrected temperature requirement.	Full revision	SBL
06/08/10	Removed serial dilution. No longer part of QSM4.1 requirement. QSM4.1 edits	Full revision	SBL
1/25/12	Added winlab software to instrumentation section	Minor	SBL

Procedure Superseded By _____Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURES

for

Mercury Analysis in Aqueous and Soil Samples by Flow Injection Mercury System (FIMS) for Cold Vapor Atomic Analysis

by

SW846 Method 7470A/7471B

Rev. 10

	Signature	Date
QA Director:	Thomas B Sawle	6/15/10
Lab Director:	[signature]	6/15/10

Effective Date: 6/22/10

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Mercury Analysis in Aqueous and Soil Samples by Flow Injection Analysis System for Cold Vapor Atomic Analysis

by

SW846 Method 7470Aand 7471B

Rev. 10

1. Scope and Application

This SOP describes the procedures applicable to the preparation and analysis of mercury in aqueous and soil samples. Matrices include ground waters, aqueous samples, soils, sediments, sludges, and TCLP. All samples require digestion prior to analysis.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

3.1 The aqueous samples are digested with concentrated HNO_3, concentrated H_2SO_4, potassium permanganate and potassium persulfate at 95°C. The procedure converts various organically bound compounds and inorganic forms

of mercury into mercuric ions, which can be analyzed with a Flow Injection Analysis System (FIAS) for atomic spectroscopy. The soil/sediment samples are digested using aqua regia and potassium permanganate at 95±3°C and analyzed same as the aqueous samples.

3.2 The mercury ions formed during the digestion step are reduced to the elemental state and aerated into an absorption cell. Absorbance is measured at 253.7nm and is a function of mercury concentration.

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For mercury analysis by SW 846 method 7470A water samples are collected in one-liter plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 All samples are stored at room temperature until analyzed.

4.3 Sample hold time for mercury analysis by SW 846 methods 7470A and 7471B is 28 days from date of sample collection.

5. Interferences and Potential Problems

5.1 Sulfides at levels above 20 mg/L or 20mg/Kg interfere. Potassium permanganate is added to the samples to eliminate possible interference of sulfides.

5.2 Copper at levels above 10 mg/L or 10mg/Kg interfere.

5.3 Seawaters, brines and industrial effluents high in chlorides interfere and require additional potassium permanganate for conversion to free chlorine. Free chlorine also absorbs radiation at 253.7nm. Therefore, the free chlorine is removed by addition of hydroxylamine sulfate reagent. In addition, the dead air space in the BOD bottle must be purged before adding the stannous sulfate.

5.4 Some volatile organic materials absorb at 253.7nm and may interfere.

6. Equipment and Apparatus

Equipment and instrumentation used in this analysis method include:

6.1 Equipment:

6.1.1 Perkin-Elmer FIMS 100 with WinLab32 software.

6.1.2 Printer.

6.1.3 Wheaton BOD bottles.

6.1.4 Top loading balance - capable of accurate measurement to 0.01gram.

6.1.5 Hot Plate with graphite block digester

6.2 Preventative Maintenance:

6.2.1 Pump tubing is replaced every 48 hours of instrument run time.

6.2.2 The windows of the optical cell are cleaned whenever the cell is replaced.

6.2.3 The inside of the optical cell is cleaned once every 48 hours of instrument run time.

6.3 Troubleshooting - Refer to the FIMS Analysis manual.

6.4 Glassware

6.4.1 100mL Class “A” volumetric flasks.

6.4.2 Class “A” volumetric pipettes ranging from 10µL to 1.0mL.

6.4.3 100 and 250mL Class “A” graduated cylinder or equivalent.

7. Reagents and Standards

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

7.1 Sulfuric Acid: Ultra Trace Grade

7.2 Nitric Acid: Ultra Trace Grade

7.3 Reagent water (ASTM Type I water). Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. Reagent water is also referred to as DI water.

7.4 Stannous chloride solution:
15 g $SnCl_2$ to 1000mLs of 3% HCl solution.

7.5 Sodium chloride-hydroxylamine sulfate solution:
12 g NaCl and 12g hydroxylamine sulfate to 100mLs of reagent water.

7.6 5% Potassium permanganate solution:
Dissolve 50g of $KMnO_4$ in 1000mLs of DI H_2O.

7.7 5% Potassium persulfate solution:
Dissolve 50g of $K_2S_2O_8$ in 1000mL of DI H_2O.

7.8 Stock mercury solution(Primary):
1000 mg/L purchased commercially.

7.9 Stock mercury solution(Independent Source):
1000 mg/L purchased commercially.

7.10 Mercury standards are stored in Wet Chem cupboards. Expiration dates for the stock standards are designated by the manufacturer. Prepared reagents and intermediate standards have a 28-day expiration period.

7.11 3% HCl: 30mL concentrated HCl diluted to 1L with DI H_2O.

7.12 HCl: conc., Ultra Trace grade.

7.13 Aqua Regia: prepare immediately before use by carefully adding three volumes of conc. HCl to one volume HNO_3.

8. Procedure

8.1 Preparation

8.1.1 Aqueous Samples: Transfer 100mL of sample or an aliquot diluted to 100mL to a reagent water-rinsed BOD bottle with a graduated cylinder. Add 5mL concentrated H_2SO_4 and 2.5mL concentrated HNO_3, mix. Add 15mL of potassium permanganate solution. Additional permanganate may be required until the purple color persists for at least 15 minutes. Add 8mL potassium persulfate solution; mix. Heat for 2 hours at 95°C on hot plate with graphite

holders. Cool samples and add 6mL sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. **CAUTION**: Do this addition under the hood, Cl_2 could be evolved. Record all volumes and reagents in the Mercury Digestion logbook, (**Attachment 1)**. Immediately prepare for analysis.

8.1.2 Soil Samples: Weigh a 0.50 – 0.60g portion of a well homogenized untreated sample and place in the bottom of a BOD bottle. See SOP 110.0039 for sub-sampling techniques. Add 5mL of reagent water and 5mL of aqua regia. Heat 2 minutes on hot plate with graphite holders at 95±3°C. Cool; then add 50mL reagent water and 15mL of the potassium permanganate solution to each sample bottle. Mix thoroughly and place on hot plate with graphite holders for 30 minutes at 95±3°C. Cool and add 6mL of the sodium chloride – hydroxylamine sulfate solution to reduce the excess permanganate. **CAUTION**: Do this addition under the hood, Cl_2 could be evolved. Add reagent water to a final volume of 100mL. Pour an aliquot into a polyethylene tube for analysis. Be careful to avoid pouring the sediment into the tube. Record all volumes and reagents in the Mercury Digestion logbook.

8.2. Calibration Standards

8.2.1 Working Calibration standards are prepared from a mercury intermediate standard at 500µg/L. The intermediate is prepared by pipetting 50µL of 1000mg/L Primary stock standard into a 100mL volumetric flask. Bring up to volume with 3% HCl.

Label standard as IIyymmdd#, where:

I I = Inorganic Intermediate
yy = year of preparation
mm = month of preparation
dd = day of preparation and
# = Sequential letter of intermediate standards prepped on this day.

All standard preparation information is documented in the Metals Standard Receipt/Preparation Logbooks (Primary, Intermediate and Working) or the LIMS.

Into BOD bottles containing approximately 10mL DI H_2O, pipet the following volumes of intermediate standard in order to achieve the corresponding final working standard concentrations:

Volume	Concentration (µg/L Hg)	Standard ID

40µL	0.2	S1
200µL	1.0	S2
400µL	2.0	S3
1mL	5.0	S4
2mL	10.0	S5
0mL	0.0	S0

8.2.2 Depending on the matrix of samples being prepared, follow the digestion as follows:

8.2.2.1 Soil Calibration standards, add 5mL of aqua regia. Heat 2 minutes on hot plate with graphite holders at 95±3°C. Cool; then add 50 mL reagent water and 15 mL of the potassium permanganate solution to each sample bottle. Mix thoroughly and place on hot plate with graphite holders for 30 minutes at 95±3°C. Cool, transfer to the Mercury Analysis Lab where 6mLs of the sodium chloride – hydroxylamine sulfate solution will be added to reduce the excess permanganate. **CAUTION**: Do this addition under the hood, Cl_2 could be evolved. Calibration working standards are prepped with each batch of samples and undergo a digestion of at least 30 minutes. Soil standards are brought up to 100mL final volume with DI H_2O prior to analysis.

8.2.2.2 Aqueous Calibration standards, add approximately 90 mL DI H_2O to each BOD bottle. Add 5mL concentrated H_2SO_4 and 2.5mL concentrated HNO_3, mix. Add 15mL of potassium permanganate solution. Additional permanganate may be required until the purple color persists for at least 15 minutes. Add 8mL potassium persulfate solution; mix. Heat for 2 hours at 95°C on hot plate with graphite holders. Cool, transfer to the Mercury Analysis Lab where 6mLs of the sodium chloride – hydroxylamine sulfate solution will be added to reduce the excess permanganate. Do not volumize to 100 mL with DI H_2O prior to analysis. **CAUTION**: do this addition under the hood, Cl_2 could be evolved

8.2.3 A Calibration Blank, ICB/CCB, is prepared as the S0 calibration standard.

Sample concentrations are not reported below the lowest non-zero calibration standard of 0.2ug/L (aqueous samples, or 0.033 mg/Kg for soils) without method modification. This effectively becomes the method Reporting Limit

(RL) also referred to as Project Quantitation Limits (PQL). DoD QSM refers to this limit as the Limit of Quantitation (LOQ).

8.2.4 ICV/CCV Intermediate standards are prepared at 5.0µg/L from an **Independent Source** Hg Primary stock standard. The intermediate Hg CCV standard is prepared by pipetting 50µL of 1000mg/L Hg into a 100mL volumetric flask. The standard is brought to volume with 3%HCl for a final concentration of 500ug/L.

8.2.5 The working ICV/CCV is prepared by pipetting 1mL of the above intermediate standard into a BOD bottle and digesting as in **section 8.2.2.** An ICV/CCV is prepped with each batch of samples. Final concentration is 5µg/L and the standard is labeled as a mercury working calibration standard:

Label standard as IWyymmdd#, where:

I W = Inorganic Working
yy = year of preparation
mm = month of preparation
dd = day of preparation and
# = Sequential letter of intermediate standards prepped on this day.

8.2.6 LCS/Matrix spike standard is also an intermediate concentration standard that is prepared by combining 45.5 uL of the **Independent Source** Hg Primary stock standard into a 100 mL volumetric flask and volumized to 100 mL with 3% HCl. The standard is labeled as for Hg working standards, as in **section 8.3**.

8.2.7 1mL of the above LCS/Matrix spike is added into 100 mL of DI H_2O and digested as an aqueous sample for the LCS-Water (LSCW) as in **section 8.1.1** . The true value of the LCS = 4.55µg/L at the instrument level.

8.2.8 1mL of the LCS/spike working spike is added to 0.6 g of acid-washed Teflon chips used to simulate a soil matrix for the LCS-Soil (LCSS). The true value of the LCS = 4.55µg/L at the instrument level.

8.2.9 1mL of the LCS/spike working spike is added to a chosen sample, and digested as per the sample matrix. This is the Matrix Spike. At the time of analysis, the spike = 4.55µg/L at the instrument level, times any sample dilution factor.

8.3 Instrumental Analysis

8.3.1 Adjust the argon pressure to 50 psi for FIMS analysis. Turn on the FIMS system with the auto-sampler. Turn on the computer, printer. Allow the lamp to stabilize for 35-45 minutes.

8.3.2 Prepare the reductant solution, stannous chloride in **section 7.4**, and the carrier solution, 3% HCl in **section 7.11**.

8.3.3 Bring up the appropriate element file (Hg comm).

8.3.4 Set up the sample info file to coincide with the locations and sample identifications that will be analyzed in the run.

8.3.5 Fill out the Automated Control Window:

8.3.5.1 Type in a data file name.

8.3.5.2 Type in the name of the sample info file.

8.3.6 Load the auto-sampler tray.

8.3.7 Place the carrier tubing inlet into the carrier solution, and the reductant tubing inlet in the reductant solution. The reductant solution tubing has a red tab on it, and the carrier solution a yellow tab. Remove the cap from the gas chamber and start the pump to ensure it is working properly. Turn off the pump and replace the cap.

8.3.8 Hg lamp intensity, measured as absorbance for the 10ug/L mercury standard, is recorded daily in the instrument run logbook.

8.3.9 On the AS-90 control window either choose “Run All” to run both standards and samples or “Calibrate” to run only the standards. If you choose only to calibrate at this time, you will need to click on “Reset Sampler” and “Run Samples” when calibration is complete. Analyze standards and samples. Samples that exceed the upper calibration range must be diluted and reanalyzed.

8.3.10 When analysis is complete place both carrier tubing inlet and the reductant tubing inlet with the auto-sampler probe in a beaker of DI water. Allow the water to pump through the system. Continue to flush all the water through until no more bubbles appear in the waste tubing. Turn off the pump.

8.3.11 Turn off the FIMS unit.

8.3.12 No rinse between samples is necessary per manufacturer's instructions.

8.3.13 The following Analytical Sequence should be used.

1. Calibration Blank(S0)
2. Standard #1 (S1)
3. Standard #2 (S2)
4. Standard #3 (S3)
5. Standard #4 (S4)
6. Standard #5 (S5)
7. ICV
8. ICB
9. Method blank
10. LCS
11. Samples (≤6)
12. CCV
13. CCB
14. MS
15. *S*ample Duplicate
16. Samples (<5)
17. CCV
18. CCB

8.3.14 All information including the analytical sequence is documented in the FIMS 100 Run Logbook, **Attachment 2.**

9. Data Reduction and Calculations

9.1 Sample data should be reported in units of ug/L for aqueous samples, and ug/Kg dry weight for soils/solids. Results are reported to two significant figures.

9.1.1 For aqueous results, report the data generated directly from the instrument with allowance for sample dilution. Upload the data from the FIMS directly into the Omega LIMS system for reporting.

9.1.2 For soil/solid samples, upload the data as for aqueous samples. The LIMS will calculate the final results in soil units, with allowance for dilution, sample weight and percent moisture. Make sure the Pmoist data is available.

9.2 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

% Recovery (%R) = 100 x (SSR-SR)/ (SA)

where: SSR = spiked sample result
SR = sample concentration
SA = spike added

9.3 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

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

where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

10 Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results.

10.2 Linear correlation for the standard curve must be **≥0.995**. The Calibration curve must not be forced to go through the origin zero point.

10.3 Method blanks - A preparation blank is prepped and analyzed with every batch not to exceed 20 samples. Method Blanks must not contain Mercury at a concentration ≥ the MRL. If mercury is present in the Method Blank and the lowest sample concentration in the batch exceeds 10 times the blank concentration, no corrective action is needed. Otherwise, corrective action for method blank contamination involves determining the source of the contamination and re-prepping the entire batch.

DoD QSM - Method blank concentrations must be less than 1/2 RL.

10.4 Calibration verification – A second source ICV prepped with the associated samples is analyzed immediately after the curve and must be within 90-110% of its true value. The ICV concentration is at the mid-level of the calibration curve.

10.5 CCVs are analyzed at least every 10 samples with 80-120% recovery requirements.

10.6 An ICB/CCB is run immediately after the ICV/CCV set; the mercury value in the ICB/CCB is not to exceed the MRL.

DoD QSM– Mercury is not to exceed the LOD in the ICB/CCB.

10.7 A matrix spike and a matrix duplicate are prepped and analyzed with every batch not to exceed 20 samples. The RPD for duplicates is 20% and the aqueous spike recovery control limit is 75-125%. The soil spike recovery control limit is 80-120%.

DoD QSM – Spike recoveries must be within 80-120% for both water and soil

10.8 Laboratory Control Sample (LCS) -An LCS is prepped with a minimum of every 20 samples of the same matrix. Control limits are 80-120% of the true value for mercury. If the LCS is outside the acceptance limits, the corresponding samples are re-prepped and reanalyzed. Corrective action includes re-digestion/reanalysis for all samples and QC in the batch.

10.9 Sample concentrations that exceed the highest calibration are diluted and rerun so that their concentration falls within the calibration range.

10.10 The Inorganic Laboratory Supervisor/Manager authorizes any method deviations.

10.11 The intermediate standard is stable for 28 days. All standards made from a primary standard expire on or before the primary standard's expiration date.

10.12 Method detection limits (MDLs) are established when the instrument is set up or when there is significant instrument maintenance performed that would affect its sensitivity. The MDL is obtained by multiplying the standard deviation of seven or eight analyses by the appropriate one-sided 99% t-statistic. The value of this statistic equals 3.143 if the number of analyses is seven. An MDL verification check is performed immediately following MDL study and quarterly each year in lieu of the annual MDL study. An MDL verification check sample is spiked at approximately 2-3 times the current MDL.

DoD QSM – The MDL verification check sample's concentration sets the LOD.

10.13 Hg lamp intensity, measured as absorbance for the 10ug/L mercury standard, is recorded daily in the maintenance logbook. Trends are to be monitored to show the instrument is within the optimal absorbance range. The normal/optimal absorbance range is from 0.17-0.24.

11. Data Validation and Reporting

11.1 Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the Supervisor/Lab Manager. The Supervisor/Lab Manager reviews 100% of the data prior to report generation. The QA Director randomly reviews 10% of the data reported by the laboratory. After each review, the appropriate section of the Data Review Checklist is checked off, **Attachment 3**.

11.2 Reports are generated by the data reporting group. The data submitted for report preparation is dependent on project requirements.

11.3 Data is always reported to the RL. If clients require reporting limits different from the RL/PQL, the data will need to be reported using a special form set through the Omega LIMS system depending on project needs.

11.4 Electronic files (EDD) are generated by the Data Management Department and are stored in the server until downloaded to tapes by the server backup system.

12. Corrective Action Procedures

12.1 Corrective Action to be implemented in the event QC results are outside of the acceptance range is covered in **section 10**.

12.2 Corrective Action reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a corrective action report for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Health and Safety Manual. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

13.2 Concentrated nitric, sulfuric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.

13.3 The cell-heating compartment maintains a temperature of 100°C throughout the analysis. Care should be taken to avoid burns from the cell-heating compartment.

13.4 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. U.S. Environmental Protection Agency: Test Methods for Evaluating Solid Waste, Update IIB, SW-846 Method 7470A and Update IV SW-846 Method 7471B.
2. Department of Defense, Quality Manual for Environmental Laboratories. Final Version 4.1, 4/22/09, or current version.

Attachments:

Attachment 1: Mercury Digestion Logbook
Attachment 2: FIMS Instrument Logbook
Attachment 3: Data Review Checklist Form

Attachment 1
Mercury Digestion Logbook

MITKEM LABORATORIES

Mercury Digestion Logbook

					Reagents Added					Analyst:		
Date	Bottle No.	Sample ID		Initial pH	Sample Vol (ml)/ Wt (g)	Conc. H_2SO_4 (ml)	Conc. HNO_3 (ml)	5% $KMnO_4$ (ml)	5% $K_2S_2O_8$ (ml)	Aqua-regia (ml)	Final Volume (ml)	Comments

Waters
In: _____
Out: _____
Matrix: Aqueous
Method # _____

Soils
In: _____ Out: _____
In: _____ Out: _____
Matrix: Soil/Solid

Hotblock ID: _____ Temp: _____ °C/ MT-_____
LCSS ID _____ H_2SO_4 Lot # _____
LCSS volume: _____ HNO_3 Lot # _____
Spike ID _____ HCl Lot # _____
Spike volume: _____ KMnO4 ID # _____
K2S2O8 ID# _____

Digestate Relinquished to: _____ Reviewed by: _____

Attachment 2
FIMS Instrument Logbook

MITKEM LABORATORIES: FIMS 100 RUN LOGBOOK

Date: **Analyst:**

Analysis:			Analysis:			Analysis:			Analysis:		
SEQ#	Bottle ID	LAB ID	SEQ#	Bottle ID	LAB ID	SEQ#	Bottle ID	LAB ID	SEQ#	Bottle ID	LAB ID
1			26			51			76		
2			27			52			77		
3			28			53			78		
4			29			54			79		
5			30			55			80		
6			31			56			81		
7			32			57			82		
8			33			58			83		
9			34			59			84		
10			35			60			85		
11			36			61			86		
12			37			62			87		
13			38			63			88		
14			39			64			89		
15			40			65			90		
16			41			66			91		
17			42			67			92		
18			43			68			93		
19			44			69			94		
20			45			70			95		
21			46			71			96		
22			47			72			97		
23			48			73			98		
24			49			74			99		
25			50			75			100		

Calibration/ICV Prep Date:_____ $SnCl_2$ _____ HCl: _____

Reviewed By: _____ NaCl: _____ Hydroxylamine Sulfate: _____

Logbook ID: 100.0164-06/10

Attachment 3
Data Review Checklist Form

Spectrum Analytical, Inc. Rhode Island Division

Hard Copy Due:_____

Fax Copy Due:_____

CLP/CLP-like Deliverable Check List for Inorganic Analysis

Project Number:_____
Client._____
Input by/date:_____
Forms generated on/date:_____

Analysis:_____
Category:_____
Reviewer:_____
(1) Date Reviewed:_____
(2) Date Reviewed:_____
Corrections by:_____

Elements Required:

Al	Sb	As	Ba	Be	Cd	Ca	Cr	Co	Cu	Fe	Pb	Mg	Mn	Ni	K	Se	Ag	Na	Tl	V	Zn	Sn	B	Mo	CN	Hg

Items:	Pages	Check	OK/Unusual Observation
Sample Log-In Sheet	_____	_____	_____
Prep Log Sheet (AQ/SL)	_____	_____	_____
% Solid Bench Sheet	_____	_____	_____
Tumbling Log (TCLP/SPLP)	_____	_____	_____
Filter Log	_____	_____	_____

	Check	Lab ID	OK/Unusual Observation/Deviation/Flags
ICV / CCV	_____	_____	_____
Matrix Spike (N)	_____	_____	_____
Duplicate Samples (*)	_____	_____	_____
Serial Dilutions (E)	_____	_____	_____
Post Digestion Spike	_____	_____	_____
LCS	_____	_____	_____
ICP Interference	_____	_____	_____
Method Blank	_____	_____	_____

Prep/Analysis Notes:

	Yes	No
Client ID Check:	_____	_____
ID Truncation:	_____	_____
Special Request:	_____	_____

Preparation of Soil Samples by Acid Digestion By SW846 Method 3050B for Analysis by ICP/MS

Contents SOP NO. 100.0100

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		3/29/10
Quality Assurance Director		3/29/10
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	Dept manager	3/7/11			
	supervisor	8/8/12			

Revision Record

Revision Date	Revision Description	Comments	Initials

Procedure Superseded By _____ **Date:** _____
Procedure Discontinued By: _____ **Date:** _____
Procedure Archived By: _____ **Date:** _____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Preparation of Soil Samples by Acid Digestion By SW846 Method 3050B for Analysis by ICP/MS

SOP No. 100.0100

Rev. 0

	Signature	Date
QA Director:		3/29/10
Lab Director:		3/29/10
Effective Date:	4/05/10	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Preparation of Soil Samples by Acid Digestion By SW846 Method 3050B for Analysis by ICP/MS

Rev. 0

1. Scope and Application

This Standard Operating Procedure (SOP) deals with the preparation of soil samples utilizing USEPA SW846 Method SW3050B for analysis by ICP/MS Method 6020A. Discussion includes sample digestion and sample concentration technique for the analysis of metals in soil samples.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. Supervisors/lab managers are responsible for ensuring that this SOP is accurate and up to date and that it is implemented appropriately. Supervisors/lab managers review logbooks and data generated for this procedure and approve all reported results.

3. Summary of Procedure

A 1.00 to 2.00 gram soil/solid sample is digested with the addition of acids and hydrogen peroxide for metals analysis by Inductively Coupled Argon Plasma (ICAP). This method has been adapted to utilize polyethylene digestion tubes rather than the 250mL bottles noted in the original method. The reagent volumes have been reduced to accommodate the method as well.

4. Sample Preservation, Containers, Handling, and Storage

4.1 Hold time for ICP analysis is 180 days from date collected.

4.2 Samples are stored at 4°C in amber glass jars with Teflon lined caps.

5. Interferences and Potential Problems

Possible sources of contamination:

5.1 Hood fall-out.

5.2 Acid bath for glassware.

5.3 Acid dispensers.

5.4 Sample matrix effects: Extreme organic samples.

5.5 Disodium Stannate preservative for peroxide: Do not use this peroxide when analyzing tin in soils unless proven not to contribute significant tin by analysis of associated Method Blanks. If method blanks show significant tin, an alternative peroxide source must be used.

6. Equipment and Apparatus

Equipment used in this preparation method include:

6.1 10% HNO_3 acid bath.

6.2 50 mL graduated polyethylene tubes.

6.3 125 mL HDPE digestate containers.

6.4 Watch Glasses.

6.5 Hot plates.

6.5 Centrifuge.

6.6 Balance for weighing out sample (calibrated daily prior to use).

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

7.1 Concentrated HNO_3, ACS trace metals grade.

7.1.1 1:1 (v/v) HNO_3, ACS trace metals grade.

7.2 Concentrated HCl, ACS trace metals grade.

7.3 30% Hydrogen peroxide (H_2O_2), ACS certified.

7.4 Spiking solutions, High Purity Standards.

7.5 Teflon chips, Chemware Ultra PTFE Boiling Stones.

8. Procedure

See SOP 110.0039 for sub-sampling procedures.

8.1 Digestion of soil samples for ICP/MS analysis:

8.1.1 Note in Soil/Solid Sample Preparation Logbook the presence of any artifacts (see **Figure 1**).

8.1.2 Mix the sample thoroughly so that a homogenous representative aliquot can be taken. Weigh an approximate1.00 to 2.00 gram (wet weight) or 1.00 gram (dry weight) aliquot of the sample to the nearest 0.01g and transfer it to a 50 mL digestion tube.

8.1.3 Add 5 mL of 1:1 (v/v) HNO_3, mix well. Cover the tube with a ribbed watch glass. Heat to 95°C ± 5 °C in graphite holders on hotplates in the hood, and reflux for 10 minutes without boiling.

8.1.4 Remove from hotplate and allow to cool.

8.1.5 Add 2.5mL of conc. HNO_3. Replace on hotplate and reflux for 30 minutes. If brown fumes are generated at this step repeat the addition of 2.5mL conc. HNO_3 with heating steps until no more brown fumes are given off. Do not allow the volume to fall below 5mL.

8.1.6 Allow the sample to cool and add 1mL of DI water and 1.5 mL of 30% H_20_2. Return the tube to hotplate to start peroxide reaction, making sure no losses occur due to excessive effervescence action. Heat until effervescence subsides and then allow tube to cool

8.1.7 Continue adding 30% H_2O_2 in 0.5mL aliquots with warming until minimal effervescence occurs, the appearance of the sample remains unchanged, or 5mL have been added. If the blank or a sample no

longer has effervescence, set them aside, do not continue to add H_2O_2. When all are done proceed to next step.

Note: do not add more than a total of 5mL H_2O_2

8.1.8 Take tubes off the hotplate and cool to room temp.

8.1.9 Prepare the batch of sample digestate containers by first measuring 100mL of DI Water into a Class A volumetric cylinder. Pour this volume into each of the clean containers and mark the 100mL line. Once marked and labeled the sample digestates may be transferred to the containers.

8.1.10 Volumize to 100mL with DI Water.

8.1.11 Centrifuge sample for approximately 5 minutes at 3000 rpm to settle any insoluble material.

8.1.12 All digestion information is documented in the Soil/Solid Sample Preparation Logbook (**figure 1**). Transfer the digestates to Inorganic Instrument Lab.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are established to ensure generation of data of known quality. QA/QC procedures associated with the Inorganic prep lab include preparation of Method Blanks, Lab Control Samples, matrix spikes and sample duplicates.

10.1 Preparation Blanks:

A method blank must be run with every discrete batch of samples that is being prepped. A batch of samples cannot exceed 20 samples.

10.1.1 The method blank includes all reagents used to prepare samples and is treated as if it were a regular sample. Label appropriately.

10.2 Laboratory Control Samples:

A laboratory control sample containing all target analytes must be prepped and analyzed for each batch of samples. The number of samples per batch cannot exceed 20.

10.2.1 For the LCS, measure 1.00-2.00g of the metals SRM (manufactured by Environmental Resource Associates) into a 50 mL digestion tube. Digest in the same manner as all other samples. Record the SRM Standard ID in the preparation logbook.

10.3 Matrix Spikes and Duplicate Samples:

Prepare 3 digestion tubes to be used for the same aqueous sample. Designate one tube as the sample, one as the duplicate of that sample, and the other as a spiked portion of the sample. Into each tube, measure 1.00-2.00 g (wet weight) or 1 gram (dry weight) of sample (the same sample).

10.3.1 Matrix Spikes:

- To one aliquot add the following spiking standards containing all target analytes: Spike 0.5 mL of an intermediate standard containing Mg, Ca, Na, and K, 50 µL of an intermediate standard containing Sb, As, Cd, Pb, Se, and Tl, 50 µL of CLP Spike 1A and CLP Spike 1B into the tube. Label this sample with an "MS" suffix, and digest as described in **Section 8**.

10.3.2 Duplicates: Label the tube designated for the sample duplicate with a "D" suffix and digest as described in **Section 8**.

10.4 Digestion Tubes and Pipettes.

10.4.1 All lots of digestion tubes must be tested for conformance. A group of 10 tubes are measured for volume by weight. The weights are recorded and the average weight is calculated. See SOP 80.0030 Labware Volume Verification for acceptance criteria.

10.4.2 Pipettes: Two sizes of pipettes are used in the method; 0 to 1000 µL and 0 to 100 µL. Five replicate weights are recorded. The average of the five replicates is calculated. See SOP 80.0030 Labware Volume Verification for acceptance criteria.

10.5 Standard Preparation

All standards made from a primary standard expire on or before the primary standard's expiration date.

11. Data Validation and Reporting

Data (logbook entries) generated in the inorganic preparation laboratory will be reviewed and signed by a peer, the supervisor or the department manager.

12. Corrective Action Procedures

All corrective action will stem from the analytical results in the Metals Laboratory. See the specific SOP for details on QC requirements.

12.1 If any method blank shows contamination, associated samples are scheduled for reprep by the department supervisor or manager.

12.2 If any spike recovery or duplicate RPD is outside of control limits, samples are scheduled for reprep by the department supervisor or manager.

12.3 If a Spike sample was not spiked, it will be re-prepped and re-analyzed with the appropriate sample and duplicate.

13. Health and Safety

Health and safety hazards in the Inorganic Preparation Laboratory (prep lab) include exposure to concentrated acids, their fumes and toxic metals standards. Labcoats, gloves and safety glasses must be worn in the prep lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

U. S. Environmental Protection Agency. Test Methods for Evaluating Solid Waste, SW-846, Update III, Revision 2, December 1996, Method 3050B.

Attachments:

Figure 1: Soil/Solid Sample Preparation Logbook

Figure 1

Soil/Solid Sample Preparation Logbook

Start time: **MITKEM LABORATORIES: Soil/Solid Metals Prep Logbook**

Date	Sample ID	Sample Wt. (g)	Sample Color Before	Sample Texture	Artifiacts (Y/N)	1:1 HNO_3 (ml)	Conc. HNO_3 (ml)	30% H_2O_2 (ml)	Conc. HCl (ml)	Sample Color After	Sample Clarity After	Final Volume (ml)	Comments

HCl Lot# _____

HNO3 Lot# _____

H2O2 Lot# _____

Analyst:_____

Method#:_____

Hotblock ID: _____

Digestion Temp:_____oC/MT-____

LCS/Spike Lot # _____

_____

_____

Digestate Relinquished to:_____

Reviewed By: _____

Narrative Notes on Reverse Side

Yes / No

Logbook ID 100.0124-xx/10

Preparation of Soil Samples by Acid Digestion By SW846 Method 3050B for Analysis by ICP/AES

Contents SOP NO. 100.0104

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		3/29/10
Quality Assurance Director		3/29/10
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	Dept. manager	3/7/11			
	supervisor	8/8/12			

Revision Record

Revision Date	Revision Description	Comments	Initials
5/15/07	Volume edits of some reagents	Per method 3050	SBL
3/16/08	Clarify LCS/MS contains all elements, lab name change.		SBL
6/23/08	Edited LCS/MS spike volume		SBL
11/28/08	Added warning about peroxide preservative		SBL
3/4/09	Added ICP/MS info		SBL
03/16/10	New revision with updated spiking details, new title	Removed ICP/MS	SBL

Procedure Superseded By _____ **Date:** _____
Procedure Discontinued By: _____ **Date:** _____
Procedure Archived By: _____ **Date:** _____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Preparation of Soil Samples by Acid Digestion
for ICP /AES Analysis

by

SW846 Method 3050B

SOP No. 100.0104

Rev. 8

	Signature	Date
QA Director:	Thomas B Faulk	3/29/10
Lab Director:		3/29/10
Effective Date:	4/05/10	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Preparation of Soil Samples by Acid Digestion
for ICP /AES Analysis

by

SW846 Method 3050B

Rev. 8

1. Scope and Application

This Standard Operating Procedure (SOP) deals with the preparation of soil samples utilizing USEPA SW846 Method SW3050B for analysis by ICP/AES Method 6010. Discussion includes sample digestion and sample concentration technique for the analysis of metals in soil samples.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. Supervisors/lab managers are responsible for ensuring that this SOP is accurate and up to date and that it is implemented appropriately. Supervisors/lab managers review logbooks and data generated for this procedure and approve all reported results.

3. Summary of Procedure

A 1.00 to 2.00 gram soil/solid sample is digested with the addition of acids and hydrogen peroxide for metals analysis by Inductively Coupled Argon Plasma (ICAP). This method has been adapted to utilize polyethylene digestion tubes and a final 50mL digestate volume, rather than the 100mL volume noted in the original method. The reagent volumes have been reduced to accommodate the method as well.

4. Sample Preservation, Containers, Handling, and Storage

4.1 Hold time for ICP analysis is 180 days from date collected.

4.2 Samples are stored at 4°C in amber glass jars with Teflon lined caps.

5. Interferences and Potential Problems

Possible sources of contamination:

5.1 Hood fall-out.

5.2 Acid bath for glassware.

5.3 Acid dispensers.

5.4 Sample matrix effects: Extreme organic samples.

5.5 Disodium Stannate preservative for peroxide: Do not use this peroxide when analyzing tin in soils unless proven not to contribute significant tin by analysis of associated Method Blanks. If method blanks show significant tin, an alternative peroxide source must be used.

6. Equipment and Apparatus

Equipment used in this preparation method include:

6.1 10% HNO_3 acid bath.

6.2 50 mL graduated polyethylene tubes.

6.3 Watch Glasses.

6.4 Hot plates.

6.5 Centrifuge.

6.6 Balance for weighing out sample (calibrated daily prior to use).

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

7.1 Concentrated HNO_3, ACS trace metals grade.

7.1.1 1:1 (v/v) HNO_3, ACS trace metals grade.

7.2 Concentrated HCl, ACS trace metals grade.

7.3 30% Hydrogen peroxide (H_2O_2), ACS certified.

7.4 Spiking solutions, High Purity Standards.

7.5 Teflon chips, Chemware Ultra PTFE Boiling Stones.

8. Procedure

See SOP 110.0039 for sub-sampling procedures.

8.1 Digestion of soil samples for ICP/AES analysis:

8.1.1 Note in Soil/Solid Sample Preparation Logbook the presence of any artifacts (see **Figure 1**).

8.1.2 Mix the sample thoroughly so that a homogenous representative aliquot can be taken. Weigh an approximate1.00 to 2.00 gram (wet weight) or 1.00 gram (dry weight) aliquot of the sample to the nearest 0.01g and transfer it to a 50 mL digestion tube.

8.2.1 Add 5 mL of 1:1 (v/v) HNO_3, mix well. Cover the tube with a ribbed watch glass. Heat to 95°C ± 5 °C in graphite holders on hotplates in the hood, and reflux for 10 minutes without boiling.

8.2.2 Remove from hotplate and allow to cool.

8.2.3 Add 2.5mL of conc. HNO_3. Replace on hotplate and reflux for 30 minutes. If brown fumes are generated at this step repeat the addition of 2.5mL conc. HNO_3 with heating steps until no more brown fumes are given off. Do not allow the volume to fall below 5mL.

8.2.4 Allow the sample to cool and add 1mL of DI water and 1.5 mL of 30% H_20_2. Return the tube to hotplate to start peroxide reaction, making sure no losses occur due to excessive effervescence action. Heat until effervescence subsides and then allow tube to cool

8.2.1 Continue adding 30% H_2O_2 in 0.5mL aliquots with warming until minimal effervescence occurs, the appearance of the sample remains unchanged, or 5mL have been added. If the blank or a sample no longer has effervescence, set them aside, do not continue to add H_2O_2. When all are done proceed to next step.

Note: do not add more than a total of 5mL H_2O_2

8.2.1 ***For ICP/AES samples ONLY***: Add 5mL of concentrated HCl and return to the hotplate and reflux for an additional 15 minutes.

8.2.2 Take tube off the hotplate and cool to room temp.

8.2.3 Volumize to 50mL with DI Water.

8.2.4 Centrifuge sample for approximately 5 minutes at 3000 rpm to settle any insoluble material.

8.2.5 Label the tube properly. All digestion information is documented in the Soil/Solid Sample Preparation Logbook (**figure 1**). Transfer the digestates to Inorganic Instrument Lab.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are established to ensure generation of data of known quality. QA/QC procedures associated with the Inorganic prep lab include preparation of Method Blanks, Lab Control Samples, matrix spikes and sample duplicates.

10.1 Preparation Blanks:

A method blank must be run with every discrete batch of samples that is being prepped. A batch of samples cannot exceed 20 samples.

10.1.1 The method blank includes all reagents used to prepare samples and is treated as if it were a regular sample. Label appropriately.

10.2 Laboratory Control Samples:

A laboratory control sample containing all target analytes must be prepped and analyzed for each batch of samples. The number of samples per batch cannot exceed 20.

10.2.1 Measure 1.0 ±.01 g acid washed Teflon chips into a 50mL -digestion tube. Spike 455 uL of CV-1 and 45.5 uL each of CV-2 and CV-3 into the tube and digest as described in **Section 8**. Label appropriately. .

10.3 Matrix Spikes and Duplicate Samples:

Prepare 3 digestion tubes to be used for the same aqueous sample. Designate one tube as the sample, one as the duplicate of that sample, and the other as a spiked portion of the sample. Into each tube, measure 1.00-2.00 g (wet weight) or 1 gram (dry weight) of sample (the same sample).

10.3.1 Matrix Spikes:

- To one aliquot add three spiking standards containing all target analytes; High Purity Standard CLP-CV-1 at 455 uL; CLP-CV-2 and CLP-CV-3 at 45.5 uL each. Label this sample with an "MS" suffix, and digest as described in **Section 8**.

10.3.2 Duplicates:

- Label the tube designated for the sample duplicate with a "D" suffix and digest as described in **Section 8**.

10.4 Standard Preparation

All standards made from a primary standard expire on or before the primary standard's expiration date.

10.5 Digestion Tubes and Pipettes

10.5.1 All lots of digestion tubes must be tested for conformance. A group of 10 tubes are measured for volume by weight. The weights are recorded and the average weight is calculated. See SOP 80.0030 Labware Volume Verification for acceptance criteria.

10.5.2 Pipettes: Two sizes of pipettes are used in the method; 0 to 1000 µL and 0 to 100 µL. Five replicate weights are recorded. The average of the five replicates is calculated. See SOP 80.0030 Labware Volume Verification for acceptance criteria.

11. Data Validation and Reporting

Data (logbook entries) generated in the inorganic preparation laboratory will be reviewed and signed by a peer, the supervisor or the department manager.

12. Corrective Action Procedures

All corrective action will stem from the analytical results in the Metals Laboratory. See the specific SOP for details on QC requirements.

12.1 If any method blank shows contamination, associated samples are scheduled for reprep by the department supervisor or manager.

12.2 If any spike recovery or duplicate RPD is outside of control limits, samples are scheduled for reprep by the department supervisor or manager.

12.3 If a Spike sample was not spiked, it will be re-prepped and re-analyzed with the appropriate sample and duplicate.

13. Health and Safety

Health and safety hazards in the Inorganic Preparation Laboratory (prep lab) include exposure to concentrated acids, their fumes and toxic metals standards. Labcoats, gloves and safety glasses must be worn in the prep lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

U. S. Environmental Protection Agency. Test Methods for Evaluating Solid Waste, SW-846, Update III, Revision 2, December 1996, Method 3050B.

Attachments:

Figure 1: Soil/Solid Sample Preparation Logbook

Figure 1

Soil/Solid Sample Preparation Logbook

Start time: **MITKEM LABORATORIES: Soil/Solid Metals Prep Logbook**

Date	Sample ID		Sample Wt. (g)	Sample Color Before	Sample Texture	Artifiacts (Y/N)	1:1 HNO_3 (ml)	Conc. HNO_3 (ml)	30% H_2O_2 (ml)	Conc. HCl (ml)	Sample Color After	Sample Clarity After	Final Volume (ml)	Comments

HCl Lot# _____ **Analyst:**_____ **Digestion Temp:**_____**oC/MT-**____

HNO3 Lot# _____ **Method#:**_____ **LCS/Spike Lot #** _____

H2O2 Lot# _____ **Hotblock ID:** _____ _____

_____

Digestate Relinquished to:_____ **Reviewed By:** _____ **Narrative Notes on Reverse Side**

Yes / No

Logbook ID 100.0124-xx/10

Determination of Metals in Water and Soils by SW-846 Method 6020A Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

Contents SOP NO. 100.0110

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		4/22/10
Quality Assurance Director		4/20/10
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	Supervisor	8/19/10			
	QAD	10/21/11			
	Supervisor	12/6/11			
	Supervisor	01/14/13			

Revision Record

Revision Date	Revision Description	Comments	Initials
11/29/09	DOD QSM V.4.1		SBL
2/11/10	Misc revisions	New values in Table 1	TS
03/29/10	Raw data codes, SRM limits	Minor edit	SBL
04/16/10	Addition of data qualifier explanation on raw data	Major rev since a lot of new info was already on document	SBL
07/20/10	Table 1 LLICV edits, correction to section 10.2	Minor edit (LIMS correct), frequency noted for LRA check	TS/SBL
10/21/11	Table 1 ICV/LCS/MS correction for B and Mo.	Minor edits	SBL
12/13/11	Table 2 Cal Std conc edit for copper PQL	Minor edit	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By: _____ Date: _____
Procedure Archived By: _____ Date: _____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

For

Determination of Metals in Water and Soils by SW-846 Method 6020A Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

SOP No.100.0110
Rev. 2

	Signature	Date
QA Director:		4/22/10
Lab Director:		4/20/10
Effective Date:	4/29/10	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

For

Determination of Metals in Water and Soils by SW-846 Method 6020A

Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

SOP No.100.0110
Rev. 2

1. Scope and Application

This SOP describes the procedures applicable to the analysis of elements listed on page 1 of SW-846 Method 6020A. ICP technology was built upon the same principles used in atomic emission spectrometry. Samples are decomposed to neutral elements in high temperature argon plasma and analyzed based on their mass to charge ratios. An ICP-MS can be thought of as having four main processes, including sample introduction and aerosol generation, ionization by an argon plasma source, mass discrimination, and the detection system.

All matrices, including surface and ground waters, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments and other solid wastes, wipes and air filters are applicable to this analysis. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Total recoverable metals require acid digestion prior to analysis. This SOP pertains to the analysis of digestates and standards.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts and technicians are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. Supervisors/Managers are responsible for ensuring that SOPs are accurate and up to date, and that they are implemented appropriately. Supervisors/Managers review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

3.1 Prior to analysis, total (acid-soluble) metals samples must be digested using appropriate sample preparation methods. Dissolved metals do not require digestion. (see **Section 8.1**)

3.2 An ICP-MS combines a high-temperature ICP (Inductively Coupled Plasma) source with a mass spectrometer (MS). The ICP source converts the atoms of the elements in the sample to ions. These ions are then separated and detected by the mass spectrometer. For a complete description of ICP-MS see the primer at http://www.cee.vt.edu/ewr/environmental/teach/smprimer/icpms/icpms.htm, this gives a concise breakdown of how an ICP/MS works.

4. Sample Preservation, Containers, Handling, and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the client. For metals analysis, water samples are collected in 500 ml plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Soils are collected in 8-ounce glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 Sample holding time for metals analysis is 180 days from VTSR for both water and soil for all metals other than mercury.

5. Interferences and Potential Problems

Several types of interference effects may contribute to inaccuracies in the determination of an analyte by ICP-MS

5.1 Isobaric elemental interferences are caused by isotopes of different elements, which form singly or doubly charged ions of the same nominal mass-to-charge ratio.

5.2 Abundance sensitivity is affected by ion energy and quadruple operating pressure. Wing overlap interferences may result when a small m/z peak is being measured adjacent to a large one (also called wing overlap).

5.3 Isobaric Polyatomic Ion Interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use.

5.4 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 salts or high acid concentrations. If physical

interferences are present, they must be reduced by such means as a high-salts nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved salts is a salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This problem can be controlled by a high-salts nebulizer, wetting the argon prior to nebulization, using a tip washer, or by diluting the samples. A mass flow controller is used to control the argon gas flow rate.

5.5 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. These effects can be minimized by careful selection of operating conditions, by buffering of the sample, by matrix matching, and by standard-addition procedures.

5.6 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 or from the buildup 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 the rinse solution between samples. This method requires a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed using a longer rinse period.

5.7 Contamination by samples: Significant laboratory or instrument contamination may result when untreated effluents, in-process waters, landfill leachates, and other samples containing high concentrations of inorganic substances are processed and analyzed.

5.8 Contamination by indirect contact: It is imperative that every piece of the apparatus that is directly or indirectly used in the collection, processing, and analysis of ambient water samples be cleaned.

5.9 Contamination by airborne particulate matter: Samples may be contaminated by airborne dust, dirt, particles, or vapors from unfiltered air supplies; nearby corroded or rusted pipes, wires, or other fixtures; or metal-containing paint.

5.10 Physical, chemical and spectral interferences are primarily attributed to the sample matrix. Mitkem performs the following analyses in each analytical sequence.

a) Serial Dilution Test: One sample per matrix per batch; A 5x dilution must agree to within 10% of the original determination if the analyte concentration is sufficiently high (minimally a factor of 10 times the PQL of the sample). If not, a chemical or physical interference effect is suspected. Samples identified as field blanks or PEs should not be used for serial dilutions.

b) Post Digestion Spike Addition: If the spike (pre-digestion) recovery of an analyte falls outside of the control limits, a portion of the same sample (digestate) is spiked with a post-digestion spike. The PDS should be recovered to within 80-120% of its true value.

The spike addition should produce a minimum level of 10 x and a maximum of 100x the PQL If both the Matrix spike and the PDS fail, matrix effects are confirmed.

6. Equipment and Apparatus

6.1 Glassware

6.1.1 Class A volumetric flasks:

- 100 mL and 50 mL

6.1.2 Class A volumetric pipettes:

- 10-100µL adjustable Eppendorf.
- 100-1000µL adjustable Eppendorf.
- 1-5mL adjustable Fisher Scientific.

6.1.3 15mL Evergreen Polystyrene ICP tubes.

6.1.4 100µL and 1mL fixed Wheaton.

6.2 ICP-MS- Thermo Electron Xseries2 (Instrument ID: X1)

The built-in radio-frequency generator is FCC compliant.

The high purity grade (99.9%) argon gas is piped in from a main storage tank that is located in the rear of the building. The gas is stored in liquid form, drawn-off as a gas and then piped into the building for distribution to the instruments. The liquid argon supply is monitored remotely by the supplier, and resupplied on an automatic-delivery basis.

6.2.1 Operating conditions: The analyst should follow the instructions provided by the instrument manufacturer. Method detection limits, precision and interference effects must be investigated and established for each analyte. All measurements must be in the instrument operational range where spectral interference correction factors are valid. 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.

6.2.2 Instrument Maintenance: See manufacturer's operation manual for additional information.

6.2.2.1 Peristaltic pump tubing should be replaced after approximately 20 hours of instrument run time. Relative standard deviations within samples containing measurable analytes that are above 3% indicate

excessive tubing wear. Record routine maintenance in the instrument run logbook.

6.2.2.2 Torch and cones should be inspected daily for obvious buildup. Degraded performance will be clear when the daily performance check is run and fails or barely passes. Problems may be rectified by adjusting the instrument settings in a tune, and this should be attempted before cleaning the system. Light elements (Be) will be the first to be impacted by a dirty sample introduction system or cones.

6.2.2.3 The daily performance check will also indicate the need for mass calibration if responses are acceptable and mass verification differs by more than 0.1 from the true value

6.2.3 Troubleshooting: See manufacturer's operation manual.

7. Reagents and Standards

All standard solutions (multi-, and single element), and second source QC solutions are purchased from outside vendors. All solutions are traceable and meet with Mitkem's high purity requirements. Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. ***Please note that standards and reagents from other vendors than listed below could be used as long as the standards are of high purity (>96%) and traceable to reference materials*.** All standards are labeled as instructed in Mitkem SOP# 80.0001 Standard Preparation, Equivalency and Traceability. Reagents are labeled as instructed in Mitkem SOP# 80.0013 Reagent Purchasing and Tracking.

7.1 Hydrochloric acid (conc.), HCl, Trace Grade.

7.2 Nitric acid (conc.), HNO_3, Trace Grade.

7.3 Reagent water. Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. It is also referred to as DI Water.

7.4 Primary Standards: Mixed calibration standards and individual analyte standards; obtained from High Purity Standards, come in several stock solutions.

7.4.1 Intermediate (Secondary) and Working Standards: Dilutions of the above primary standards in acidified reagent water as appropriate.

7.4.1.1 A high level working calibration standard containing all elements required for calibration is prepared from the primary standards. This standard ranges in concentration from 0.2 to 50 ppm and is used as the

upper level of the curve (S6). From this standard the other calibration standards are prepared (S5 to S1) by dilution.

S5=1:2x dilution of S6
S4=1:10x dilution of S6
S3=1:40x dilution of S6
S2=1:100x dilution of S6
S1=1:200x dilution of S6

See **Table 2** for individual element concentrations.

7.4.2 Working Calibration standards and QC solutions are made up on a daily basis. All standards and solutions are documented in the lab's standard logbooks. Each laboratory area has Primary, Intermediate and the Working Standard Logbooks in hardcopy or in LIMS. Concentrations of working standards and the associated QC limits can be found in LIMS within the individual testcode SPECs, as identified by Standard or QC Sample name (i.e.: ICAL1) or refer to **Table 1**.

7.5 Tuning Solution and Mass Calibration: Prepare a dilution of mixed calibration standards with Beryllium, Magnesium, Cobalt, Indium and Lead at 10ug/L. The solution should be in 1%HNO_3.

7.6 Initial Calibration Verification Standard (ICV): An independent source standard prepared at a concentration near the high end of the calibration curve. This standard may also be purchased. See **Table 1**.

7.7 Low Level Initial Calibration Verification Standard (LLICV): A custom standard at or slightly below the report limit.

7.8 Interference Check Standard (ICS): QC Standards ICSA and ICSAB stock solutions are obtained from Accustandard. Prepare ICSA and ICSAB fresh daily. See **Table 1**.

7.9 Matrix Spike and Laboratory Control Sample: See preparatory methods for vendor information. LIMS will have final concentrations expected in the digestates in the LIMS testcode SPECs. See **Table 1**.

8. Procedure

8.1 The methods used for sample digestion or preparation are as follows:

8.1.1 For water samples to be analyzed for total acid soluble/recoverable metals determination: a 50mL aliquot of the unfiltered (well-shaken) sample is digested with nitric and hydrochloric acids using Mitkem **SOP #100.0003,** Methods SW-846 3005/3010.

8.1.2 For water samples to be analyzed for dissolved metals determination: a 50mL aliquot of the filtered (0.45µm filter) sample is nitric acid preserved using Mitkem **SOP #100.0003,** Methods SW-846 3005/3010. No digestion is required.

8.1.3 For soil samples to be analyzed for total metals determination: a representative sample (between 1.0 and 2.0 gram) is digested with nitric acid using Mitkem **SOP #100.0104,** Method SW-846 3050.

8.2 Precalibration routine

8.2.1 Prior to calibration, the instrument must be allowed to become stable. This may take up to a half-hour. Conduct any necessary mass calibration and resolution routines to bring peak width within the manufacturer's specifications and adjust mass calibration to within 0.1u over the range of 6 to 210u. The resolution must be verified to be less than 0.9amu full width at 10% peak height.

8.2.2 Performance Check: Demonstrate instrument stability and precision by analyzing the tuning solution as a single analysis with at least five integrations. The %RSD of the absolute signals for all of the multiple integrations in the tuning solution (as calculated by the instrument) must be $\leq$ 5.0% for each analyte.

8.2.3 Internal Standardization: For full range mass scans, a minimum of 5 internal standards (IS) must be used. The IS chosen must be consistent throughout the entire run sequence. IS shall be present in all samples, standards and blanks at the same levels. No IS are used in the tuning solution. If dilutions are performed on the digested samples, then the IS must be added after the dilution. IS are added by the instrument. The IS used at Mitkem are listed in Table 3.

8.3 Calibration: Instrument calibration is required each time the instrument is set up for a sequence or after a continuing calibration failure. Extensive instrument maintenance or significant analytical changes would require re-calibration as well.

8.3.1 Calibrate the instrument with one blank standard and at least 3 non-zero standards. See **Table 2**. One standard must be at, or below, the PQL. With a multi-point curve, the upper quantitation limit (UQL) may exceed the highest concentration calibration point and can be defined as the "linear dynamic" range. Alternately, the calibration curve could be prepared daily with a minimum of a calibration blank and a single standard at the appropriate concentration to effectively outline the desired quantitation range. The multi-point curve is the preferred method of calibration at Mitkem.

8.3.2 A minimum of 3 replicate integrations are required for data acquisition. Use the average of the integrations for instrument calibration and data reporting.

8.3.3 The calibration curve shall be calculated using linear regression by plotting the concentration of the standard in ug/L on the X-axis versus the corrected instrument response on the Y-axis. The correlation coefficient, r, for the calibration curve must be ≥ 0.998.

DoD requires a correlation coefficient r $\geq$0.995

8.3.3.1 If the recommended linear response cannot be attained using a minimum of three non-zero calibration standards, consideration should be given to adding more standards, particularly at the lower concentrations, in order to better define the linear range and the lower limit of quantitation. Conversely, the extreme upper and lower calibration points may be removed from the multi-point curve as long as three non-zero points remain such that the linear range is narrowed and the non-linear upper and/or lower portions are removed.

8.4 Calibration Verification

8.4.1 Initial Calibration Verification (ICV): After successful initial calibration, the accuracy of the curve is verified for every analyte by the analysis of the second or independent source ICV for each mass used to report final data results. The concentration of the ICV is near the upper end of the calibration range. Recovery should be within 90-110% recovery to verify the curve.

8.4.2 Low Level Initial Calibration Verification (LLICV): After successful ICV analysis, it is recommended that the accuracy of the low-end of the calibration range be verified by the analysis of a LLICV for each mass used to report final data results. The standard is prepared from individual elements standards. The recovery should be within ±30%. Alternatively, the low standard (S1) of a multi-level curve could be plugged back into the curve for verification.

DoD requires the LLICV meet 80-120 % recovery criteria when performing a one point calibration.

8.4.3 Continuing Calibration Verification (CCV): To ensure calibration accuracy during each analytical sequence, a CCV shall be analyzed and reported for each mass used for reporting final results for each element, at a frequency not to exceed every 10 samples during an analytical sequence. The CCV must be analyzed at the beginning of the run and after the last analytical sample. The recovery should be within 90-110% recovery to accept ongoing calibration accuracy. The analytical sequence can continue indefinitely as long as samples are being continuously analyzed without the instrument being turned off and successive CCVs meet the previously mentioned criteria.

8.4.3.1 CCV standards are prepared from the same source as the calibration and are at the mid-level (S5) concentration used during initial calibration.

8.4.3.2 The same CCV is used throughout the entire analytical sequence.

8.4.3.3 The CCV is prepared in the same acid matrix as the calibration standards.

8.5 Initial and Continuing Calibration Blanks (ICB/CCB): The ICB and CCB are identical in composition to the calibration blank (S0) used in the initial calibration. The ICB/CCB are analyzed immediately after the ICV/CCV to monitor for potential carryover of analytes. If the absolute value of the ICB/CCB result exceeds the PQL, the analysis is stopped and the problem corrected. The instrument must then be recalibrated.

8.6 Sample Analysis

8.6.1 Interference Check Sample (ICS): To verify corrections for elemental and polyatomic isobaric interferences and to monitor for all interferents, an ICS is analyzed and reported for all elements on the Target Analyte List (TAL) immediately after the initial calibration but not before the ICV/ICB pair. The analysis of the ICS is immediately followed by the analysis of the CCV/CCB pair. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents and Solution AB consists of the analytes mixed with the interferents. The ICS analysis consists of analyzing both solutions consecutively, starting with Solution A.

8.6.2 Matrix Spike Sample (MS): Spiked samples are analyzed to provide information about the effect of sample matrix on the digestion and/or measurement methodology. The spike is added before the digestion reagents. At least one spike sample is performed on each batch of samples of a similar sample matrix type (water, soil...). Samples identified as field blanks or PEs should not be used for matrix spike samples. In some cases the sample requested for use as a matrix spike will be identified. The MS is spiked with the same solution as is used for the LCS, at the same concentration. Percent recovery is calculated for each analyte added.

8.6.3 Duplicate Sample (DUP): Duplicate samples are analyzed to provide information about the precision of the methodology for each element. Samples identified as field blanks or PE should not be used as duplicates. In some cases the sample requested for use as a duplicate will be identified. Relative Percent Difference is calculated for each analyte detected in the duplicate samples.

8.6.4 Laboratory Control Sample (LCS): The LCS is prepared by spiking an aliquot of reagent water or digesting an aliquot of an SRM. One LCS is prepared per

preparation batch of water or soil samples in a batch. Percent Recovery is calculated for each analyte in the LCS. The LCS must include all elements of interest.

8.6.5 After completion of the initial requirements of this method, field samples should be analyzed in the same operational manner used in the calibration routine. A rinse blank is run between all sample solutions, quality control samples, method blanks, and check solutions. Samples that exceed the linear dynamic range must be diluted and reanalyzed. Dilution factors should be appropriate to bring the readings within the upper 75% limit of the linear range.

Analytical Sequence:

1. Performance Check
2. Standard S0 (Blank std)
3. Standard S1 (Low std)
4. Standard S2
5. Standard S3
6. Standard S4
7. Standard S5 (Mid-range std =CCV)
8. Standard S6 (High std)
9. ICV
10. ICB
11. LLICV
12. ICSA
13. ICSAB
14. CCV
15. CCB
16. Samples
17. CCV (not to exceed 10 samples since last CCV)
18. CCB
19. Samples
20. CCV(not to exceed 10 samples since last CCV)
21. CCB

Note: If the last CCV, CCB meet the QC criteria the analytical run can be continued with using the same sequence protocol outlined above.

All analyses are documented in the ICP/MS Instrument Run Log.

9. Data Reduction and Calculations

9.1 Raw data can be evaluated on-screen using the PlasmaLab software package. PlasmaLab will identify all non-conforming data points and provide the analyst with details as to what elements or masses are meeting or not meeting pre-set quality control limits. Any data point

can be chosen using the cursor and right clicked to enable the *tooltip* reading pane on the bottom of the screen. The *tooltip* screen shows additional information including the type of quantitation or detector mode used for the mass calibration and data result. Data that are underlined also have been flagged to alert the analyst to the following (more information may be found in the Thermo Electron Xseries2 operation manual):

I=Invalid Calibration; alerts the analyst that there was a QC failure during the calibration and is common with certain alternate masses not used for quantitation.
T=Tripped; alerts the analyst that the detector was switched from pulse to analog as is done with high concentration analyses.
D= Semi-quantitative calibration mode used instead of Fully-; result is an estimate due to calibration issues as in "I".
M=Maximum exceeded; result is above the upper calibration range however not necessarily the linear range.

9.2 After completion of the analyses, the data files are exported to the server (\CLP Files directory). Data may be printed at this point or saved to pdf print file.

9.3 Soil samples are reported on a dry weight basis.

9.3.1 Percent Solids are calculated using the following formula:

$$\% \text{ solids} = \frac{DW \times 100}{WW}$$

DW = Sample weight (g) dried
WW = Sample weight (g) before drying

10. Quality Assurance/Quality Control

10.1 Instrument detection limits (IDLs) are a useful tool to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the PQL, nor should they be used in establishing this limit. IDLs are established initially when the instrument is set up and then on a biannual basis. IDL values are uploaded to the LIMS system for each instrument.

IDLs in µg/L can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). It may be helpful to compare the calculated IDLs to the testcode PQLs. It should be understood that the IDL must be less than the PQL. The PQL needs to be verified initially by analysis of a lower

limit of quantitation check (LLQC) sample and on an as needed basis to verify the lab's ability to detect elements at the reporting level. See **Section 10.4**.

DoD recommends performing an IDL study at instrument set-up or after significant maintenance or changes. The IDL ≤LOD.

10.2 The linear dynamic range (LDR) is established when the system is first setup, or whenever significant instrument components have been replaced or repaired, and on an as needed basis only after the system has been successfully calibrated using either the single or multi-point standard calibration approach. The upper limit of the linear dynamic range needs to 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 should be prepared, analyzed and quantitated against the normal calibration curve. The calculated value should be within ±10% of the true value. New upper range limits should be determined whenever there is a significant change in instrument response. The upper range should be verified every 6 months at a minimum. The analyst should be aware that if an analyte that is present above its upper range limit is used to apply a spectral correction, the correction might not be valid and those analytes where the spectral correction has been applied may be inaccurately reported.

DoD requires LDR or upper limit check standard every six months, at a minimum.

10.3 Method detection limits (MDLs) may be established annually however there is *no requirement* in the current SW-846 6020A method. During years that no MDL study is performed, quarterly LOD verification is required.

DoD requires an MDL study when the instrument is initially set up and quarterly LOD verifications thereafter. See Mitkem SOP #80.0005 for more details.

10.4 The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits (PQL) and on an as needed basis to demonstrate the desired detection capability. This check sample and the low-level calibration verification standard will be prepared at the same concentrations with the only difference being the LLQC sample is carried through the entire preparation and analytical procedure. PQLs are verified when all analytes in the LLQC sample are detected within ± 30% of their true value.

10.5 Initial Precision and Accuracy (IPANDA studies): Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results. The precision and accuracy studies are done as a combination of a preparative method and analysis. If an autosampler is

used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made.

10.6 Performance Check: If the mass calibration is not within the 0.1u over the range of 6 to 210 u, or the %RSD of all the integrations of the absolute signals of the analytes exceeds 5.0%, the analysis is terminated and the problem corrected. The instrument would then be re-tuned before any further calibration.

10.7 Initial Calibration Verification (ICV): The measurements should be within 90-110% recovery to verify the curve. If the ICV does not meet criteria the analysis shall be stopped, the problem corrected, and the instrument recalibrated before reanalyzing the ICV.

10.8 Low Level Initial Calibration Verification (LLICV): The measurements should be within 70-130% recovery to verify detection at the low end of the curve. If the LLICV does not meet criteria the analysis shall be stopped, the problem corrected, and the instrument recalibrated.

10.9 Continuing Calibration Verification (CCV): If the deviation of the CCV is greater than the control limits of 90 - 110% recovery, the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration verified, and re-analysis of all analytical samples analyzed since the last compliant calibration verification shall be performed for the elements affected.

10.10 Blanks: There are three types of blanks required by this method. The calibration blank (S0) is used in establishing the initial calibration. This same blank is used for the initial and continuing blanks (ICB/CCB) to monitor for carryover. The Method blank, or Preparation blank, is used to monitor contamination from preparation and analysis. A Rinse blank is used to flush the system between standards and samples.

10.10.1 ICB/CCB: If the absolute value of an analyte result exceeds the PQL the analysis is terminated and the problem corrected. The instrument would then require recalibration and reanalyis of all samples that were analyzed since the last compliant calibration blank. Calibration Blanks consist of the same concentrations of the same acids used to prepare the final dilution of the calibrating solutions. We use 1% (v:v) Nitric acid and 0.5% (v:v) HCl in reagent water. See section 7.2 of SW-846 6020A.

10.10.2 Method/Preparation Blanks (MB): The MB contains all the reagents in the same volumes as the samples it is prepared with. The MB is carried through the full procedure and has the same acid concentration in the final digestate as the associated samples. Analyze the MB in the same manner as a sample. Each preparation batch of 20 or less field samples has a minimum of one MB. For positive MB concentrations (the MB may be rerun once to verify results):

10.10.2.1 If the absolute value of the concentration is less than or equal to the PQL in the MB, no corrective action is needed.

10.10.2.2 If the analyte concentration is above the PQL in the MB, the lowest concentration of that analyte in the associated batch samples (not field blanks) is reportable if it is greater than or equal to 10 times the blank concentration. Otherwise all associated batch samples, with the analyte concentration less than 10 times the blank concentration and above the PQL, will be redigested and reanalyzed with new QC for that analyte. No blank correction is performed.

DoD requires no analyte in the MB detected > ½PQL or greater than 1/10 the amount measured in any associated sample. Common lab contaminants should be <PQL.

10.10.2.3 If the analyte concentration in the sample(s) is below the PQL, the samples may be reported despite the method blank contamination. This should be noted in the narrative.

10.10.3 Rinse Blanks consist of 2% HNO_3 and 0.5% HCl in reagent water.

10.11 Interference Check Sample (ICS):

10.11.1 Analytical results of Solution A (ICSA) shall fall within the control limits of ± 2 times the PQL or ±20% of the analyte's true value (the true value shall be zero unless otherwise stated or determined by multiple analyses as directed in section 9.7 of method 6020A) in the ICSA, whichever is greater. If not, the analysis shall be terminated and the problem corrected before recalibration.

DoD requires the absolute value of concentration for all non-spiked elements <LOD, unless they are a verified trace impurity from one of the spiked elements.

10.11.2 Analytical results of Solution AB (ICSAB) shall fall within the control limits of ± 2 times the PQL or ±20% of the analyte's true value for the analytes included in the ICSAB, whichever is greater. If not, the analysis shall be terminated and the problem corrected before recalibration.

DoD requires recoveries within ±20% of the analyte's true value for those analytes included in the ICSAB.

10.12 Matrix Spike analysis is acceptable if recovery is within **±**25%. If recovery is outside of this limit for any analyte then the associated samples are flagged. The only exception to these criteria is when the sample concentration exceeds the concentration of the spike added by a

factor of four or more. In this case no flagging is applied. When recoveries fail to meet criteria, a post digestion spike (PDS) is required.

DoD recovery limits for MS analyses are the same as LCS limits. DoD QSM does not give a set of limits for method SW6020, therefore, method SW6010 LCS limits or 80-120% will be used (silver in soil is 75-120%).

10.13 Post digestion spike (PDS): When matrix spike recovery fails, a post digestion spike is performed on the same sample as the spike was done originally. Spike the unspiked aliquot of the undiluted digestate to produce a minimum level of 10 x PQL and a maximum of 100x PQL. PDS recoveries outside of ±20% of the true value confirm matrix interference and the associated samples are flagged.

DoD allows PDS recoveries up to ±25%.

10.14 Duplicate: A control limit of **±**20% for RPD is used for original and duplicate samples. Corrective action required as a result of the duplicate analyses would be to analyze a serial dilution on the sample.

10.15 Serial Dilution (SD): If the analyte concentration is sufficiently high (minimally, a factor of 10 times above the PQL), a 5 times dilution should agree to within ±10% of the original sample analyte determination. If not, a chemical or physical interference effect is suspected and the associated samples are flagged.

10.16 Laboratory Control Sample (LCS): Recovery is calculated for all analytes and is to be within the control limits of **±**20%. In the event that the LCS does not meet criteria it may be reanalyzed once. If still unacceptable, associated samples must be redigested. Solid Reference Material (SRM) may be used as an LCS for soil sample batches. The manufacturer's established PT PAL acceptance criteria should be used instead of the ±20% criteria.

10.17 Internal Standards (IS): Internal standard response is monitored throughout the analytical sequence. Ratios of the raw uncorrected IS responses between isotopes should also be monitored routinely. This information can be useful for correcting problems caused by mass dependent drift, errors incurred in the IS addition, or background contributions from samples that cause high bias. If the intensity of any internal standard in a sample falls below 70% of the intensity of that internal standard in the initial calibration standard S0, a significant matrix effect must be suspected. Under these conditions, the established PQL has degraded and the correction ability of the internal standard technique becomes questionable. Make sure the instrument has not drifted by observing the internal standard intensities in the nearest clean matrix. If the low internal standard intensities are also seen in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilution of the affected sample. The sample must be diluted fivefold and reanalyzed with the

addition of appropriate amounts of internal standards. If the first dilution does not eliminate the problem, this procedure must be repeated until the internal-standard intensities rise to the minimum 70% limit.

DoD Internal standard intensity limits are 30-120% of the IS in the initial calibration.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and sample results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, notebooks and instrument logs are reviewed and signed regularly by the supervisor. The laboratory supervisor or another senior chemist reviews 100% of the data prior to report generation. The QA Director randomly reviews 10% of the data reported by the laboratory. Refer to Section 11 of the QAP for details.

11.2 The reporting group generates reports. The data submitted for report preparation is dependent on project requirements.

12. Corrective Action Procedures

12.1 Corrective actions to be implemented in the event QC results are outside of the acceptance range are covered in **Section 10**.

12.2 Corrective action reports (CARs) are initiated in the event of an out of control situation that cannot be corrected by the analyst. The procedure for initiating a CAR for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel. These sheets are stored in the bookcase adjacent to the Organic Analysis lab. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Health and Safety Manual. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

13.2 Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.

13.3 Many metal salts are extremely toxic if inhaled or swallowed. Use good housekeeping practices in areas where metal salts are being used and wash hands thoroughly after handling.

13.4 Inductively coupled plasma sources emit radio frequency radiation and intense UV radiation.

13.5 Basic good housekeeping practices such as the wiping up of spills immediately and regular cleaning of counters and hoods will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Method 6020A, Inductively Coupled Plasma- Mass Spectrometry. Revision 1, February 2007, U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.1 April 2009.

Operation manual(s) for Thermo Electron Xseries2

http://www.cee.vt.edu/ewr/environmental/teach/smprimer/icpms/icpms.htm, internet reference for how an ICP/MS works.

Attachments:
Table 1: QC/ Standard Concentrations
Table 2: Calibration Standard Concentrations
Table 3: Internal Standards
Attachment 1: DoD Table F-8

Table 1: QC/ Standard Concentrations

ELEMENT	MASS	PPB 6020 LLICV	PPB 6020 ICV	PPB 6020 CCV	PPB 6020 ICSA	PPB 6020 ICSAB	PPB 6020 LCS	PPB 6020 MS
Be	**9**	**1**	**50**	**100**	**X**	**X**	**50**	**50**
B *	**10**	**50**	**500**	**1000**	**X**	**X**	**500**	**500**
Na	**23**	**500**	**15000**	**25000**	**250000**	**250000**	**5000**	**5000**
Mg	24	500	10000	25000	100000	100000	5000	5000
Mg	**25**	**500**	**10000**	**25000**	**100000**	**100000**	**5000**	**5000**
Mg	26	500	10000	25000	100000	100000	5000	5000
Al	**27**	**20**	**2000**	**2000**	**100000**	**100000**	**2000**	**2000**
K	**39**	**500**	**50000**	**25000**	**100000**	**100000**	**5000**	**5000**
Ca	**44**	**500**	**5000**	**25000**	**300000**	**300000**	**5000**	**5000**
V	**51**	**5**	**500**	**100**		**200**	**500**	**500**
Cr	**52**	**2**	**200**	**100**		**200**	**200**	**200**
Cr	53	2	200	100		200	200	200
Fe	54	100	1000	10000	250000	250000	1000	1000
Mn	**55**	**5**	**500**	**100**		**200**	**500**	**500**
Fe	56	100	1000	10000	250000	250000	1000	1000
Fe	**57**	**100**	**1000**	**10000**	**250000**	**250000**	**1000**	**1000**
Co	**59**	**1**	**500**	**100**		**200**	**500**	**500**
Ni	**60**	**1**	**500**	**100**		**200**	**500**	**500**
Ni	61	1	500	100		200	500	500
	63	2	250	100		200	250	250
Cu	**65**	**2**	**250**	**100**		**200**	**250**	**250**
Zn	**66**	**5**	**500**	**100**		**100**	**500**	**500**
Zn	67	25	500	100		100	500	500
Zn	68	25	500	100		100	500	500
As	**75**	**2**	**400**	**100**		**100**	**40**	**40**
Se	78	5	500	100		100	50	50
Se	**82**	**5**	**500**	**100**		**100**	**50**	**50**
Mo *	**95**	**2**	**250**	**100**	**2000**	**2000**	**200**	**200**
Mo *	97	2	250	100	2000	2000	200	200
Ag	**107**	**1**	**50**	**100**		**50**	**50**	**50**
Ag	109	1	50	100		50	50	50
Cd	**111**	**1**	**50**	**100**		**100**	**50**	**50**
Cd	114	1	50	100		100	50	50
Sb	**121**	**2**	**100**	**100**	**X**	**X**	**100**	**100**
Sb	123	2	100	100	X	X	100	100
Ba	**135**	**10**	**2000**	**1000**	**X**	**X**	**2000**	**2000**

Ba	137	10	2000	1000	X	X	2000	2000
Tl	203	1	500	100	X	X	50	50
Tl	205	1	500	100	X	X	50	50
Pb	206-208	1	500	100	X	X	20	20

Bold are Reported Mass
X= no requirement for 6020A
*B and Mo not listed in 6020A

Table 2: Calibration Standard Concentrations

		ppb	ppb	ppb	ppb	ppb	ppb	ppb
ELEMENT	MASS	S1	S2	S3	S4	S5	S6	PQL
Be	**9**	1	2	5	20	100	200	1
B	**10**			50	200	1000	2000	50
Na	**23**	250	500	1250	5000	25000	50000	500
Mg	24	250	500	1250	5000	25000	50000	500
Mg	**25**	250	500	1250	5000	25000	50000	500
Mg	26	250	500	1250	5000	25000	50000	500
Al	**27**	20	40	100	400	2000	4000	20
K	**39**	250	500	1250	5000	25000	50000	500
Ca	**44**	250	500	1250	5000	25000	50000	500
V	**51**		2	5	20	100	200	5
Cr	**52**		2	5	20	100	200	2
Cr	53		2	5	20	100	200	2
Fe	54	100	200	500	2000	10000	20000	200
Mn	**55**	1	2	5	20	100	200	5
Fe	56	100	200	500	2000	10000	20000	200
Fe	**57**	100	200	500	2000	10000	20000	200
Co	**59**	1	2	5	20	100	200	1
Ni	**60**	1	2	5	20	100	200	1
Ni	61	1	2	5	20	100	200	1
Cu	63		2	5	20	100	200	2
Cu	**65**		2	5	20	100	200	2
Zn	**66**		2	5	20	100	200	5
Zn	67		2	5	20	100	200	5
Zn	68		2	5	20	100	200	5
As	**75**	1	2	5	20	100	200	2
Se	78			5	20	100	200	5
Se	**82**			5	20	100	200	5
Mo	**95**	1	2	5	20	100	200	2
Mo	97	1	2	5	20	100	200	2
Ag	**107**	1	2	5	20	100	200	1
Ag	109	1	2	5	20	100	200	1
Cd	**111**	1	2	5	20	100	200	1
Cd	114	1	2	5	20	100	200	1
Sb	**121**		2	5	20	100	200	2
Sb	123		2	5	20	100	200	2
Ba	**135**	10	20	50	200	1000	2000	10
Ba	137	10	20	50	200	1000	2000	10
Tl	203	1	2	5	20	100	200	1
Tl	**205**	1	2	5	20	100	200	1
Pb	**206-208**	1	2	5	20	100	200	1

Table 3: Internal Standards

Concentrations chosen to yield approximately 700,000 raw counts

element	ppb
Li6	100
Sc	50
Y	15
Rh	20
In	15
Tb	15
Ho	15
Lu	10
Bi	15

Attachment 1:
DoD Table F-8

Table F-8. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be ≤ LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Tuning	Prior to ICAL.	Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height; For stability, RSD ≤ 5% for at least four replicate analyses.	Retune instrument then reanalyze tuning solutions.	Flagging criteria are not appropriate.	No analysis shall be performed without a valid MS tune.
Initial calibration (ICAL) for all analytes (minimum one high standard and a calibration blank)	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.

Table F-8. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analytes within ± 10% of true value.	Verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All analytes within ± 10% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard	Daily, after one-point ICAL.	Within ± 20% of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.
Linear dynamic range or high-level check standard	Every 6 months.	Within ±10% of true value.	NA.	NA.	
Method blank	One per preparatory batch.	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-8. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS-A and ICS-AB)	At the beginning of an analytical run and every 12 hours.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes to be reported	One per preparatory batch.	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests (dilution test and post-digestion spike addition) are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD < 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table F-8. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020) (continued)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Dilution test	One per preparatory batch.	Five-fold dilution must agree within ± 10% of the original measurement.	Perform post-digestion spike addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post digestion spike addition	When dilution test fails or analyte concentration for all samples < 50 x LOD.	Recovery within 75-125% (see Table B-1).	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Internal standards (IS)	Every sample.	IS intensity within 30-120% of intensity of the IS in the ICAL.	Reanalyze sample at 5-fold dilution with addition of appropriate amounts of internal standards.	Flagging criteria are not appropriate.	
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Determination of Metals in Water and Soil by Inductively Coupled Argon Plasma Atomic Emission Spectrometry using Method SW846 6010C

Contents SOP NO. 100.0111

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		12/28/10
Quality Assurance Director		12/28/10
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	SR Analyst	3/31/11			
	Supervisor	8/8/11			

Revision Record

Revision Date	Revision Description	Comments	Initials
2/11/08	Section 7.5.9 LDR standards, lab name	Revised	SBL
4/3/08	Draft 6010C to final update IV edits. Removal of upper range determination stds per 6010 method clarification.	Removed letters of prep methods so future versions will not req. updates to this SOP.	SBL
6/24/08	Edit volume of LCS spike: rounding error for many years.	0.45ml to 455uL, 0.045mL to 45.5 uL	SBL
11/7/08	Edited some numbering, bulleting, references to LIMS conc, MDLs	Correlation coefficient, low ICV,	SBL
2/03/09	Changed low level ICV requirement	Full	SBL
11/24/09	QSM4.1 added	Minor	SBL
12/22/10	Frequency of LRA, ICSA/B, and IEC, include LLQC, and a few std prep corrections plus IS per Tom Sawyer	Full rev	SBL
03/31/11	Add B, Mo and Sn info for stnds	Minor rev	SBL
04/08/11	Edited Yttrium to Lutetium as IS	Minor rev	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Determination of Metals in Water and Soil by Inductively Coupled Argon Plasma Atomic Emission Spectrometry using Method SW846 6010C

Rev. 13

	Signature	Date
QA Director:		12/28/10
Lab Director:		12/28/10
Effective Date:	1/4/2011	

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Determination of Metals in Water and Soil by Inductively Coupled Argon Plasma Atomic Emission Spectrometry using Method SW846 6010C Rev. 13

1. Scope and Application

This SOP describes the procedures applicable to the analysis of the elements listed in **Attachment 1**. All matrices, including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. **Section 8.1** provides the Method references for sample digestion procedures. See **LIMS Test Information/ Test/Limits** for analytes and their associated MDL/PQLs.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

3.1 Prior to analysis, samples must be digested using appropriate sample preparation methods.

3.2 The method measures element specific emitted light by optical spectrometry. The samples are nebulized and the resulting aerosol is transported to the plasma torch. The metals pass through the hot zone of the plasma, where they take up energy. Subsequently the metals pass through the cold zone (relatively) of the plasma where they give up the excess energy at element specific wavelength. The spectra are dispersed by a grating spectrometer, and the intensity of the emitted light is measured by a solid state photomultiplier. Background correction is required for trace element determination. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the

analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured.

4. Sample Preservation, Containers, Handling, and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For metals analysis by Method 6010C, water samples are collected in 500 ml plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Soils are collected in 8-ounce glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 Soil samples are stored at 4°C ± 2°C until analyzed.

4.3 Sample holding time for metals analysis by method 6010C is 180 days from the date of sample collection for both water and soil.

5. Interferences and Potential Problems

Several types of interference effects may contribute to inaccuracies in the determination of an analyte by ICAP-AES.

5.1 Spectral interferences - Can be categorized as (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena. The first of these can be compensated for by utilizing a computer correction of raw data, requiring monitoring and measurement of the interfering element. The second effect may require selection of an alternative wavelength. In addition one could select an alternate wavelength where interference is minimal or absent. The 4300DV and the 3100XL used at Mitkem have many spectral lines from which to choose. The third effect can usually be compensated by a background correction adjacent to the analyte line.

5.2 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 salts or high acid concentrations. If physical interferences are present, they must be reduced by such means as a high-salts nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved salts is a salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This problem can be controlled by a high-salts nebulizer, wetting the argon prior to nebulization, using a tip washer, or by diluting the samples. A mass flow controller is used to control the argon gas flow rate.

5.3 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. These effects can be minimized by careful selection of

operating conditions, by buffering of the sample, by matrix matching, and by standard-addition procedures.

5.4 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 or from the buildup 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 the rinse solution between samples. This method requires a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed using a longer rinse period.

5.5 Physical, chemical and spectral interferences are primarily attributed to the sample matrix. If interference caused by a particular sample matrix is known, in many cases it can be circumvented. However, when the nature of the sample is unknown, following tests can be used to ensure the analyst that neither positive nor negative interference effects are operative on any of the analyte elements thereby distorting the accuracy of the reported values.

5.5.1 Dilution Test -If the analyte concentration is sufficiently high (minimally a factor of 10x above the method detection limit (MDL)), an analysis of a 1:5 dilution should agree within ±10% of the original determination. If not, a chemical or physical interference effect could be suspected.

5.5.2 Post Digestion Spike Addition - If the matrix spike (pre-digestion) recovery falls outside of the control limits (75% - 125%), matrix interference is suggested. In this case an analyte spike is added to a portion of a prepared sample or its dilution at a level just below the mid-point of the calibration curve. Post digestion spikes should be recovered to within 80% - 120% of the known value. If not, a matrix effect should be suspected.

5.5.3 Comparison with alternative method analysis-when investigating a sample matrix, comparison tests may be performed with other analytical techniques, such as atomic absorption spectrometry, or ICP-mass spectrometry. This should only be done after consultation with the client.

5.5.4 Internal Standard Addition technique can be used (Also refer to Section 4.4.2 of Method 6010C). Internal Standard correction is used to minimize the impact of system wide fluctuations in intensity resulting from diverse external factors such as temperature change, sample introduction changes and plasma conditions.

Internal standard intensities in each sample and standard are reported as a percentage of the intensity measured in the calibration blank (S0). Although there are no required internal standard recoveries, we would remark in the case narrative if standard recoveries are outside of a +/- 50% window.

Depending on sample matrix issues one of the following metals could be used as the internal standard: Yttrium, Lutetium or Indium. The internal standard is prepared at 5ppm in a solution of 1% HNO3 :1.5% HCl. The element chosen for use is highly dependent on the fact that batch samples may naturally contain these same elements. This can create bias with regards to concentration measurement. The option to not use the internal standard for concentration calculations is always available.

Internal standard is mixed into each sample and standard prior to measurement using a mixing block (Perkin Elmer, Cat# B0507962).

The sample is introduced into the mixing block using 0.76mmID (black-black) PVC tubing and the internal standard is introduced using 0.51mm ID (orange-yellow) PVC tubing.

6. Equipment and Apparatus

6.1 Inductively coupled argon plasma emission spectrophotometer (ICAP).

The ICAPs used at Mitkem are a Perkin-Elmer Model 4300DV and a 3100XL. The 4300DV is outfitted with an AS-93plus, 157-position autosampler and a high precision, three channel, peristaltic pump. The 3100XL is outfitted with an AS-91, 160-position autosampler and a high precision, three-channel peristaltic pump. Both ICPs have axial viewing capability, as compared to the more traditional radial viewing ICAP. Axial viewing provides greater sensitivity for all elements analyzed. The solid state detector is capable of analyzing at approximately 6000 wavelengths.

The built-in radio-frequency generator is FCC compliant.

The systems are computer controlled through a 32-Bit, Microsoft Windows NT operating system. This system allows for great flexibility in controlling the instrument. The software program used is WinLab 32.

The required high purity argon gas is piped in from a main storage tank that is located in the rear of the building. The gas is stored in liquid form, drawn-off as a gas and then piped into the building for distribution to the instruments. The liquid argon supply is monitored remotely by the supplier, and resupplied on an automatic-delivery basis.

6.1.1 Operating conditions - The analyst should follow the instructions provided by the instrument manufacturer. Instrument detection limits, linear dynamic ranges, and interference effects are established for each analyte line used. All measurements must be in the instrument linear range where spectral interference correction factors are valid. 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.

6.1.2 Daily emissions of the highest standard for arsenic, copper, lead and selenium are recorded as a means to ensure the plasma is stable before analysis and also to chart potential problems especially with the power tube, sample introduction system, or RF generator. The normal range of the four analytes above are (the unit is intensity generated by instruments):

Optima 2 (3100 XL)	Optima 3 (4300 DV)
As 2,000 - 3,000	As 2,000 - 2,500
Cu 600,000 - 900,000	Cu 400,000 - 600,000
Pb 12,000 - 20,000	Pb 4,000 - 8,000
Se 2,000 - 4,000	Se 1,800 - 2,400

6.1.3 For analysis of normal environmental samples use the following standard operating conditions:

Wattage: 1450 – 1500
Argon flow rate (L/min): 15
Nebulizer flow rate (L/min): 0.55
Sample flow rate (mL/min): 2
Rinse time (sec): 60
Read delay (sec): 60

6.1.4 For regular day-to-day ICP measurements, the plasma need not be optimized prior to analysis. The plasma needs to be optimized each time a major change occurs in instrument configuration, such as replacement of the torch and/or maintenance, unless response is still within normal ranges. Responses and alignment are routinely checked after torch cleaning or replacement with the analysis of a 1 mg/L Mn solution for axial view calibration and 10 mg/L Mn solution for radial view calibration. “Optimization” is a function of a series of procedures; refer to the Mitkem SOP 100.0006 for additional information on how to perform general maintenance and troubleshooting procedures.

6.1.5 To optimize the plasma, follow the instructions in the Optima Software Guide.

6.2 ICAP Instrument Maintenance:

6.2.1 Preventative Maintenance:

- Peristaltic pump tubing will be replaced every 16 hours of instrument time or sooner if memory effects are manifested.
- The plasma torch is cleaned using concentrated HNO_3 when torch and sample injector buildup is noted.
- The spray chamber and nebulizer are cleaned approximately every month as needed. Replacement is done when needed.

- Air filters are cleaned once every two weeks or as needed upon visual inspection.

- The RF coil, window and cone are cleaned every 2-3 weeks.

- The instrument undergoes extensive maintenance by a manufacturer's service engineer as needed.

6.2.2 Troubleshooting:

6.2.2.1 Sudden drop in CCV concentration occurs in a run:

- Check to see that sample introduction system (tubing) has not become disconnected.

- Check to see if a clog has occurred in the sample introduction system.

- Check CCV Standard to ensure it is not empty.

- Check room temperature to see if a fluctuation has occurred.

- Recalibrate and rerun if necessary.

6.2.2.2 CCVs drift up and down:

- Remake the CCV standard.

- Check room temperature to see if a fluctuation has occurred.

- If problem persists, call Perkin-Elmer. The problem is usually indicative of a power tube failure.

6.2.2.3 Plasma goes out during analysis:

- Sample is not reaching spray chamber (tubing came apart or there is a clog)

- High levels of salt in the sample caused plasma temperature changes.

- The sample injector is coated with high salt material and needs to be changed.

- The power tube is going (after all others fail repeatedly) and Perkin-Elmer must be called for service.

6.3 Glassware:

6.3.1 Class A volumetric flasks: 100 mL and 50 mL.

6.3.2 Class A volumetric pipettes:

6.3.3 10-100µL adjustable Eppendorf.

6.3.4 100-1000 µL adjustable Eppendorf.

6.3.5 5mL adjustable Fisher Scientific.

6.3.6 100µL and 1mL fixed Wheaton.

6.3.7 10 mL Wheaton ICP tubes.

7. Reagents and Standards

All standard solutions (multi-, and single element), and second source QC solutions are purchased from outside vendors (Perkin-Elmer, AccuStandard, Inorganic Ventures, ERA, and High Purity Standards, Inc.). All these solutions are traceable and meet with Mitkem's high purity requirements. ***Please note that standards and reagents from other vendors could be used as long as the standards are of high purity (>96%0 and traceable to reference materials.*** All standards are labeled as instructed in Mitkem SOP # 80.0001 Standard Preparation, Equivalency and Traceability.

7.1 Hydrochloric acid (conc.), HCl, Trace Grade, Fisher Scientific.

7.2 Nitric acid (conc.), HNO_3 , Trace Grade, Fisher Scientific.

7.3 Reagent water (ASTM Type II water). Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. Reagent water is also referred to as DI water.

7.4 Mixed calibration standard solutions. Mixed calibration standards, obtained from High Purity Standards, come in three stock solutions. Calibration standards and QC solutions are made up on a daily basis.

7.4.1 CLP_CAL_1 consists of two solutions:

- CLP_CAL_1B contains silver only.
- CLP_CAL_1A contains all other elements other than antimony, arsenic, cadmium, lead, thallium, and selenium.

7.4.2 CLP_CAL_2 contains antimony only.

7.4.3 CLP_CAL_3 contains arsenic, cadmium, lead, thallium, and selenium.

7.5 Single element standards are also used. Boron, Molybdenum, Titanium and Tin are received as primary standards at 1000mg/L from High Purity.

Please note that the following preparation procedures pertain to the use of the primary stock standards listed. Different preparation schemes are needed if different stocks are used or different final volumes are needed. All standards' preparation is documented in the appropriate Metals Standard Logbook and/or LIMS.

7.6 High standard (**Cal Standard 1**):

- Pipette 1.0mL HNO_3 (conc) and 1.5mL HCl (conc) into a 100mL volumetric flask.
- Pipette 1.0mL each of CLP_CAL_1A & 1B stock solution and 0.1mL each of intermediate CLP_CAL_2 and 3 into the flask.
- Pipette 0.1mL of Titanium stock standard into the flask.
- When required, also pipette 0.5 mL of Boron, 0.2mL of Molybdenum, and 1.0mL of Tin stock standards into the flask. (Or, this standard can also be made alone for a separate calibration mix).
- Bring to volume with DI water.

7.7 Second (middle) standard (**Cal Standard 2**):

- Pipette 5.0mL of a 1% HNO_3 and 1.5% HCl acid mixture into a 10 mL ICP tube.
- Add 5.0mL of the Cal Standard 1.

7.8 Third (low) standard (**Cal Standard 3**):

- Pipette 10mL of 1% HNO_3 and 1.5% HCl solution into a 10 mL ICP tube.
- Withdraw 100µL.
- Add 100µL of Cal Standard 1 and mix well.

7.9 **Second source** ICV/CCV standards (CCV100XCONC, Antimony and Titanium Stock solutions) are obtained from Accustandard.

- Pipette 1.0mL of HNO_3 (conc.) and 1.5mL HCl (conc.) into a 100mL volumetric flask.

- Pipette 1.0mL (for ICV) of CCV100XCONC stock solution into the flask.
- Pipette 0.05mL each of Antimony and Titanium Stock solutions into the flask.
- When required, also pipette 0.25mL of Boron, 0.1mL of Molybdenum and 0.5mL of Tin Stock solutions (AccuStandard) into the flask, or prepare a separate solution of these elements.
- Bring to volume with DI water.
- Transfer the solution to 50mL plastic ICP tubes.
- This standard is prepared as needed (in amounts consistent with the ratios above) usually every 1 – 2 days.

7.10 QC Standards ICSA and ICSB solutions are obtained from High Purity Standards.

ICSAB solution: In addition to the normal element composition of the ICSA solution, sodium, potassium, lead and selenium are added to the ICSAB solution. These stock solutions are also obtained from High Purity Standards.

7.10.1 Pipette 1mL of HNO_3 (conc.) and 1.5mL HCl into a 100mL volumetric flask.

7.10.2 Pipette 1mL of ICSB stock solution (ANALCS-R) into the flask.

7.10.3 Pipette 10mL of ICSA (CLP-INF-1) stock solution into the flask.

7.10.4 Pipette 0.5mL of the sodium/potassium stock solution (INFCS-5) into the flask.

7.10.5 Pipette 1mL of 45mg/L Pb intermediate standard solution into the flask.

7.10.5.1 The intermediate is prepared by adding 4.5mL of Pb at 1000mg/L (#100028-1) up to 100mL with 1%HNO_3 and 1.5% HCL acid solution in a volumetric flask.

7.10.6 Pipette 1mL of 45mg/L Se intermediate solution into the flask.

7.10.6.1 The Selenium intermediate standard is prepared by adding 4.5mL of Se at 1000mg/L (#031) up to 100mL with 1% HNO_3 and 1.5% HCl acid solution in a volumetric flask

7.10.7 Bring to volume with DI water.

7.11 Laboratory control samples (LCS) and Matrix spikes: The LCS standards for soils and waters are obtained from High Purity Standards.

- LCS/Spike standard 1 is prepared by adding 455uL of CLP-CV-1 to the digestion tube.

- LCS/Spike standard 2 and 3 are prepared by adding 45.5uL each of CLP-CV-2 and CLP-CV-3 to the digestion tube.

Concentrations of Calibration Standards and QC Samples can be found in **Attachment 3**.

7.12 Linear Dynamic Range

The stock linear dynamic range (LDR) standards are purchased from Ultra Scientific, Inorganic Ventures and High Purity. The LDR is determined as the highest concentration of standard in which the determined value is within 10% of the true value. Linear Dynamic Range is not determined for Boron, Molybdenum or Tin. These elements are diluted if above the highest calibration standard concentration in any sample.

- *LDR Standard I* is prepared by addition of 1 mL each of Pb, Co, and Ni at 1000 mg/L, volumized to 10 ml with 1% HNO3 and 1.5% HCl. Final concentration 100mg/L.

- *LDR Standard II* is prepared by addition of 5 mL of mix QCS-19 and 0.5mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 50 mg/L.

- *LDR Standard III* is prepared by addition of 2.5 mL of mix QCS-19 and 0.25 mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 25 mg/L.

- *LDR Standard IV* is prepared by addition of 1.0 mL of mix QCS-19 and 0.1 mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 10mg/L.

- *LDR Standard V* is prepared by addition of 0.5 mL of mix QCS-19 and 0.05ml of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 5mg/L.

- *LDR Standard VI* is prepared by addition of 0.5 mL of Al and 0.5ml of Mg at 10,000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 500mg/L.

- *LDR Standard VII* is prepared by addition of 0.5mL of Fe at 10,000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 500mg/L.

- *LDR Standard VIII* is prepared by addition of 0.5 mL of Ca at 10,000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 500mg/L.

- *LDR Standard IX* is prepared by addition of 0.5 mL of mix INFCS-5 (High Purity) and volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 250mg/L.

8. Procedure

8.1 The methods in SW-846 for sample digestion or preparation are as follows:

8.1.1 Method 3005, SOP No.100.0003, prepares ground water and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with hydrochloric and nitric acids prior to metal determination.

8.1.2 Method 3010, SOP No.100.0003, prepares aqueous samples, mobility-procedure extracts and waste samples that contain suspended solids for total metal determination by ICP. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid.

8.1.3 Method 3050, SOP No.100.0104, prepares solid waste samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrochloric acid.

8.2 The wavelengths and background correction locations in the reference method can be substituted if they can provide the needed sensitivity and are corrected for spectral interferences. The analyst should follow the instrument manufacturer's instructions, and if possible, approximate the recommended operating conditions.

For each analyte there are a number of possible wavelengths at which analyses could be made. The wavelengths used were selected based on consultations with the ICP specialists at Perkin-Elmer and our own experience.

8.3 Background correction factors are obtained by aspirating a concentrated solution, usually about 200-500ppm, of the interfering element, and measuring the resulting concentrations at the wavelength used. The background correction factors for that interfering element are obtained by dividing the measured concentrations by the actual concentration. For example: by aspirating a 500ppm solution of aluminum, one measures a cadmium concentration 0f 0.010ppm. The aluminum correction factor for cadmium is (0.010)/500 = 0.000020. This means that if the aluminum concentration in a sample is, say, 120ppm, one must subtract 120*0.000020 = 0.0024ppm from the measured cadmium concentration in that sample.

8.4 Interelement Correction (IEC) factors are established for the major salts: aluminum, calcium, iron, magnesium, and also for chromium, copper, manganese, nickel, thallium, titanium and vanadium. Correction factors should not exceed 20% difference from the previous IEC values. IEC factors must be verified daily (when in use) and be updated every 6 months. Daily verification is performed using ICSA/ICSAB standards.

8.5 Linear Dynamic Range is established for each wavelength. Following instrument calibration, solutions with varying concentrations (**section 7.5.13**) of each analyte (LDR standards) are analyzed. The highest concentration, within ±10 % of the true value, establishes the linear range. The upper range limits should be determined whenever there is a significant change in instrument response. At a minimum, the range should be checked every 6 months.

8.6 Allow the plasma to become thermally stable before beginning the analyses. This usually requires at least 30 minutes. If the plasma is (for whatever reason) extinguished and needs to be re-lit, subsequent re-stabilization of the plasma takes only 15 minutes provided the plasma is re-lit immediately after it is extinguished.

8.7 The nebulizer flow rate used is constant for all aqueous sample extracts and needs not be reset prior to analysis.

8.8 The instrument operating conditions finally selected as being optimum should provide the lowest reliable instrument detection limits (IDL). See **Section 10** for IDL calculation.

8.9 The calibration curve consists of a blank (S0) and at least 3 calibration standards. The low concentration calibration standard concentration is less than or equal to the reporting limit (RL=PQL or LOQ). Sample concentrations less than the lowest standard can only be reported after method modifications have taken place, or with estimated results flagging. The highest concentration must be within the instrument's linear dynamic range. The standards are run in a sequence from high concentration to low concentration.

8.10 The minimum correlation coefficient for the calibration curve is 0.998. If a correlation coefficient of less than 0.998 is obtained, the calibration must be repeated.

8.11 A mid-range ICV (Initial Calibration Verification) Standard from an independent source than the calibration standards) precedes the analysis of the samples. Recovery limits for the ICV are ± 10% of the true value. If the ICV recoveries do not meet acceptance criteria, corrective action must be taken and/or the calibration must be repeated. An ICV can be reanalyzed only once prior to corrective action.

8.11.1 A low level ICV (not required to be second source) is run to verify the lower level of calibration. Recovery limits for the low level ICV are ± 30% of the true value. The low level ICV is the same concentration as the reporting limit. If the low level ICV recoveries do not meet acceptance criteria, corrective action must

be taken and/or the calibration must be repeated. A low level ICV can be reanalyzed only once prior to corrective action.

8.12 A CCV (Continuing Calibration Verification Standard) is analyzed after at least every tenth sample and at the end of the sample run. Recovery limits for the CCV are ±10% and in the case of failure for a particular element, all samples following the last acceptable CCV must be reanalyzed for that element. A CCV can be reanalyzed only once prior to corrective action.

8.13 The ICB is analyzed after the ICV and is of the same source as the calibration blank. The CCB (Continuing Calibration Blank) is analyzed after each CCV. Concentrations of any analytes detected must not exceed the PQL or corrective action, such as a single reanalysis and evaluation, must be taken. If the ICB/CCB still fails, reanalysis of all samples since the last valid ICB/CCB for that element is required.

DoD QSM: The acceptable absolute value of the ICB and CCB must be < LOD.

8.14 The ICS (ICSA and ICSAB) standards must be run at the beginning of each analytical run. Additional analyses of the ICSA/ICSAB standard set during the analytical sequence are optional.

8.14.1 The ICSA solution, containing the Al, Ca and Mg at 500mg/L, and Fe at 200mg/L, must be run at all wavelengths used for each analyte reported. The analytical results for those target analytes with PQLs ≤ 10ug/L shall fall within ± 2x PQL of the analyte's true value (the true value shall be zero unless otherwise stated). If the results for these analytes fall outside the ± 2x PQL window, check that the background correction factors are appropriate, and readjust if necessary. Recalibration may be necessary. For analytes with a PQL > 10 ug/L, the ICSA results shall fall with ± one PQL of the analytes true value (0).

DoD QSM: the absolute value of all non-spiked analytes < LOD unless a verified trace impurity.

8.14.2 The ICSAB contains Al, Ca, Mg and Fe at the same concentrations as the ICSA as well as all other analytes of interest. Recovery limits for the ICSAB must be within ± 20% of the true value. If the ICSAB recoveries do not meet acceptance criteria, corrective action must be taken. Check that the background correction factors are appropriate, and readjust if necessary. Recalibration may be necessary. If the ICSAB at the end of an analytical sequence fails for a particular element, all samples must be reanalyzed for that element.

8.15 After completion of the initial requirements of this method, samples should be analyzed in the same operational manner used in the calibration routine. A 60 second rinse (rinse solution: 1% HNO_3 and 1.5% HCl) is conducted between all sample solutions, quality control samples, method blanks, and standards.

Analytical Sequence: The following QC protocol should be employed.

1. Standard S0
2. Standard S1
3. Standard S2
4. Standard S3
5. mid-range ICV(second source)
6. ICB
7. low-level ICV (either source)
8. ICSA
9. ICSAB
10. Sample 1 to 8
11. CCV
12. CCB
13. Sample 9 to 16
14. ICSA*
15. ICSAB*
16. CCV
17. CCB

Note: * Optional analyses. If run, the ICSA and ICSAB sample count as analytical samples between CCV/CCBs.

Any deviations must be approved by the Inorganic Laboratory Manager or the Supervisor before they can be implemented.

All analyses are documented in the Instrument Run Log (**Attachment 4**).

9. Data Reduction and Calculations

9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/Kg dry weight for solid samples. Results are reported to two or three significant figures.

9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with allowance for sample dilution. Check off "DISS" in LIMS Analytical Sequence once uploaded, to identify the samples as dissolved metals. Do not report analyte concentrations below the reporting limit (RL), unless specifically requested by the client.

9.3 Soil concentrations are calculated using the equation below:

$$\text{Sample Conc. (mg/Kg)} = \frac{C \times V \times Df}{W}$$

Where: C = Concentration in extract (µg/L)
V = Volume of extract (L, 100mL = 0.1L)

Df = Dilution factor (undiluted =1)
W= Dry weight of sample aliquot extracted (g)

9.4 Recovery Calculations:

The recovery of a spiked analyte is calculated as follows:

% Recovery (%R) = 100 x (SSR-SR)/ (SA)

Where: SSR = spiked sample result
SR = sample concentration
SA = spike added

9.5 Relative Percent Difference Calculations:

The relative percent difference (RPD) between replicate determinations is calculated as follows:

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

Where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value (replicate)

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results.

10.2 Method blanks - A preparation blank is prepped and analyzed with every batch not to exceed 20 samples. Method blank concentration must be less than or equal to reporting limits unless the sample concentration is at least 10 times greater than the blank concentration or less than reporting limits. Corrective action for method blank contamination involves determining the source of the contamination and re-prepping the affected samples in the batch. The analyst may rerun the method blank once as the first step of corrective action.

DoD QSM -Method blank concentrations must be less than or equal to one-half the reporting limit (or no greater then 1/10th the amount in any associated sample). For common contamination analytes, method blank concentration must be less than or equal the reporting limit.

10.3 Calibration verification –

10.3.1 The mid-range ICV analyzed immediately after standards must be within ±10% of the true value. The ICV is an independent source standard purchased from a different vendor than the calibration standards.

10.3.1.1 Method 6010C recommends an additional calibration verification using a low-level ICV with 70-130% criteria, however only the mid-range ICV is required to be from an independent source. In lieu of a second analysis, the response from the S3 standard can be entered back into the curve for verification.

10.3.2 The CCV is analyzed a minimum of every 10 samples in the analysis and at the end of the analytical sequence. If the closing CCV does not meet the criteria, the CCV and all analyses from the opening CCV must be re-analyzed after the problem has been eliminated.

10.3.3 The ICV and CCV analyses are followed by the ICB and CCB analyses, respectively. The ICV and CCV must pass the ±10% criteria or be re-analyzed.

10.4 Use of all standards made from a primary standard must not exceed the primary standard's expiration date.

10.5 Matrix spike (MS) samples - A matrix spike is processed with each batch of samples. Spike recoveries must be within 75-125% of the expected value. If the native sample results exceed 4x the spike added, no further action is needed. Note in narrative. Unless superseded by project requirements, it is not necessary to spike Na, K, Ca, and Mg for waters; or Na, K, Ca, Mg, Fe, Al and Mn for soil.

DoD QSM: Spike recoveries must be within 80-120%. Precision requirements are ≤ 20 % RPD for both aqueous and soil matrices. There is no corrective action for MS recoveries outside the acceptance range other than data qualification.

10.6 Matrix Duplicate (DUP) or matrix spike duplicate (MSD) samples (Both options are allowed in method SW6010) - Duplicates are prepped and analyzed with every batch not to exceed 20 samples. Relative Percent Difference (RPD) is calculated for the results of duplicate samples.

10.6.1 A limit of 20% RPD shall be used for sample values greater than 5x the PQL
.

10.6.2 A control limit of (±) the PQL level must be used if either the sample or the duplicate value is less than 5x PQL.

10.6.3 If one result is above 5x PQL and one below, use the ± PQL criteria.

10.6.4 If both values are below the PQL, no RPD is calculated.

DoD QSM- DUP precision is evaluated for all analytes. Precision requirements are ≤ 20 % RPD for both water and soil. There is no corrective action for MS/MD precision outside the acceptance range other than data qualification.

10.7 Laboratory Control Sample (LCS) - is prepped with a minimum of every 20 samples of the same matrix.

10.7.1 For an aqueous LCS sample, mixed standards are spiked into a beaker of DI water resulting in concentrations approximately ½ the concentration of the high calibration standard and prepped as an aqueous sample. Recoveries must be within the established control limits. The ID of the aqueous LCS sample is LCSW.

10.7.2 For soils, approximately 1g of Teflon Chips (Chemware Ultra-pure PTFE boiling stones, acid washed) may be used to simulate solid matrix is spiked with standards at the approximate mid-point of the calibration curve. The LCS is then prepped as a soil. The ID for the solid LCS sample is LCSS.

DoD QSM –The acceptance range for both water and soil is 80-120% except silver which has a 75-120% range for soils. When Mitkem in-house LCS limit fall within DoD limit, in-house limits may be reported. If Mitkem in-house limits are wider than DoD limits, the 80-120 % limits must be used.

Laboratory control sample acceptance limits are based on control charts, which are established at Mitkem. These are referred to as in-house limits. Mitkem may also adopt limits from outside agencies when necessary.

10.8 The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the laboratory reporting limits (PQL/LOQ) and on an as-needed basis to demonstrate the desired detection capability. This check sample and the low-level calibration verification standard will be prepared at the same concentrations with the only difference being the LLQC sample is carried through the entire preparation and analytical procedure. PQL/LOQs are verified when all analytes in the LLQC sample are detected within ± 30% of their true values.

10.9 Post digestion spike (PDS) addition - An analyte spike added to a portion of the sample digestate. A PDS is analyzed with each preparation batch of samples (PDS spiking of the same sample as used for matrix spiking is recommended) and is suggested when a new or unusual matrix is encountered within a batch. The acceptance criteria for PDS are 80-120%. The PDS is spiked at the same concentrations as the matrix spike. The PDS concentration should be >10 times the PQL, but < 100 times. If the PDS is not recovered within the specified limits, a matrix effect is confirmed.

DoD QSM: A PDS is required when the serial dilution test fails. In addition, a PDS is recommended for projects where all samples are below 50X LOD. The acceptance window is 75-125%.

Corrective action should be taken if the spike concentration is at least 2x the native sample concentration:

(a) If the MS fails for any analyte but the PDS passes, the sample may be redigested and analyzed, except when low MS recovery can be explained as a historically poor performer (example: Antimony (Sb) for soils) in that matrix. In these instances the analyst may want to discuss the outliers with the project manager or department supervisor. When re-digestion is warranted, and the MS fails in the re-digestate, a matrix issue is confirmed. If the MS passes for the re-digestate, the initial results were due to human error during digestion or spiking.

(b) If both the MS and PDS fail, the laboratory will perform a dilution test, or may make a reasonable effort to address the matrix interference by performing the method of standard additions (MSA) or internal standard addition.

10.10 Dilution test (Serial Dilution) – should be performed with each preparation batch. If the analyte concentration is sufficiently high (minimally, a factor of 10 above the MDL), an analysis of a 5X dilution should agree within ±10% of the original determination. If not, a chemical or physical interference effect is confirmed. A serial dilution should be run whenever the PDS fails, to confirm matrix effect.

DoD QSM: A Serial Dilution is required once per preparation batch. If the SD fails, a PDS must be run.

As a general rule, Mitkem performs a PDS and SD with each preparation batch of samples.

10.11 Instrument detection limits (IDLs) are established at the time the instrument is set up and every six months thereafter. Ten solutions of diluted acid (1% HNO3 and 1.5% HCl) are analyzed on three non-consecutive days. The IDL for a particular element is three times the average standard deviation of the measured concentration for that element.

DoD QSM: IDLs are established at the time the instrument is set up and whenever significant changes are made. IDLs must be < LOD.

10.12 Method detection limits (MDLs) are established at the time the instrument is set up and whenever significant changes are made. They are verified annually or more often as required by specific programs. The MDL is obtained by multiplying the standard deviation of seven analyses by the appropriate one-sided 99% t-statistic. The value of this statistic equals 3.143 if the number of analyses is seven. The concentration of the

analyte in the analyzed solution should be between three to five times the calculated MDL. An MDL verification check is performed immediately following the MDL study. The MDL verification check sample is spiked at approximately 1-4 times the current MDL and prepared as if it were a sample. Mandatory MDL determination has been removed from the SW846 6010C method however until regulatory agencies are in agreement, Mitkem will continue to perform MDL studies and/or quarterly LOD checks. The MDL verification check is used to establish the DoD LOD concentration. The LOD is verified quarterly on all instruments by matrix.

10.13 Sample Dilutions: When analytes exceed the established linear dynamic range, the sample must be diluted sufficiently to bring those elements into the linear range. The dilution factor (Df) must be integrated into the final concentration calculation.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and sample results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the supervisor. The laboratory manager reviews 100% of the data prior to report generation. The QA Director or designee randomly reviews 10% of the data reported by the laboratory. Refer to Section 11 of the QAP for details.

11.2 Electronic files are validated by the analyst and transferred to the report generation group via the LIMS. Once data is in LIMS it is reviewed and validated again by the supervisor or his/her designee. The data are then locked and forms may be generated.

11.3 Reports are generated by the reporting group in conjunction with the metals lab. The data submitted for report preparation is dependent on project requirements.

12. Corrective Action Procedures

12.1 Corrective action to be implemented in the event QC results are outside of the acceptance range is covered in **Section 10**.

12.2 Corrective action reports (CARs) are initiated through the LIMS in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for initiating a CAR for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel on the bookcase between the GC/MS and OPrep labs. In addition, laboratory

personnel should follow the precautions outlined in the laboratory's Health and Safety Plan, and have read the Mitkem Contingency Plan.

In general, use gloves, a lab coat, and goggles when handling these reagents and work under a hood whenever possible.

13.2 Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.

13.3 Many metal salts are extremely toxic if inhaled or swallowed. Use good housekeeping practices in areas where metal salts are being used and wash hands thoroughly after handling.

13.4 Inductively coupled plasma sources emit radio frequency radiation and intense UV radiation.

13.5 Basic good housekeeping practices such as wiping up spills immediately and regularly cleaning counters and hoods will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's Quality Assurance Plan.

15. References

Fassel, V.A. et al. Simultaneous Determination of wear metals in lubricating oils by Inductively-Coupled Plasma Atomic Emission Spectrometry . Anal. Chem. 48: 516-519, 1976.

Patel, B.K.; Raab, G.A.; et al. Report on a single laboratory evaluation of Inductively coupled optical emission Method 6010; EPA contract No. 68-03-3050, December 1984.

U.S. Environmental Protection Agency. I*nductively Coupled Plasma Atomic Emission Spectroscopy* Method 6010C, SW-846 test methods for evaluating solids waste, Revision 3, Final Update IV, February 2007.

U.S. Environmental Protection Agency. Inductively Coupled Plasma-Atomic Emission Spectrometry Method for the Analysis of Waters and solids, EMMC, July 1992.

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lubricating oils by Inductively-Coupled Plasma Atomic Emission Spectrometry . Anal. Chem. 48: 516-519, 1976.

Winge, R.K. et.al. Inductively Coupled Plasma- Atomic Emission Spectroscopy: An Atlas of spectral Information, Physical Science Data 20. Elsevier Science Publishing, New York,1985.

Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.1 April 2009 or current version.

Attachments:

1. **Attachment 1**: Analytes of Interest (Table 1 from SW-846 6010C)
2. **Attachment 2:** DoD current QSM QC Requirements.
3. **Attachment 3**: Concentrations of Calibration Standards and QC Samples
4. **Attachment 4**: Instrument Run Logbook

Attachment 1
Analytes of Interest

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.

Attachment 2:
DoD QC Requirements

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be ≤ LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months.	Within ± 10% of true value.	NA.	NA.	

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes **ICP: minimum one high standard and a calibration blank;** **GFAA: minimum three standards and a calibration blank;** **CVAA: minimum 5 standards and a calibration blank**	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	ICP: within ± 10% of true value; GFAA: within ± 20% of true value; CVAA: within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard (ICP only)	Daily, after one-point ICAL.	Within ± 20% of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes to be reported	One per preparatory batch.	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution test (ICP and GFAA only)	One per preparatory batch.	Five-fold dilution must agree within ± 10% of the original measurement.	ICP: Perform post-digestion spike (PDS) addition; GFAA: Perform recovery test.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x LOD.	Recovery within 75-125% (see Table B-1).	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25 x LOD.	Recovery within 85-115%.	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table G-18. LCS Control Limits for Metals SW-846 Methods 6010 and 7470 Water Matrix[19]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	97	5	80	120	80	120
Antimony	98	4	80	120	80	120
Arsenic	98	4	80	120	80	120
Barium	99	4	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	100	4	80	120	80	120
Calcium	98	4	80	120	80	120
Chromium	100	4	80	120	80	120
Cobalt	99	3	80	120	80	120
Copper	99	3	80	120	80	120
Iron	102	4	80	120	80	120
Lead	99	4	80	120	80	120
Magnesium	98	4	80	120	80	120
Manganese	100	4	80	120	80	120
Mercury	100	5	80	120	No ME	No ME
Molybdenum	95	5	80	120	75	120
Nickel	100	4	80	120	80	120
Potassium	98	4	80	120	80	120
Selenium	98	6	80	120	75	120
Silver	97	5	80	120	75	120
Sodium	99	4	80	120	80	120
Thallium	97	4	80	120	80	120
Vanadium	99	4	80	120	80	120
Zinc	100	4	80	120	80	120

[19] The as-generated limits have been adjusted to reflect Method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for Method 6010, depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits.

Table G-19. LCS Control Limits for Metals SW-846 Methods 6010 and 7471 Solid Matrix [20]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	95	5	80	120	75	120
Antimony	96	5	80	120	75	120
Arsenic	95	4	80	120	80	120
Barium	98	3	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	97	4	80	120	80	120
Calcium	97	4	80	120	80	120
Chromium	99	5	80	120	80	120
Cobalt	98	4	80	120	80	120
Copper	97	3	80	120	80	120
Iron	100	4	80	120	80	120
Lead	95	4	80	120	80	120
Magnesium	96	3	80	120	80	120
Manganese	97	4	80	120	80	120
Mercury	100	6	80	120	No ME	No ME
Molybdenum	96	5	80	120	75	120
Nickel	97	4	80	120	80	120
Potassium	96	4	80	120	80	120
Selenium	93	4	80	120	75	120
Silver	96	7	75	120	70	125
Sodium	96	4	80	120	80	120
Thallium	94	4	80	120	80	120
Vanadium	99	3	80	120	80	120
Zinc	95	5	80	120	75	120

[20] The as-generated limits have been adjusted to reflect Method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for Method 6010, depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits.

Attachment 3:

Concentrations of Calibration Standards and QC Samples (ug/L)

ELEMENT	S1	S2/CCV	S3	ICSA	ICSB	ICV	LCS
Aluminum	20000	10000	200	500000	500000	10000	9100
Antimony	1000	500	10	0	600	500	455
Arsenic	1000	500	10	0	100	500	455
Barium	20000	10000	200	0	500	10000	9100
Beryllium	500	250	5	0	500	250	227
Cadmium	500	250	5	0	1000	250	227
Calcium	50000	25000	500	500000	500000	25000	22700
Chromium	2000	1000	20	0	500	1000	910
Cobalt	5000	2500	50	0	500	2500	2270
Copper	2500	1250	25	0	500	1250	1130
Iron	10000	5000	100	200000	200000	5000	4550
Lead	1000	500	10	0	500	500	455
Magnesium	50000	25000	500	500000	500000	25000	22700
Manganese	5000	2500	50	0	500	2500	2270
Nickel	5000	2500	50	0	1000	2500	2270
Potassium	50000	25000	500	0	25000	25000	22700
Selenium	1000	500	10	0	500	500	455
Silver	2500	1250	25	0	200	1250	1130
Sodium	50000	25000	500	0	25000	25000	22700
Thallium	1000	500	10	0	100	500	455
Vanadium	5000	2500	50	0	500	2500	2270
Zinc	5000	2500	50	0	1000	2500	2270
Boron	5000	2500	50	0	0	2500	2250
Molybdenum	2000	1000	20	0	0	1000	0
Tin	10000	5000	100	0	0	5000	900

Attachment 4:
Instrument Run Logbook

MITKEM LABORATORIES	SAMPLE RUN LOG: ICAP/4300DV	Date:	Analyst:
RUN ID:	LAB ID: OPT3		

POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID	
1			21			41			61			81		
2			22			42			62			82		
3			23			43			63			83		
4			24			44			64			84		
5			25			45			65			85		
6			26			46			66			86		
7			27			47			67			87		
8			28			48			68			88		
9			29			49			69			89		
10			30			50			70			90		
11			31			51			71			91		
12			32			52			72			92		
13			33			53			73			93		
14			34			54			74			94		
15			35			55			75			95		
16			36			56			76			96		
17			37			57			77			97		
18			38			58			78			98		
19			39			59			79			99		
20			40			60			80			100		
Comments:												101		
												102		
												103		

HNO3: _____

HCL: _____

Mixed Acid: _____

STANDARD: _____

ICV/CCV: _____

LLICV/CRI: _____

ICSA/ICSAB: _____

Logbook ID: 100.0129-12/10

Reviewed by: _____

MITKEM LABORATORIES	SAMPLE RUN LOG: ICAP/3100XL	Date:	Analyst:
RUN ID:	LAB ID: OPT2		

POS	Lab ID	POS	Lab ID	POS	Lab ID	POS	Lab ID	POS	Lab ID
1		21		41		61		81	
2		22		42		62		82	
3		23		43		63		83	
4		24		44		64		84	
5		25		45		65		85	
6		26		46		66		86	
7		27		47		67		87	
8		28		48		68		88	
9		29		49		69		89	
10		30		50		70		90	
11		31		51		71		91	
12		32		52		72		92	
13		33		53		73		93	
14		34		54		74		94	
15		35		55		75		95	
16		36		56		76		96	
17		37		57		77		97	
18		38		58		78		98	
19		39		59		79		99	
20		40		60		80		100	
Comments:								101	
								102	
								103	

HNO3: _____

HCL: _____

Mixed Acid: _____

STANDARD: _____

ICV/CCV: _____

LLICV/CRI: _____

ICSA/ICSAB: _____

Logbook ID: 100.0126-xx/10

Reviewed by: _____

Analysis of Hexavalent Chromium in Soil & Solid Samples

Contents SOP NO. 100.0208

Procedure Signatures

Title:	Signature	Date
Operations Director/Technical Director		1/8/10
Quality Assurance Director		1/6/10
Operations/Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	Supervisor	12/16/11			
	Supervisor	9/14/13			

Revision Record

Revision Date	Revision Description	Comments	Initials
3/18/11	Clarified DoD CCV criteria in main part of document, replaced logbook page	Revised prep log included	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

SBC 3/19/11

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Inorganic Preparation and Analysis of Hexavalent Chromium in Soil and Solid Samples

by

SW846 Methods 3060A and 7196A

SOP No. 100.0208
Rev. 8

	Signature	**Date**
QA Director:		1/6/10
Lab Director:		1/8/10

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Inorganic Preparation and Analysis of Hexavalent Chromium in Soil and Solid Samples

by

SW846 Methods 3060A and 7196A
Rev. 8

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of soil samples by USEPA SW846 Method 3060A and analysis by USEPA SW846 Method 7196A. Discussion includes sample digestion and analysis of hexavalent chromium in soil and solid samples. For aqueous samples refer to the SOP No.100.0308, Hexavalent Chromium by SM3500 D. Included in this document are specific quality control procedures for Massachusetts WSC-CAM, New Jersey DEP, Connecticut DEP, and the Department of Defense (DoD) projects.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts and technicians are responsible for performing analyses in accordance with the SOP and documenting any variation in the protocol. Supervisors/managers review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

A 50mL digestate is prepared from 2.5 g of sample, and analyzed. The reaction of an acidic buffer combined with 1,5-diphenylcarbohydrazide added to the sample produces a purple color when hexavalent chromium is present. The reaction is very sensitive and is measured by either manual or automated spectrophotometer at 540nm.

4. Sample Preservation, Containers, Handling and Storage

4.1 The client will collect all samples, and should use only glass or plastic tools and containers.

4.2 Holding time for soils is 30 days from collection, and the soil alkaline digestates are stable up to 168 hours (7 days) from preparation.

4.3 Samples are not preserved.

4.4 Samples are stored at 4 ±2°C.

5. Interferences and Potential Problems

5.1 Concentrations as high as 200mg/L of Mo or Hg can be tolerated.

5.2 Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause trouble.

5.3 Iron in concentrations greater than 1mg/L may produce a yellow color, but the ferric iron color is not strong and difficulty is not normally encountered if the absorbance is measured photometrically at the appropriate wavelength.

6. Equipment and Apparatus

6.1 1000 mL Class “A” volumetric flasks

6.2 100 mL Class “A” volumetric flasks

6.3 Spectronics Specgenesis 20 Spectrophotometer (540 nm, with 1cm light path) or Lachat QuikChem8000 Automated Ion Analyzer with pump, auto-sampler, and data system.

6.4 50mL PTFE digestion tubes with watch glasses.

6.5 Graphite block heater with hotplate, able to maintain 90-95° C.

6.6 Stir rods.

6.7 Meter, pH.

6.8 FilterMate Filtration Device, Environmental Express Cat. No. SC0407, with 0.45 and 2 µm PTFE filters.

6.9 100 mL Class “A” graduated cylinders.

6.10 25 mL Class “A” graduated cylinders

6.11 Fisher brand thermometers to ≥110° C.

6.12 1 mL Brandtech and Eppendorf pipettes.

6.13 Ohaus Top Loader balance calibrated to read to 2 decimal places.

6.14 Class "A" 25 mL mixing cylinders.

6.15 50 and 100 mL beakers.

6.16 Ottawa Sand.

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used, provided the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Diphenylcarbizide solution for manual analysis: Dissolve 0.25g 1, 5-diphenylcarbazide in 50mL of reagent grade acetone. This solution is stable for 1 month or until it becomes turbid, whichever occurs first.

7.2 Diphenylcarbizide solution for Lachat analysis: Dissolve 0.40g of diphenylcarbazide into 200 mL of propanol in a 1000 mL Class "A" volumetric flask. Stir until dissolved. Add 80 mL of concentrated H_2SO_4. **Be careful as solution becomes very hot!** Slowly volumize to 1000 ml with DI water. De-gas standard with nitrogen gas prior to use.

7.3 Chromium Stock Standard, (Primary) 1000mg/L concentration, ERA. Used to prepare calibration curve, LCS, and soluble MS.

7.4 Chromium Voluette Ampule Standard, (Second Source) 12.5mg/L Cr^{+6}, HACH Cat. No. 14256-10. Used to prepare ICV/CCV.

7.5 10% (v/v) H_2SO_4, Fisher, ACS trace metals grade: Dilute 10 mL or reagent grade sulfuric acid to 100 mL with DI water.

7.6 5N NaOH, Fisher Cat. No. S318-1, ACS certified or NaOH pellets.

7.7 Sodium carbonate (Na_2CO_3): anhydrous, ACS certified, Fisher Cat. No. S263-500.

7.8 Digestion reagent: dissolve 20.0 ± 0.05g NaOH and 30.0 ± 0.05g Na_2CO_3 in DI water in a 1000 mL Class "A" volumetric flask and dilute to the mark. Store the solution in a tightly capped polyethylene bottle at 20-25° C and prepare fresh monthly. *The pH of*

the digestion solution must be checked before using. The pH must be 11.5 or greater, if not, discard.

7.9 Concentrated HNO_3, ACS trace metals grade. Do not use if acid has taken on a yellow tinge as this may indicate a photo reduction of nitrate to nitrite, a reducing agent for hexavalent chromium.

7.10 Diluted HNO3: Prepare at 1:1.

7.11 Magnesium Chloride ($MgCl_2 \bullet 6H_2O$): crystals, ACS certified, Fisher Cat. No.M33-3.

7.12 Potassium dihydrogen phosphate (KH_2PO_4): Aldrich Cat. No. 22,130-9 and Fisher Cat. No. P285-500.

7.13 Dipotassium Hydrogen phosphate (K_2HPO_4): Fisher Cat. No. P288-500, ACS certified.

7.14 Phosphate buffer; 0.5 M K_2HPO_4/0.5 M KH_2PO_4 at pH 7: dissolve 87.09g of K_2HPO_4 and 68.04g of KH_2PO_4 into 700mL of DI water. Transfer to a 1000 mL Class "A" volumetric flask and dilute to volume.

7.15 Acidified carrier solution for Lachat analysis.

7.16 Lead Chromate, ($PbCrO_4$): Sigma Aldrich, ACS certified. Store in dry location at 20-25° C in a tightly sealed container. It is used to prepare the insoluble MS.

8. Procedure

8.1 Preparation of Calibration Standards:

8.1.1 Intermediate Standard:
In a Class "A" 100 mL volumetric flask, add 1mL Primary Stock Standard (ERA, 1000 mg/L). Dilute to the mark with digestion reagent. The final concentration is 10mg/L Cr. Document preparation in the Wet Chem Intermediate Standard Logbook.

8.1.2 Working Calibration Standards:
Into 50 mL digestion tubes, pipette the following volumes of the Intermediate Standard (10mg/L). Volumize to 50mL with digestion reagent and digest as a sample. Document preparation in the Wet Chem Working Standard Logbook. These standards form the initial calibration curve (ICAL):

S0	0 mL
S0.1mg/L	0.5 mL
S0.5mg/L	2.5 mL
S1.0mg/L	5 mL

S1.5mg/L	7.5 mL
S2.0mg/L	10 mL

8.1.3 The CCV (calibration check verification standards) at 0.2, 0.4 and 0.8 mg/L are prepared by pipetting 0.16, 0.32, and 0.64 mL of the Chromium Voluette Ampule (**Second Source**) standard into 25 mL Class "A" graduated cylinders and volumizing to 25 mL with DI water. The CCV is analyzed after the Initial Calibration, before samples, at the end of the analytical run, and between at least every **10 samples**.

8.1.3.1 New Jersey DEP requires the CCV be a digested mid-range second source standard. NJ DEP refers to the CCV as the calibration check standard (CCS).

8.1.3.1.1 The mid-point CCS is digested with every batch of up to 20 field samples. Concentration of the CCS is 1.0mg/L and is analyzed immediately after the instrument is calibrated, at least every 10 samples, and at the end of the analytical run.

8.1.3.1.2 The CCS is prepared by pipetting 4 mLof the second source Chromium Voluette Ampule standard into a 50 mL digestion tube and volumizing to 50mL with digestion reagent. Smaller or larger volumes of this standard can be made accordingly.

8.1.4 The ICB/CCB (calibration check blanks) are prepared using 50mL digestion reagent, and are digested as a sample. The calibration blank is digested with every batch of up to 20 field samples for all New Jersey DEP, Connecticut DEP, and Massachusetts WSC-CAM projects.

8.2 Method SW3060A/ Procedure for digestion of soil samples:

8.2.1 Adjust the temperature setting of each heating device used in the alkaline digestion by preparing and monitoring a temperature blank (a digestion tube filled with 50mL of digestion reagent). Maintain a temperature of 90-95° C. Document the temperature of the temperature blank at the 30 and 60 minute mark during the heating period, in the Hexavalent Chromium Soil Digestion Logbook, (**Figure 1).**

8.2.2 Place 2.5 ± 0.1g of the field-moist sample into a clean and labeled 50 mL PTFE digestion tube. The sample should be mixed thoroughly before an aliquot is removed. See SOP No. 110.0039 for more instruction.

8.2.3 Check the pH of the digestion reagent to verify it is at 11.5 or greater.

8.2.4 A method blank (MB) and a laboratory control sample (LCS) are digested with every batch of up to 20 samples. The method blank and LCS are

prepared using 2.5 ± 0.1g of Ottawa Sand and 50 mL of digestion reagent. The LCS is also spiked as a soluble matrix spike (**section 10.3**).

8.2.5 For one sample, weigh out three additional 2.5 ± 0.1g aliquots of the sample into 50 mL digestion tubes. Two of these aliquots will be used for matrix spikes. One aliquot will be used for the matrix duplicate.

8.2.6 Spike the two matrix spike samples (See **Sections 10.3 and 10.4)** and LCS (See **Section 10.3)**.

8.2.7 Add 50 mL of the digestion solution to each sample, blank and spike. Add a stir rod to each tube.

8.2.8 Add approximately 0.4g (400mg) of $MgCl_2$ and 0.5mL of the 1M phosphate buffer.

8.2.9 Stir the samples continuously (unheated) for at least **5 minutes** using the stir rod.

8.2.10 Move samples to graphite holder on hotplate.

8.2.11 Heat the samples and maintain a temperature of 90-95° C with constant stirring for 60 minutes. **Do not let samples spatter, boil, or go to dryness.**

8.2.12 Remove the samples from the hotplate, cool each solution to room temperature, and temporarily remove the stir rods. Volumize each digested sample to 50mL.

8.2.13 Insert the FilterMate filter and gently push filter through digestion tube until all solids are trapped at bottom.

8.2.14 Transfer 20 mL of filtered sample into a 50mL beaker, saving the remaining digestate.

Caution: ***CO_2 will be evolved when adding the nitric acid. This step must be performed in a fume hood.***

8.2.15 With constant swirling, **slowly** add 1:1 nitric acid to the beaker **drop wise**. Adjust the pH of the solution to 7.5 ± 0.5 and monitor the pH with a pH meter. If the pH of the digestate should drop below 7.0, discard the solution and use another 20mL portion from **section 8.2.14**.

8.2.16 Record the final neutral pH readings in the digestion logbook, under the pH 1 column.

8.2.17 Volumize the sample to 40 mL with DI water. Record the aliquot color in the digestion logbook.

8.3 Method 7196A/ Procedure for analysis of soil samples (Manual Spectrophotometer):

8.3.1 Adjust the pH of the sample from **Section 8.2.16** with 10% sulfuric acid to a pH of 2.0± 0.5. Volumize to 50 mL with DI water. Test the pH with a meter after all effervescence is complete. Record the final pH in digestion logbook**,** under the pH 2 column.

New Jersey DEP requires the color development and measurement procedure must be started within one hour after the last sample in the batch is filtered, and adjusted for pH.

8.3.2 Transfer 25 mL of the digestate to a second 50 mL beaker. Add 0.5mL of diphenylcarbazide solution and mix well. The remaining sample in the first beaker will have no diphenylcarbazide added and will therefore be the background sample.

8.3.3 If samples are still turbid or flocculent is present they can be filtered again through a 0.45 µm filter cartridge. If turbidity or flocculent material still remains, the samples can be further filtered through a 0.1 µm membrane.

8.3.4 Let sample aliquots treated with diphenylcarbazide solution stand 5-10 minutes for color development prior to reading the absorbance on the spectrophotometer.

8.3.5 Analyze the original sample without the color reagent (the background correction sample from **Section 8.3.2**) immediately after the colored portion. The absorbance of the uncolored sample will be subtracted from that of the colored to calculate the final concentration. Document all readings in the Hexavalent Chromium Analysis Logbook, **(Figure 2)**.

8.3.6 Analysis on the Spectrophotometer yield results in absorbance that is converted to concentration with the use of Beer's Law.

8.3.7 Calibration is done first by coloring the calibration standards as would be done for samples. (The establishment of the calibration curve can be found in **Section 8.1.3**). The digested S0 must be run first. The calibration curve is established mathematically as absorbance versus standard concentration. A linear correlation (r^2) of $\geq$0.995 must be established before sample analysis can occur. The curve is established quarterly or when the opening calibration does not meet criteria. Sample concentration is calculated from the regression equation. Samples exceeding the highest standard should be diluted and reanalyzed.

New Jersey DEP, Department of Defense (DoD), and Massachusetts WSC-CAM require that the instrument must be calibrated daily (once every 24 hours) or each time the instrument is set up, whichever is more frequent. Connecticut DEP requires the calibration be performed every 6 months or whenever the ICV fails.

8.3.8 Each day that the spectrophotometer is used requires that it be zeroed. A distilled water S0 is run prior to any standards or samples. When the curve is being run, the S0 is a digested blank.

8.3.9 Calibration Check Verifications are analyzed at varying concentrations (0.2, 0.4, 0.8 mg/L) bracketing every 10 samples. These are second source standards.

8.3.9.1 The mid-point calibration is also run after the initial calibration to verify its validity. When run immediately after the ICAL, it is referred to as the Initial Calibration Verification (ICV). The required recovery range for the ICV is 90-110 %.

8.3.9.2 Varying concentrations are then analyzed throughout the sequence, bracketing every 10 samples. The required recovery range for the Continuing Calibration Verification (CCV) is 80-120 %.(See **Section 8.1.3** for preparation).

DoD QSM4.1 CCV required recovery range for CCV is 90-110 %.

8.3.10 Calibration Check Blanks (ICB/CCB) are digested blanks analyzed after every ICV/CCV standard in the analytical sequence to evaluate analytical sensitivity and contamination. These are required for Massachusetts WSC-CAM, Connecticut DEP, and New Jersey DEP projects.

8.3.11 A Post Digestion Spike (PDS) is analyzed once per preparation batch. To an additional 25mL aliquot of a pH adjusted filtrate, add 8uL of 1000mg/L chromium standard to achieve a 0.8mg/L concentration of hexavalent chromium (a 2.5X dilution factor is included to include volume previously added during pH adjustment), to verify that neither a reducing condition or chemical interference is affecting color development. A smaller volume of sample may be used, adjust spiking volume accordingly. Acceptable recoveries are 85-115%.

8.4 Procedure for analysis of soil samples (Automated Spectrophotometer):

8.4.1 Adjust the pH of each standard and sample to 6.5 with 1:1 HNO_3.

8.4.2 Set up the instrument manifold per manufacturer's instructions.

8.4.3 Put appropriate reagent lines in the reagents and carrier solution.

8.4.4 Choose proper method and sample run log from the Lachat program on the computer.

8.4.5 Following manufacturer's instructions begin the analysis by setting up the calibration curve and then analyzing all relevant samples.

8.4.6 Perform one PDS per analytical batch of samples. Spike an additional aliquot of the pH adjusted filtrate with stock chromium standard as in **Section 8.3.6**.

9. Data Reduction and Calculations

9.1 Calculate the concentration of Hexavalent chromium using Absorbance and applying Beer's law; $y = mx + b$.

9.2 Hexavalent chromium, Cr+6, in mg/kg is calculated from the following equation:

$$\text{Cr+6 in mg/kg} = \frac{A \times B \times E}{C \times D}$$

A = Concentration from the calibration curve in mg/L
B = Final digested volume in liters
C = Wet weight of the sample
D = Percent solids in decimal format
E = Dilution factor of the sample if necessary

9.3 Sample spike recovery in percent is calculated from the following equation:

$$\% \text{ Recovery} = \frac{(SSR - SR)}{SA} \times 100$$

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

9.4 Calibration Check Verification (CCV) recovery is calculated from the following equation:

$$CCV = \frac{\text{CCV result}}{\text{True value of CCV}} \times 100$$

9.5 Relative Percent Difference (RPD) between the original sample and its duplicate are calculated from the following equation:

$$RPD = \frac{2\,(S-D)}{S + D} \times 100$$

S = Original Sample Value in mg/kg
D = Duplicate Sample Value in mg/kg

9.6 True Value Calculation for mg/L Hexavalent Chromium from a solid lead chromate (PbCrO4) spike:

PbCrO4 (molecular wt=323.2) 1g yields 0.161 g Cr+6

Therefore:
15mg into 50mL digestion solution = 300mg/L PbCrO4, or 48.3mg/L Cr+6
10mg into 50mL digestion solution = 200mg/L PbCrO4, or 32.2mg/L Cr+6
5 mg into 50mL digestion solution = 100mg/L PbCrO4, or 16.1mg/L Cr+6

This is the raw concentration prior to calculating the mg/kg result based on soil weight and percent moisture.

As an example, a 5mg spike on a 2.51g sample with 85%solids would have a true value expected concentration of 377.31 mg/kg Cr+6:

16.1mg/L * 0.050L/.00251kg * 0.85 = 377.31 mg/kg Cr+6.

10. Quality Assurance/Quality Control (Also see Table 1)

Quality assurance and quality control (QA/QC) procedures are established to ensure generation of data of known quality. QA/QC procedures associated include preparation of Method Blank, LCS, standards, matrix spike and sample duplicate.

10.1 Method Blank –a clean reference matrix (Ottawa Sand) that is carried through the entire preparation and analysis procedure. It is used to determine the level of contamination or any memory effects that may be occurring.

10.1.1 A Method Blank is prepared once per batch of up to 20 samples of similar matrix.

10.1.2 Blank concentration must not exceed one half PQL.

Massachusetts WSC-CAM and Connecticut DEP projects method blanks may not contain Cr+6 > PQL. NJDEP requires that method blanks do not contain Cr+6 > MDL.

10.2 Laboratory Control Sample (LCS) – is a spiked reference matrix (Ottawa Sand) that is carried through the entire preparation and analysis procedure and evaluated for recovery.

10.2.1 An LCS is prepared once per batch of up to 20 samples of similar matrix.

10.2.2 It is prepared by spiking the reference matrix with 50 uL of the stock chromium standard **(Section 7.3).** The spike is brought through the entire preparation and analysis process, and is evaluated for recovery.

10.2.3 The recovery range for the LCS is 80 to 120%.

Massachusetts WSC-CAM projects require an LCSD. The LCSD must be analyzed immediately following the LCS. The RPD must be $\leq$ 35%. ***A project specific MD/MSD may be substituted in lieu of an LCSD.***

10.3 The **Soluble** Matrix Spike is prepared once per batch of up to 20 samples of similar matrix. It is prepared by spiking the sample with 50 uL of the stock chromium standard **(Section 7.3).** The spike is brought through the entire preparation and analysis process, and is evaluated for recovery. Acceptable recovery limits for the matrix spike is 75- 125%.

10.4 The **Soluble** Matrix Spike Duplicate is prepared once per batch of up to 20 samples of similar matrix. It is prepared by spiking the sample with 50 uL of the stock chromium standard **(Section 7.3).** The spike is brought through the entire preparation and analysis process, and is evaluated for recovery. Acceptable recovery limits for the matrix spike is 75- 125%, with a $\leq$ 25% RPD.

Massachusetts WSC-CAM projects require a Soluble MSD **or a** Matrix Duplicate. They recommend performing the MS if analytes are not suspected to be detected and a DUP if they are. The RPD must be $\leq$ 35%. MSD recovery limits are set at 75-125%.

10.5 The **Insoluble** Matrix Spike is prepared once per batch of up to 20 samples of similar matrix. It is prepared by spiking the sample with 5-10mg of $PbCrO_4$.

Note: *It is imperative that the weight of the lead chromate added be approximately 1:10 w:v ratio of the final digestate volume (i.e. 5mg $PbCrO_4$: 50 mL final volume).*

The spike is brought through the entire preparation and analysis process, and is used to evaluate dissolution during the digestion process. Recommended recovery limits for the matrix spike per method SW3060A is 75-125%, (See **Section 9.6** for true value calculation) however in our experience these limits are extremely tight. Until adequate recovery data points are collected, we will use a wider 30 to 150% limit.

10.6 A Matrix Duplicate is prepared once per batch of up to 20 samples of similar matrix. The sample duplicate is brought through the entire preparation and analysis process and has an acceptance criterion of 20% RPD. There is no corrective action if the RPD exceeds 20% and both the sample and duplicate are greater than four times the PQL. Method 3060A allows for the use of ± the PQL to be used as the control limit for duplicates when the values are less than four times the PQL.

Department of Defense (DoD) allows for a 30%RPD between soil sample duplicates.

Massachusetts WSC-CAM / Connecticut DEP projects allows for a 35% RPD for soil duplicates where results are >5xPQL, and for samples with results <5xPQL, use difference of ≤2x PQL.

10.7 A Post Digestion Spike (PDS) is required with each batch of up to 20 samples of similar matrix. It is prepared by spiking an aliquot of the pH adjusted filtrate with the stock chromium standard. Do not choose a sample with high concentration. If necessary dilute the sample aliquot so the native concentration plus the spike fall within the calibration range. Recovery of the PDS should fall within 85-115%. If recoveries of the PDS are less than 85%, add 1N NaOH to an aliquot of the sample to pH 8.0-8.5. Record the final pH. Re-spike as before and reanalyze.

10.8 Method detection limits (MDLs) are established initially with the test development. The MDL is obtained by multiplying the standard deviation of seven or eight analyses by the appropriate one-sided 99% t-statistic. The value of this statistic equals 3.143 if the number of analyses is seven. The concentration of the analyte in the analyzed solution should be between three to five times the expected MDL. Since Hexavalent Chromium is not reported below the laboratory PQL, which is set at the low level of the calibration range, an annual MDL study is not performed.

DoD QSM4.1 LOD requirement: Quarterly analysis of check sample spiked at 2-3 times the established MDL to be used to determine/re-evaluate the limit of detection (LOD).

10.9 A PQL verification is performed annually by performing a low level LCS at 1-2 times the PQL. The recovery should be within a modified range appropriate to the spiking level.

DoD QSM4.1 LOQ requirement: Quarterly analysis of LOQ (PQL) check sample spiked at 2-3 times the LOQ.

11. Data Validation and Reporting

Results are completely checked by the analyst and again by the department supervisor. Data are then uploaded to the Omega LIMS system for reporting. A level 2 report is used for most Wet Chemistry tests. The uploaded data will go through a QA validation step prior to release to the client. Any corrective actions associated to the project will be documented on the data review checklist. Any comments pertaining to QA/QC or sample issues will then be documented in the project narrative.

12. Corrective Action Procedures

12.1 If the correlation coefficient for the curve is less than 0.995, new calibration standards are prepared and analyzed.

12.2 If the Initial Calibration Verification (ICV) is outside the ±10% range, the check standard may be rerun once. If still outside of the ±10% range, the analysis is stopped and the calibration curve is re-established.

Connecticut DEP allows for a ± 20% recovery of the Initial Calibration Verification.

12.2 If the LCS fails, the entire batch must be redigested and analyzed.

12.3 If the ***soluble*** Matrix Spike fails to meet recovery criteria, the entire batch must be redigested and analyzed. If the redigested samples have the same results, the data is qualified.

Massachusetts WSC-CAM projects require evaluation of the sample's oxidation/reduction characteristics when soluble MS recoveries fail and LCS recoveries are acceptable. Analysts should confer with the project manager when this happens. The project manager will contact the client for guidance.

12.4 If the ***insoluble*** Matrix Spike fails to meet recovery criteria, the entire batch should be redigested and analyzed per method guidance. If the redigested samples have the same results, the data is qualified.

12.4.1 Insoluble spikes prepared on a clean matrix, like an LCS, have shown approximately >80% recovery. Therefore, when recoveries are found to be below this level, it is acceptable to assume a probable matrix interference that suppresses the ability to efficiently digest the insoluble Cr+6 in the sample. See Method SW3060A Section 8.5 for further information. Also, <u>discuss the recovery issues with the project manager before making any redigestion decisions based on the ***insoluble*** Matrix Spike.</u>

12.4.2 Per USEPA Office of Solid Waste "Even though the method suggests that the recovery criteria for both the soluble and insoluble spikes be 75 -125%, it is understood that depending on the sample composition the insoluble spike recovery criteria may not be achievable for all matrix applications. We would therefore, suggest that the LCS be prepared with lead chromate and hopefully you should achieve the recommended recovery limits of 80 to 120%. The sample data would then be considered acceptable based on a probable matrix interference that suppresses the ability to efficiently digest the insoluble Cr+6" (4).

Massachusetts WSC-CAM projects require evaluation of the sample's oxidation/reduction characteristics when insoluble MS recoveries fail and LCS recoveries are acceptable. Analysts should confer with the project manager when this happens. The project manager will contact the client for guidance.

12.5 If the PDS fails to meet criteria, compare results to Matrix Spikes. Low recoveries may indicate high fulvic acid content. If recoveries of the post-verification spike are less than 85%, add 1N NaOH to an aliquot of the sample to pH 8.0-8.5. Record the final pH. Re-spike as before and analyze per **Section 8.3**.

12.6 For PDS that exceed the calibration range it is necessary to dilute the sample aliquot with DI water to within the calibration range. Dilution occurs prior to the addition of the PDS and of the reagents. Results for the PDS will need to be adjusted appropriately.

Note: See Method SW3060A Section 8.6.2 and Method SW7196 Section 7.3. for further information.

12.7 If the correlation coefficient for the curve is less than 0.995, new calibration standards are prepared and analyzed.

12.8 If the method blank exceeds one half the PQL it is rerun once. If it still exceeds one half the PQL, the associated samples are redigested and reanalyzed unless sample concentration exceeds 10 times the blank concentration. In this situation, sample results may be reported. The blank contamination must be noted on the data review form for inclusion in the narrative.

12.9 Calibration Check Blanks (ICB/CCB) must not contain Cr+6 > PQL. If the Cr+6 > PQL, recalibrate and re-analyze all samples since last compliant CCB.

12.10 Dilute samples if they exceed the calibration range or if highly colored. Use the smallest dilution factor possible. It is necessary to use the least dilution necessary to both bring the concentration into the calibration range and/or to minimize the effects aliquot color will have on the analysis. This dilution factor is recorded in the analysis logbook.

13. Health and Safety

Precautions to protect analysts include the nature of toxicity or carcinogenicity of analytes of reagents used in the method. Lab coats, gloves, and safety glasses must be worn at all times in the lab.

14. References

1. U.S. Environmental Protection Agency. Chromium, Hexavalent (Colorimetric) Method 7196A, SW846 Test Methods for Evaluating Solid Waste, Update III, Revision 1, July 1992.
2. U.S. Environmental Protection Agency. Alkaline Digestion for Hexavalent Chromium, Method 3060A, SW846 Test Methods for Evaluating Solid Waste, Update III, Revision 1, December 1996.

3. Department of Defense Quality Manual for Environmental Laboratories, Final Version 4.1 April 2009
4. Email dated Friday, December 07, 2007 8:17 PM:The MICE Service is operated by Science Applications International Corporation (SAIC) under contract to the USEPA Office of Solid Waste. All MICE Service staff are contractors. As such, they do not create or interpret USEPA policies. The role of the MICE service is to provide answers and take comments regarding the OSW methods manual known as "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846)."
5. Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup Quality Assurance and Quality Control Requirements for SW-846 Method 7196A, Hexavalent Chromium by UV-Visible Spectrophotometry for the Massachusetts Contingency Plan (MCP). WSC-CAM-VI B, 20 August 2004 Final Revision No. 3.
6. Connecticut DEP RRCP, Quality Assurance and Quality Control Requirements, Determination of Hexavalent Chromium by SW-846 Method 7196, Version 2.0, July 2006.
7. New Jersey Division of Remediation Management and Response, Standard Operating Procedure (SOP) for laboratory data evaluation and validation of Hexavalent Chromium analyzed in accordance with Update III (July 1998) USEPA SW-846 Method 3060A, USEPA SW-846 Method 7196A and USEPA SW-846 Method 7199, August 2005. SOP NO.: 5.A.10.

Attachments:

1. **Figure 1**: Hexavalent Chromium Soil Digestion Logbook Form.
2. **Figure 2**: Hexavalent Chromium Analysis Logbook Form (Manual Spectrophotometric).
3. **Table 1**: QC Check Summary

Figure 1

Hexavalent Chromium Digestion Logbook Form

MITKEM LABORATORIES	Weight (g)	Digestion Reagent (ml)	MgCl2	1M Phosphate Buffer (ml)	pH 1	color	pH 2	Comments
Hexavalent Chromium SW-846 3060 Soil Digestion Logbook								
Sample ID								

MS std ID: _____ Amount added_____ LCS std ID: _____ Amount added_____

Date: _____ Temp at 30 min: _____ Time on:_____

Analyst: _____ Temp at 60 min: _____ Time off:_____

Digestion Reagent ID: _____ Nitric Acid Lot #: _____

MgCL2 Lot #: _____ PbCrO4 lot #: _____ 1M Phosphate Buffer ID: _____

Logbook ID 100.0140-xx/11

Reviewed by:_____

Figure 2

Hexavalent Chromium Analysis Logbook Form (Manual Spectrophotometric)

MITKEM LABORATORIES **Hexavalent Chromium Analysis Logbook**

Analysis Date: **Analyst:**

Sample ID		pH (Aqueous Samples)	Sample Volume (ml)	Absorbance (Abs)	Calc. CONC (mg/l)	DF	Result (mg/l)	% Rec	comments

Wavelength=_____

Pathlength=_____

MRL=_____

ChromaVer 3 Lot #:_____

Diphenylcarbizide Solution ID:_____

Method# : SM3500 Cr D / SW7196A (circle one)

Conc of Analyte = (Abs-b)/m

m=_____

b=_____

r=_____

Logbook ID: 100.0056-xx/11

LIMS entry: _____

Reviewed By: _____

TABLE 1: QC SUMMARY

QC CHECK	NEW JERSEY DEP	CT DEP-RCP	MASS WSC-CAM	DoD QSM V4.1	MITKEM
ICAL	DAILY/ S0+4 LEVELS	SIX MO./ S0+5 LEVELS	DAILY/ S0+5 LEVELS	DAILY/ S0+3 LEVELS	QUARTERLY/ S0+5 LEVELS
ICV/CCS	90-110% SECOND SOURCE DIGESTED/MID RANGE	80-120% SECOND SOURCE	90-110% SECOND SOURCE	90-110% SECOND SOURCE	90-110% SECOND SOURCE
CCV/CCS	90-110% SECOND SOURCE EVERY 10 SAMPLES DIGESTED/MID RANGE	80-120% EITHER SOURCE EVERY 10 SAMPLES	80-120% SAME SOURCE AS ICAL EVERY 10 SAMPLES	90-110% EITHER SOURCE EVERY 15 SAMPLES	80-120% SECOND SOURCE EVERY 10 SAMPLES
CCB	REQUIRED AFTER ICV/CCS DIGEST <MDL	REQUIRED AFTER CCV DIGEST <PQL	REQUIRED AFTER CCV DIGEST <PQL	N/A	N/A
MB	BATCH/20 <MDL	BATCH/20 <PQL	BATCH/20 <PQL	BATCH/20 < HALF PQL	BATCH/20 <PQL
LCS	BATCH/20 80-120%	BATCH/20 80-120%	BATCH LCS/LCSD OR MD/MSD 80-120%	BATCH/20 80-120%	BATCH/20 80-120%
DUP	BATCH/20 RPD	BATCH/20 <35 RPD	OPTIONAL, OR MSD <35 RPD	OPTIONAL, OR MSD <30RPD	<20 RPD per SW3060
SOLUBLE MS	BATCH/20 75-125% 40 MG/KG	BATCH/20 75-125%	BATCH/20 75-125%	BATCH/20 75-125%	BATCH/20 75-125%
INSOLUBLE MS	BATCH/20 75-125%	BATCH/20 75-125%	BATCH/20 75-125%	BATCH/20 75-125%	BATCH/20 75-125%
PDS	85-115% per SW7196 PER PREP BATCH	NOT MENTIONED	NOT MENTIONED	85-115% per SW7196 PER PREP BATCH	85-115% per SW7196 PER PREP BATCH

Inorganic Analysis of Hexavalent Chromium in Aqueous Samples by Standard Methods SM3500 Cr +6 B

Contents SOP NO. 100.0308

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		10/12/12
Quality Assurance Director		10/12/12
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date

Revision Record

Revision Date	Revision Description	Comments	Initials
11/12/02	Added control page and renamed SOP	Was SOP WC08B1	
07/10/08	Lab name change	Full rev	SBL
11/5/10	pH check, filter for dissolved metals	NELAC audit	SBL
08/03/12	MUR update to 22nd ed, new letter version SM3500 CR+B-2009	Full rev	SBL

Procedure Superseded By _____ Date: _____
Procedure Discontinued By:_____ Date: _____
Procedure Archived By:_____ Date:_____

Spectrum Analytical, Inc.

featuring Hanibal Technology,
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Inorganic Analysis of Hexavalent Chromium in Aqueous Samples

by

Standard Methods SM3500 Cr +6 B

SOP No. 100.0308

Rev. 8

	Signature	Date
QA Director:		10/12/12
Lab Director:		10/12/12
Effective Date:	10/19/12	

Spectrum Analytical, Inc.
featuring Hanibal Technology,
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Inorganic Analysis of Hexavalent Chromium in Aqueous Samples

by

Standard Methods SM3500 Cr+6 B, Modified for Hach Reagents

SOP 100.0308 Rev. 8

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of aqueous samples for analysis by Standard Methods SM3500 Cr+6 B. Discussion also includes sample analysis of hexavalent chromium in aqueous samples.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts and technicians are responsible for performing analyses in accordance with the SOP and documenting any variation in the protocol. Supervisors/managers review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

A 25mL sample is used. An acidic buffer, (to reduce the pH to 1 ± 0.3) combined with 1,5-diphenylcarbohydrazide in a powder pillow, is added to the sample. The reaction from adding the acidic buffer and the reagent to the sample produces a purple color when hexavalent chromium is present. The reaction is very sensitive and is measured photometrically at 540nm.

4. Sample Preservation, Containers, Handling and Storage

4.1 The client will collect all samples.

4.2 Holding time for aqueous chromium sample analysis is 24 hours from collection.

4.3 Samples cannot be preserved.

4.4 Samples are stored at 4°C.

4.5 If dissolved chromium is requested, the sample must be filtered using a 0.45um membrane filter before preservation to pH <2. This should be done at time of collection.

5. Interferences and Potential Problems

5.1 Concentrations as high as 200mg/L of Mo or Hg can be tolerated.

5.2 Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause trouble.

5.3 Iron in concentrations greater than 1mg/L may produce a yellow color, but the ferric iron color is not strong and difficulty is not normally encountered if the absorbance is measured photometrically at the appropriate wavelength.

5.4 Highly buffered or extreme pH samples may exceed the buffering capacity of the reagents and require sample pretreatment. Test the pH of the sample and the reagent blank before analysis. Use HNO_3 to lower the pH and NaOH to raise the pH to the value of the reagent blank. Analyze the sample as before.

6. Equipment and Apparatus

6.1 25ml mixing cylinders, Class A

6.2 Spectrophotometer at 540 nm

6.3 pH Meter

6.4 Eppendorf-style adjustable pipettes

7. Reagents

7.1 ChromaVer 3 Chromium Reagent Powder Pillows. Available through HACH Catalog number: 12066-99. The pillows contain 1, 5-diphenylcarbohydrazide to produce the purple color indicative of hexavalent chromium. The reaction occurs in an acidic environment which is also manifested with the powder pillow.

7.2 Reagent grade water: should be monitored for impurities.

7.3 Chromium Voluette Ampule Standard, 12.5mg/L Cr+6, HACH Cat. No. 14256-10.

7.4 10% (v/v) H_2SO_4, Fisher, ACS trace metals grade.

7.5 5N NaOH, Fisher Cat. No. S318-1, ACS certified.

7.6 Chromium VI at 1000+/- 5 mg/L, ERA Cat.# 019 Potassium Dichromate in DI H2O. Used for LCS and MS spikes.

8. Procedure

8.1 Standard Preparation:

8.1.1 Calibration Curve: Pipette a chromium standard solution in measured volumes into 25 ml mixing cylinders ranging in concentration from 0.03 to 1.20mg/L when diluted to the appropriate volume. The standards are: 0.03, 0.1, 0.2, 0.4, 0.8, 1.20mg/L. The curve consists of at least 5 standards over this range. All standard preparation is recorded in the Wet Chemistry standard logbooks.

8.1.2 LCS: The LCS is made from the ERA Chromium VI standard by diluting 0.6mL to 100mL with DI water. The standard is good for 28 days. The concentration of the standard is 6.0mg/L. The LCS is analyzed at a 10x dilution.

Develop the color of the standards/LCS as for the samples (see **section 8.2**). Transfer a suitable portion of each colored solution to the 1cm absorption cell and measure the absorbance at 540nm. As reference, use reagent water/ DI H_2O. Correct the absorbance readings of the standards by subtracting the absorbance of a reagent blank carried through the method if necessary. Construct a calibration curve by plotting corrected absorbance values against mg/L of Cr+6. Do **not include the zero** standard in calculating the curve.

8.2 Procedure for Sample Preparation and Analysis:

8.2.1 If samples are turbid or highly colored, filter through 0.45um filter paper. Note in narrative if filtration is required.

8.2.2 Pour 25 ml of sample into a 25 ml mixing cylinder.

8.2.3 Use 25mL of DI H_2O for the Method Blank.

8.2.4 The sample chosen for matrix spike is spiked with 10uL of the straight ERA Chromium VI standard. The true value of the MS is 0.4mg/L.

8.2.5 Add the contents of one ChromaVer 3 Reagent Powder Pillow. Mix thoroughly. Check pH, record value in pH column.

8.2.6 Wait 5 minutes.

8.2.7 Record absorbance from the Spectrophotometer in the Colorimetric Analysis Logbook (**Figure 1**).

9. Data Reduction and Calculations

Read absorbance directly from the Spectrophotometer. Use this value to calculate the concentration based on slope and intercept from the curve:

Concentration = (Absorbance – b) / m
where: b = y intercept and
m = slope of the line

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are established to ensure generation of data of known quality. QA/QC procedures associated include preparation of Method Blank, standard, matrix spike and sample duplicate.

10.1 Method Blank – is a clean reference matrix (DI H_2O) that is carried through the entire analytical procedure. It is used to determine the level of contamination or any memory effects that may be occurring.

10.1.1 Frequency of Method Blank:

A Method Blank is prepped once for the following, whichever is more frequent:

- Each batch (not to exceed 20 samples) or
- Whenever samples are analyzed.

10.2 Calibration Check Standards – A low, medium, and high standard are prepared from the same source as the initial calibration and are run every ten samples. The recovery is to be within 10% of the true value.

10.3 Matrix spike and sample duplicate – The RPD between duplicates should be within 20%, if not the analysis is repeated once. If the spike recovery falls outside the acceptable range of 75-125 %, it is re-colored once.

10.3.1 Frequency of matrix spike and sample duplicate

- One every 20 or fewer samples

10.4 Prepare a 5-point curve quarterly, as lot numbers of reagents change, or if the calibration check standard is outside the ± 10% window.

10.5 Dilute samples if they are more concentrated than the highest standard or if highly colored.

10.6 Verify calibration with an independently (second source) prepared check standard before sample analysis. This sample is denoted as the LCS with the recovery limits of ± 20 % of the true value.

10.7 All standards made from a primary standard expire on or before the primary standard's expiration date.

11. Data Validation and Reporting

Results are checked first by the analyst and then by the supervisor. All data are uploaded to the LIMS for reporting. If dissolved results are reported, the DISS box must be checked off in the analytical run.

12. Data Management and Records Management

Electronic data generated from the preparation of Hexavalent Chromium (QC, standards, samples) is saved and managed per **SOP 110.0029** Electronic Data Management.

13. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP is not adhered to:

12.1 If the Calibration Check standard is outside the 10% range, the analysis is repeated. If the check standard fails again, the calibration curve is re-established.

12.2 If the LCS fails, a second aliquot is used. If this fails, the LCS is re-prepared and re-colored. If it still fails, an alternative source is colored. If the alternative source fails the criteria, the curve is re-established.

12.3 Duplicate results outside the 20% RPD window are repeated once in order to confirm matrix effect unless the results are below the reporting limit. These issues will be included in the narrative.

12.4 If the matrix spike falls outside the 75-125 % window, it is repeated once. No further corrective action is required.

14. Health and Safety

Precautions to protect analysts include the nature of toxicity or carcinogenicity of analytes of reagents used in the method. Lab coats, gloves, and safety glasses must be worn at all times in the lab.

15. Pollution Prevention, Waste Management, and Acronyms

See sections 19.0 (Waste Management) and 20.0 (Definitions, Acronyms, and Abbreviations) of the current Quality Assurance Plan.

16. References

APHA, AWWA, and WEF. "Standard Methods for the Examination of Water and Wastewater",22nd Edition, SM 3500 Cr+6 B (2009) Colorimetric Method.

Attachments:

Figure 1: Colorimetric Analysis Logbook

Figure 1: Colorimetric Analysis Logbook

Spectrum Analytical, Inc. RI Div. **Hexavalent Chromium Analysis Logbook**

Analysis Date: **Analyst:**

Sample ID		pH (Aqueous Samples)	Sample Volume (ml)	Absorbance (Abs)	Calc. CONC (mg/l)	DF	Result (mg/l)	% Rec	comments

Wavelength=_____ **Method# : SM3500 Cr B / SW7196A (circle one)** **m=_____**

Pathlength=_____ **b=_____**

MRL=_____ **Conc of Analyte = (Abs-b)/m** **r=_____**

ChromaVer 3 Lot #:_____

Diphenylcarbizide Solution ID:_____

Logbook ID: 100.0056-xx/12 **LIMS entry: _____** **Reviewed By: _____**

Appendix C
Health and Safety Plan Addendum

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Health and Safety Plan Addendum
for
Unrestricted Use Site Characterization
Niagara Falls Air Reserve Station
Niagara Falls, New York

Prepared for

United States Department of the Air Force
Air Force Reserve Command, 914th Airlift Wing
Niagara Falls, New York
and
Air Force Center for Engineering and the Environment
Lackland Air Force Base, Texas

Prepared by

EA Engineering, P.C., and Its Affiliate
EA Science and Technology
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August 2013
Version: FINAL
EA Project No. 62654.01

Health and Safety Plan Addendum
for
Unrestricted Use Site Characterization
Niagara Falls Air Reserve Station
Niagara Falls, New York

Prepared for

United States Department of the Air Force
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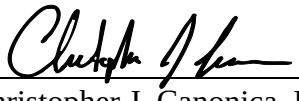


15 August 2013

Christopher J. Canonica, P.E., Program Manager
EA Engineering, P.C.

Date

15 August 2013

Peter Garger CIH, CSP
Corporate Safety and Health Director

Date

August 2013
Version: FINAL
EA Project No. 62654.01

CONTENTS

Page

LIST OF FIGURES

LIST OF ACRONYMS

CFR	Code of Federal Regulations
HASP	Health and Safety Plan
NYANG	New York Air National Guard
NYSDEC	New York State Department of Environmental Conservation
OSHA	Occupational Safety and Health Administration

1. INTRODUCTION

1.1 GENERAL

This Health and Safety Plan (HASP) Addendum is an addendum to the HASP associated with the 2010 Work Plan (EA 2010)[1] for the Installation-Wide Groundwater Monitoring Project for Niagara Falls Air Reserve Station in Niagara Falls, New York. The HASP Addendum contains site-specific information to protect the health and safety of personnel while performing investigations related to the Unrestricted Use Characterizations at sites DS001, DS003, ST009, ST011, TU956, and TU962. Activities to be conducted at these sites include soil and groundwater sampling.

This HASP Addendum describes the safety organization, procedures, and protective equipment that have been established based on an analysis of potential physical, chemical, and biological hazards. Specific hazard control methodologies have been evaluated and selected to minimize the potential for accidents or injuries to occur. One copy of the HASP and this HASP Addendum will be maintained for use during the scheduled field sampling efforts. The copies will be made available for site use and employee review at all times.

This HASP Addendum addresses regulations and guidance practices set forth in the Occupational Safety and Health Administration (OSHA) Standards for Construction Industry, 29 Code of Federal Regulations (CFR) 1926, including 29 CFR 1926.65, *Hazardous Waste Operations and Emergency Response* and 29 CFR 1926.59, *Hazardous Communications*.

The following are provided as attachments:

- **Attachment A**—Worker Training and Physical Examination Record
- **Attachment B**—Health and Safety Plan Review Record
- **Attachment C**—Site Entry and Exit Log
- **Attachment D**—Accident Investigation Report. If an accident occurs, 914th Remedial Program Manager will be contacted and provided this report.
- **Attachment E**—Emergency Telephone Numbers and Hospital Directions
- **Attachment F**—Emergency Equipment Available Onsite
- **Attachment G**—Map to Hospital
- **Attachment H**—Personal Protective Equipment Activity Record.

NOTE: This site-specific HASP Addendum should be left open to display Attachment E (Emergency Telephone Numbers and Hospital Directions) and made available to site personnel in a conspicuous location in the event of an emergency.

[1] EA. 2010. Work Plan for Installation-Wide Groundwater Monitoring Project for Niagara Falls Air Reserve Station, Niagara Falls, New York. August.

1.2 SITE AND FACILITY DESCRIPTION

The Niagara Falls Air Reserve Station is located in Niagara County, New York, approximately 15 mi north of the City of Buffalo, and 6 mi east of the City of Niagara Falls (Figure 1). The base covers approximately 547 acres in the towns of Wheatfield to the east and Niagara to the west (Figure 1). The 914th Airlift Wing has the primary installation mission, and trains reserve officers and airmen for combat-ready status for any national emergency. Current activities include airlifting troops and supplies, providing front line troops with personnel and logistical support, and providing medical evacuations.

1.3 SITE HISTORY

Niagara Falls Air Reserve Station was established as Niagara Falls Air Force Reserve Facility in November 1942. The federal government leased 468 acres of municipal airport land for use by the Army Air Corps. In 1946, 132.2 acres of the leased land were returned to the City of Niagara Falls. The 136th Fighter Squadron of the New York Air National Guard (NYANG) was established on 8 December 1948 and occupied Old Camp Bell near the Bell Aircraft Plant on the installation. The 76th Air Base Squadron was activated on 1 February 1952 as the installation host unit.

On 16 February 1953, the 518th Air Defense Group replaced the 76th Air Base Squadron as the host unit and the NYANG 47th Fighter Interceptor Squadron replaced the 136th Fighter Interceptor Squadron. In August 1955, the U.S. Air Force reactivated the 15th Fighter Group to replace the 518th Air Defense Group. On 1 July 1960, the 15th Fighter Group was deactivated and the 4621st Support Group began operations as the installation host unit. The 4621st Support Group was redesignated as the 4621st Air Base Group on 1 July 1964.

The North American Defense Command Defense System CIM-10B Boeing Michigan Aeronautical Research Center missile was deployed in the western portion of the installation in 1959. The 35th Air Defense Missile Squadron and the missiles were deactivated in the late-1960s, and the NYANG 107th Tactical Fighter Group became the tenant organization occupying the western portion of the installation.

The 49th Fighter Interceptor Squadron, 1 Detachment, assumed responsibility for the installation from the 4621st Air Base Group in March 1970. On 1 January 1971, the installation was transferred from the Aerospace Defense Command to the Air Force Reserve Command and the 914th Tactical Airlift Group became the host unit. The main tenant organization, NYANG 107th Tactical Fighter, was re-designated as the 107th Fighter Interceptor Group. In early-1992, the Niagara Falls Air Force Reserve Facility was renamed the Niagara Falls Air Reserve Station. In late 1993, the 107th Fighter Interceptor Group was re-designated as the 107th Air Refueling Wing and the 914th Tactical Airlift Wing was re-designated as the 914th Airlift Wing.

1.4 POLICY STATEMENT

EA will take every reasonable step to provide a safe and healthy work environment, and to eliminate or control hazards in order to minimize the possibility of injuries, illnesses, or accidents to site personnel. EA and EA subcontractor employees will be familiar with the HASP and this HASP Addendum for each of the project activities they perform. Prior to entering the site, the HASP and this HASP Addendum will be reviewed and an agreement to comply with the requirements will be signed by EA personnel, subcontractors, and visitors (Attachment B).

Operational changes that could affect the health and safety of the site personnel, community, or environment will not be made without approval from EA's Project Manager and Program Health and Safety Officer. This document will be periodically reviewed to ensure that it is current and technically correct. Any changes in site conditions and/or the scope of work will require a review and modification to the HASP Addendum. Such changes will be documented in the form of a revision to this addendum.

2. KEY PERSONNEL

The following table contains information on key project personnel:

Title	Name	Telephone No.
Officer-in-Charge	Gordy Porter	410-584-7000
Program Health and Safety Officer	Peter Garger, CIH	410-527-2425
Regional Program Manager/Consulting Engineer	Chris Canonica, P.E.	315-431-4610
Project Manager	Benjamin Young	770-789-5736
Site Manager	Frank DeSantis Jr.	315-395-7689
Field Team Leader	Lynette Mokry, P.G.	315-430-1786
Site Safety Health Officer	James Peterson	315-727-3308
Quality Assurance Officer	Jen Bouchard, P.G.	315-431-4610
Installation Restoration Program Project Manager	Kim Powell	716-236-3123

3. SCOPE OF WORK

This HASP Addendum was developed to designate and define site-specific health and safety protocols applicable to project activities to be implemented and followed during field activities and consulting work at the Niagara Falls Air Reserve Station, Niagara County, New York. The scope of work covered by this HASP Addendum includes:

- *In situ* groundwater and/or soil sampling at the following sites:
  - DS001 - Site 14, AFRES Hazardous Waste Drum Storage
  - DS003 - Site 12, Bldg. 850 Drum Storage
  - ST009 - Site 4, BX MOGAS Leak
  - ST011 - Site 1, JP-4 Pipeline Leak
  - TU956 - UST 304
  - TU962 - UST 600.

Each of these activities is summarized below and additional detail for each activity is provided in the Sampling and Analysis Plan (EA 2013)[2].

3.1 SOIL AND GROUNDWATER SAMPLING

Subsurface soil samples will be collected using 4-ft acetate sleeves deployed using direct-push drilling techniques and sent to an offsite laboratory for analysis.

Groundwater samples will also be collected using direct-push sample techniques (e.g., the Geoprobe® SP-22 groundwater sampler or similar) and sent to an offsite laboratory for analysis.

3.2 STORAGE AND DISPOSAL OF WASTE

EA is responsible for the proper storage, handling, and disposal of investigative-derived waste including personal protective equipment, and solids and liquids generated during the soil and groundwater sampling. Investigative-derived waste will be managed in accordance with New York State Department of Environmental Conservation (NYSDEC) Division of Environmental Remediation Technical and Administrative Guidance Memorandum 4032 (NYSDEC, 1989)[3].

2 EA. 2013. 2013–2015 Sampling and Analysis Plan, Installation-Wide Groundwater Monitoring Project. Niagara Falls Air Reserve Station, Niagara Falls, New York. April.
[3] NYSDEC. 1989. Technical and Administrative Guidance Memorandum #4032, Disposal of Drill Cuttings. 21 November.

4. POTENTIAL HAZARD ANALYSIS

Based upon the above field activities, the following potential hazard conditions may be anticipated:

- The use of mechanical equipment such as drill rigs, powered augers, and hammer drills can create a potential for crushing and pinching hazards due to movement and positioning of the equipment, movement of lever arms and hydraulics, entanglement of clothing and appendages in exposed drives and augers, and impact of steel tools, masts, and cables should equipment rigging fail or other structural failures occur during hydraulic equipment operation and drilling mast extension and operation. Heavy equipment work must be conducted only by trained, experienced personnel. If possible, personnel must remain outside the turning radius of large, moving equipment. At a minimum, personnel must maintain visual contact with the equipment operator. When not operational, equipment must be set and locked so that it cannot be activated, released, dropped, etc.

- Equipment can be energized due to contact with overhead or underground electrical lines, utilities impaired by excavation of communication or potable/wastewater lines, or a potential for fire or explosion may occur due to excavation of below ground propane/ natural gas lines. Prior to commencement of invasive operations, a drilling/excavation permit will be obtained and the area will be inspected and flagged. Personnel should be aware that although an area may be cleared, it does not mean that unanticipated hazards will not appear. Safe distances will be maintained from live electrical equipment as specified in the HASP (EA 2010)[1]. Workers should always be alert for unanticipated events such as snapping cables, digging into unmarked underground utilities, etc. Such occurrences should prompt involved individuals to halt work immediately and take appropriate corrective measures to gain control of the situation.

- Work around large equipment often creates excessive noise. Noise can cause workers to be startled, annoyed, or distracted; it can cause physical damage to the ear, pain, and temporary and/or permanent hearing loss; and it can interfere with communication. If workers are subjected to noise exceeding an 8-hour time-weighted average sound level of 85 dBA, hearing protection will be selected with an appropriate noise reduction rating to comply with 29 CFR 1910.95 and to reduce noise below levels of concern.

- Personnel may be injured during physical lifting and handling of heavy equipment, construction materials, or containers. Additionally, personnel may encounter slip, trip, and fall hazards associated with excavations, manways, and construction debris and materials. Precautionary measures should be taken in accordance with the HASP (EA 2010)[1]and this HASP Addendum.

- Field operations conducted during the winter months can impose excessive heat loss to personnel conducting strenuous activities during unseasonably cold weather days and can

impose cold-related illness symptoms during unseasonably cold weather days or when wind chill is high. In addition, heavy rains, electrical storms, and high winds may create extremely dangerous situations for employees.

- Entry into a confined space in support of this project is forbidden.

- System operations and field investigation activities intended to define potential sources of environmental contamination often require employees to be in direct proximity or contact with hazardous substances. Employees may be exposed through inhalation of toxic dusts, vapors, or gases. Normal dust particulates from surficial soil may have absorbed or absorbed toxic solvents, petroleum compounds, or toxic metal salts or metal particulates. Air monitoring equipment will be used to monitor airborne organic vapors and particulates. Real-time air monitoring for volatile organic compounds and/or particulate levels at the perimeter of the work area may be necessary. Monitoring activities will consist of a combination of continuous and periodic monitoring, which will be performed dependent upon the type of activity being conducted at the site. Water collected during well development and groundwater sampling activities may also contain toxic vapors, liquids, and gases and be inhaled during normal operations, or may be splashed onto the skin or eyes. Ingestion of toxic materials contained in dusts or particulates can be ingested if eating, smoking, drinking, and gum chewing are permitted prior to personnel washing their hands and face or removing contaminated work clothing and personal protective equipment. Some chemicals may be absorbed directly through the skin. Personal protective equipment, properly designed for the chemicals of concern, will always be provided and worn when a potential for skin contact is present.

The potential constituents of concern that may be present at the sites include volatile organic compounds and metals.

5. PERSONAL PROTECTIVE EQUIPMENT

Based upon currently available information, it is anticipated that Level D protection will be required for currently anticipated conditions and activities. If at any time the sustained level of total organic vapors in the worker breathing zone exceeds 5 parts per million above background, site workers will evacuate the area and the condition will be brought to the attention of the Site Health and Safety Officer. Efforts will then be undertaken to mitigate the source of the vapors. Once the sustained level of total organic vapors has decreased to below 5 parts per million above background, site workers will be allowed to continue activities at the direction of the Site Health and Safety Officer.

The personal protective equipment components for use during this project are detailed in the Work Plan and HASP (EA 2010)[1]. The components of Level D personal protective equipment are summarized below.

5.1 LEVEL D PERSONAL PROTECTIVE EQUIPMENT

Level D will be worn for initial entry onsite and initially for all activities and will consist of the following:

- Coveralls or appropriate work clothing
- Steel-toe, steel-shank safety boots/shoes
- Hard hats (when overhead hazards are present or as required by the Site Health and Safety Officer)
- Chemical resistant gloves (nitrile/neoprene) when contact with potentially contaminated soil or water is expected
- Safety glasses with side shields
- Hearing protectors (during drilling or other operations producing excessive noise)
- Boot covers (optional unless in contact with potentially contaminated soil or water)
- Polycoated coveralls (when contact with contaminated soil and water is anticipated, e.g., when surging/pumping wells and pressure-washing equipment)
- Insulated clothing, hats, etc. must be worn when temperatures or wind chill fall below 40°F.

6. SITE CONTROL AND SECURITY

Only authorized personnel will be permitted to conduct field activities. Authorized personnel include those who have completed hazardous waste operations initial training, as defined under OSHA Regulation 29 CFR 1910.120/29 CFR 1926.65, have completed their training or refresher training within the past 12 months, and have been certified by a physician as fit for hazardous waste operations.

6.1 SAFE WORK PRACTICES

Safe work practices that will be followed by site workers include, but are not limited to, the following rules:

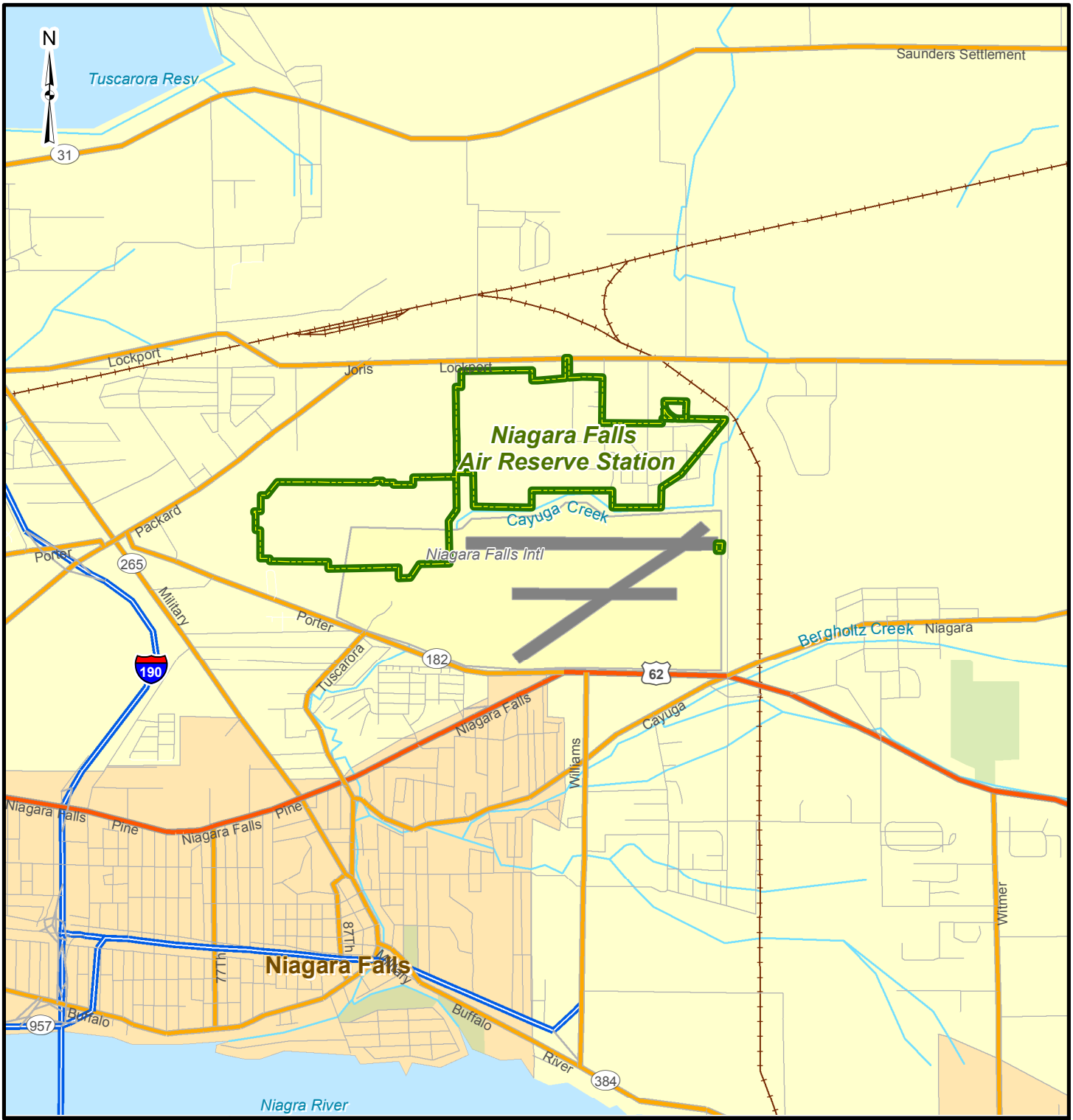
- The work day is from 7:00 a.m. to 4:00 p.m.
- Do not enter restricted or posted areas without permission and escort from base operations.
- Smoking is limited to designated areas.
- Possessing, using, purchasing, distributing, or having controlled substances in their system throughout the day or during meal breaks is prohibited.
- Consuming or possessing alcoholic beverages is prohibited.
- Good housekeeping – employees will be instructed about housekeeping throughout field activities.
- Sitting or kneeling in areas of obvious contamination is prohibited.
- Avoid to the extent possible overgrown vegetation and tall grass areas.
- Entry into flightline areas will only occur when escorted by Niagara Falls Air Reserve Station flightline trained personnel.

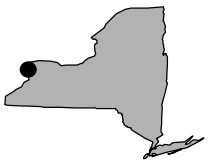
6.2 DAILY STARTUP AND SHUTDOWN PROCEDURES

The following protocols will be followed daily prior to start of work activities:

- The Site Health and Safety Officer will review site conditions to determine if modification of work and safety plans is needed.

- Personnel will be briefed and updated on new safety procedures as appropriate.
- Safety equipment will be checked for proper function.
- The Site Health and Safety Officer will ensure that the first aid kit is adequately stocked and readily available.
- The Contractor is responsible for the security of its own equipment. All onsite equipment and supplies will be locked and secure.





0 0.4 0.8 1.2 1.6 2

Miles

EA®	Health and Safety Plan Addendum Niagara Falls Air Reserve Station Niagara Falls, New York	FIGURE 1 Site Location Map

PROJECT MGR:	DESIGNED BY:	CREATED BY:	CHECKED BY:	SCALE:	DATE:	PROJECT NO:	FILE NO:
BY	FDJR	FDJR	BY	AS SHOWN	AUG 2013	6242107	G:\Projects\Federal\DOD\6265401

Attachment A

Worker Training and Physical Examination Record

ATTACHMENT A

WORKER TRAINING AND PHYSICAL EXAMINATION RECORD

SITE: Niagara Falls Air Reserve Station, Niagara Falls, New York

Name	OSHA 40-Hour Hazardous Waste Operations Training		OSHA Hazardous Waste Supervisor Training	Confined Space Training	CPR (date of expiration)	First Aid (date of expiration)	Date of Last Physical Examination
	Initial	Annual					
EA PERSONNEL							
DeSantis, Frank, --	3/26/07	3/19/13	1/9/12	7/6/10	1/15/2015	1/15/2015	10/21/2011
Miller, Megan, E.	7/12/07	6/5/12	8/28/08	---	1/15/2015	1/15/2015	10/17/2012
Mokry, Lynette, A.	2/23/90	11/21/12	6/94		1/15/2015	1/15/2015	10/13/2011
Peterson, Robert, J.	9/15/08	6/5/12	---	7/6/10	1/08/2013	1/08/2013	9/2009
Williams, Hilary, A.	8/21/09	6/15/12	---	---	1/15/2015	1/15/2015	8/28/2012
Yarrington, Charles, S.	8/31/12	N/A	---	8/31/12	8/30/2014	8/30/2014	5/2012
Young, Benjamin, A.	5/89	6/5/12	6/92	---	1/15/2015	1/15/2015	12/6/2011
SUBCONTRACTOR OR ADDITIONAL PERSONNEL							
---	---	---	---		---	---	---
---	---	---	---		---	---	---

NOTE: Prior to performing work at the site, this Health and Safety Plan must be reviewed and an agreement to comply with the requirements must be signed by all personnel, including contractors, subcontractors, and visitors. Contractors and subcontractors are ultimately responsible for ensuring that their own personnel are adequately protected. All personnel onsite shall be informed of the site emergency response procedures and any potential safety or health hazards of the operations.

Attachment B

Health and Safety Plan
Review Record

ATTACHMENT B

HEALTH AND SAFETY PLAN REVIEW RECORD

I have read the Health and Safety Plan for this site and have been briefed on the nature, level, and degree of exposure likely as a result of participation in this project. I agree to conform to all the requirements of this Plan.

SITE: Niagara Falls Air Reserve Station, Niagara Falls, New York			
Name	Signature	Affiliation	Date

Attachment C

Site Entry and Exit Log

ATTACHMENT C

SITE ENTRY AND EXIT LOG

SITE: Niagara Falls Air Reserve Station, Niagara County, New York

Name	Date	Time of Entry	Time of Exit	Initials

Attachment D

Accident Investigation Report

ACCIDENT/LOSS REPORT

THIS REPORT MUST BE COMPLETED BY THE INJURED EMPLOYEE OR SUPERVISOR AND FAXED TO EA CORPORATE HUMAN RESOURCES WITHIN 24 HOURS OF ANY ACCIDENT. THE FAX NUMBER IS **(410) 771-1780**.

NOTE WHENEVER AN EMPLOYEE IS SENT FOR MEDICAL TREATMENT FOR A WORK RELATED INJURY OR ILLNESS, PAGE 4 OF THIS REPORT MUST ACCOMPANY THAT INDIVIDUAL TO ENSURE THAT ALL INVOICES/BILLS/CORRESPONDENCE ARE SENT TO HUMAN RESOURCES FOR TIMELY RESPONSE.

A. DEMOGRAPHIC INFORMATION:

NAME OF INJURED EMPLOYEE:_____
HOME ADDRESS:_____
HOME PHONE:_____ DATE OF BIRTH:_____
AGE:_____ SEX: M F
MARITAL STATUS:_____ NAME OF SPOUSE (if applicable)_____
SOCIAL SECURITY NUMBER:_____ DATE OF HIRE:_____
NUMBER OF DEPENDENTS:_____
EMPLOYEES JOB TITLE:_____
DEPT. REGULARLY EMPLOYED:_____
WAS THE EMPLOYEE INJURED ON THE JOB: Y N
PRIMARY LANGUAGE OF THE EMPLOYEE: _____

B. ACCIDENT/INCIDENT INFORMATION:

DATE OF ACCIDENT:_____ TIME OF ACCIDENT:_____
REPORTED TO WHOM: _____ NAME OF SUPERVISOR_____
EXACT LOCATION WHERE ACCIDENT OCCURRED (**including street, city, state and County**):
_____
_____
EXPLAIN WHAT HAPPENED (include what the employee was doing at the time of the accident and how the accident occurred): _____
_____
_____
_____

DESCRIBE THE INJURY AND THE SPECIFIC PART OF THE BODY AFFECTED (i.e., laceration, right hand, third finger):
_____
_____

OBJECT OR SUBSTANCE THAT DIRECTLY INJURED EMPLOYEE:_____
_____
NUMBER OF DAYS AND HOURS EMPLOYEE USUALLY WORKS PER WEEK:_____
IS THE EMPLOYEE EXPECTED TO LOSE AT LEAST ONE FULL DAY OF WORK?_____
DOES THE EMPLOYEE HAVE A PREVIOUS CLAIM? Y N if yes, STATUS Open Closed
WAS THE EMPLOYEE ASSIGNED TO RESTRICTED DUTY? _____

C. ACCIDENT INVESTIGATION INFORMATION

WAS SAFETY EQUIPMENT PROVIDED? Y N If yes, was it used? Y N
WAS AN UNSAFE ACT BEING FORMED ? Y N If yes, describe_____
WAS A MACHINE PART INVOLVED? Y N If yes, describe _____
WAS THE MACHINE PART DEFECTIVE? Y N If yes, in what way _____
WAS A 3RD PARTY RESPONSIBLE FOR THE ACCIDENT/INCIDENT? Y N
If yes, list Name, address and phone number_____
_____
WAS THE ACCIDENT/INCIDENT WITNESSED? Y N
If yes, list Name, address and phone number: _____
_____

D. PROVIDER INFORMATION

WAS FIRST AID GIVEN ON SITE? Y N
If yes, what type of medical treatment was given _____
PHYSICIAN INFORMATION (if medical attention was administered)
NAME:_____
ADDRESS (incl. City, state and zip):_____
PHONE:_____

HOSPITAL ADDRESS (incl. Name, address, city, state, zip code & phone)
_____
_____

WAS THE EMPLOYEE HOSPITALIZED? Y N If yes, on what date_____
WAS THE EMPLOYEE TREATED AS AN OUTPATIENT, RECEIVE EMERGENCY TREATMENT OR AMBULANCE SERVICE? _____

PLEASE ATTACH THE PHYSICIANS WRITTEN RETURN TO WORK SLIP

***NOTE* A PHYSICIANS RETURN TO WORK SLIP IS REQUIRED PRIOR TO ALLOWING THE WORKER TO RETURN TO WORK**

E. AUTOMOBILE ACCIDENT INFORMATION (complete if applicable)

AUTHORITY CONTACTED AND REPORT #_____
EA EMPLOYEE VEHICLE YEAR, MAKE AND MODEL_____

V.I.N._____ PLATE/TAG #_____
OWNER'S NAME AND ADDRESS:_____
_____
DRIVER'S NAME AND ADDRESS: _____
_____
RELATION TO INSURED:_____DRIVER'S LICENSE #_____
DESCRIBE DAMAGE TO YOUR PROPERTY:_____
_____
_____
DESCRIBE DAMAGE TO OTHER VEHICLE OR PROPERTY:_____
_____
_____
OTHER DRIVER'S NAME AND ADDRESS: _____
_____
OTHER DRIVER'S PHONE:_____
OTHER DRIVER'S INSURANCE COMPANY AND PHONE:_____
_____
LOCATION OF OTHER VEHICLE:_____
NAME, ADDRESS AND PHONE OF OTHER INJURED PARTIES:_____
_____
_____
WITNESSES

NAME:_____ PHONE:_____
ADDRESS:_____
STATEMENT:_____
_____
SIGNATURE: _____

NAME:_____ PHONE:_____
ADDRESS:_____
STATEMENT:_____
_____
SIGNATURE: _____

F. ACKNOWLEDGEMENT

NAME OF SUPERVISOR:_____
DATE OF THIS REPORT: _____ REPORT PREPARED BY:_____

I have read this report and the contents as to how the accident/loss occurred is accurate to the best of my knowledge.

Signature: _____ Date: _____
Injured Employee

I am seeking medical treatment for a work related injury/illness.

Please forward all bills/invoices/correspondence to:

EA ENGINEERING, SCIENCE, AND TECHNOLOGY, INC.

11019 McCORMICK ROAD

HUNT VALLEY, MD 21031

ATTENTION: Michele Bailey
HUMAN RESOURCES

(410) 584-7000

INCIDENT REPORT

THIS REPORT IS TO BE COMPLETED WHEN A NEAR MISS OCCURS THAT COULD HAVE POTENTIALLY RESULTED IN SERIOUS PHYSICAL HARM. PLEASE FAX THIS FORM TO EA HUMAN RESOURCES DEPARTMENT AT **(410) 771-1780**.

EXPLAIN WHAT HAPPENED (include what the employee was doing at the time the near miss and how it occurred:) _____

REPORT PREPARED BY: _____ DATE:_____

Attachment E

Emergency Telephone Numbers and Hospital Directions

ATTACHMENT E

EMERGENCY TELEPHONE NUMBERS AND HOSPITAL DIRECTIONS

SITE: Niagara Falls Air Reserve Station, 2405 Franklin Drive, Niagara Falls, New York	
Police: Niagara Falls Air Reserve Station Security	(716)-236-2280
Fire: Niagara Falls Air Reserve Station Fire Department	(716)-236-2117
Ambulance:	(716)-236-2117
Hospital: Memorial Hospital, Niagara Falls, New York	(716) 278-4000
New York Regional Poison Control Center: 750 East Adams Street, Syracuse, New York	(315) 723-7000 800-222-1222
Directions to Memorial Hospital, 621 10th Street, Niagara Falls, New York Exit the base at the Lockport Road gate (main gate), turn left on Lockport Road continue 1.4 miles to Packard Road. Continue on Packard Road approximately 3 miles turn right on Pine Avenue continue approximately 0.2 miles, bear right onto Maple Avenue for 1.8 miles. Turn right on 10th Street. Head east on Lafayette Street approximately 1.5 miles. Arrive at Memorial Hospital.	
EA Program Safety and Health Officer ***Peter Garger, CIH***	(410) 527-2425
Program Manager: ***Christopher Canonica, P.E.***	(315) 431-4610
Niagara Falls ARS Project Manager: **Kim Powell/Ellen Marien**	(716) 236-3123/(716) 236-3126
Versar Project Manager: ***Nathan Mullens***	(843) 338-1851
EA Project Manager: ***Ben Young***	(770) 789-5736
EA Medical Services All One Health ***Contact: Dr. Jerry Berke***	800-350-4511
Site Manager/Site Health and Safety Officer ***Frank DeSantis Jr.***	(315) 395-7689
In case of accident or exposure incident, contact EA Corporate Health and Safety Officer ***Peter Garger, CIH***	(410) 527-2425

Attachment F

Emergency Equipment Available Onsite

ATTACHMENT F

EMERGENCY EQUIPMENT AVAILABLE ONSITE

Type of Equipment	Location
Communications Equipment	
Mobile Telephone	In EA vehicle
Medical Support Equipment	
First Aid Kits	In EA vehicle/Site 10 Trailer
Eye Wash Station	In EA vehicle/Site 10 Trailer
Fire Fighting Equipment	
Fire Extinguishers	In EA vehicle/Site 10 Trailer

Attachment G

Map to Hospital

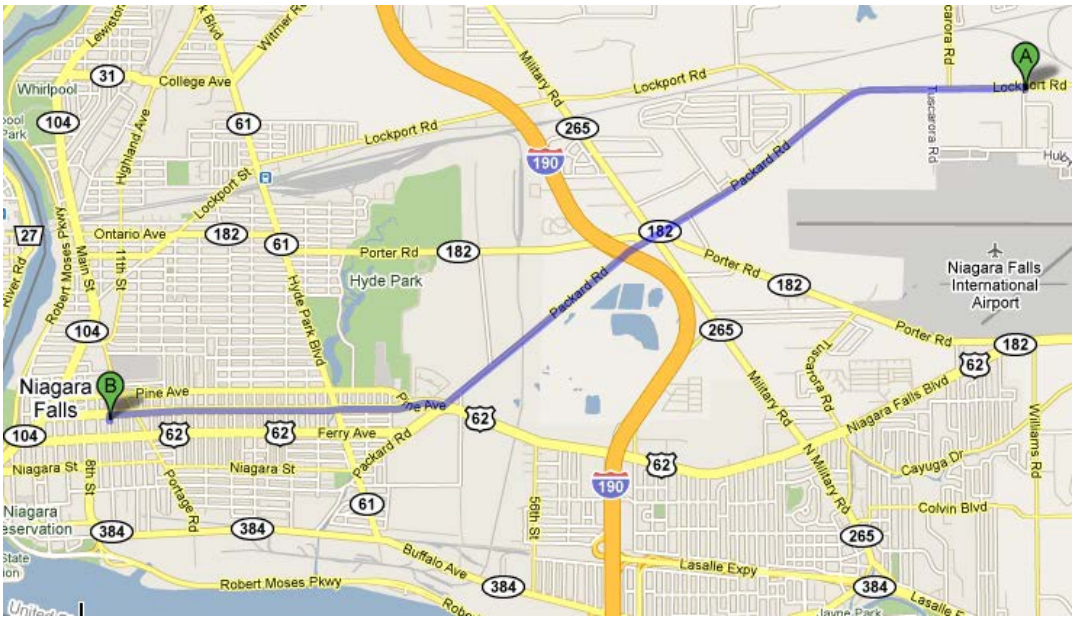
ATTACHMENT G

MAP TO HOSPITAL

Directions to Niagara Falls Memorial Medical Center, 621 10th St Niagara Falls, NY 14301

Exit the base at the Lockport Road gate (main gate), turn left on Lockport Road continue 1.4 miles to Packard Road. Continue on Packard Road approximately 3 miles turn right on Pine Avenue continue approximately 0.2 miles, bear right onto Maple Avenue for 1.8 miles. Turn right on 10th Street. Head east on Lafayette Street approximately 1.5 miles. Arrive at Memorial Hospital.

Total trip is 6.2 miles; travel time is approximately 15 minutes.



Attachment H

Personal Protective Equipment Activity Record

ATTACHMENT B

PERSONAL PROTECTIVE EQUIPMENT ACTIVITY RECORD

SITE: Niagara Falls Air Reserve Station, Niagara Falls, New York		
Weather Condition:		Onsite Hours: From To
Changes in Personal Protective Equipment Levels[(a)]	Work Operations	Reasons for Change
Site Health and Safety Plan Violations	Corrective Action Specified	Corrective Action Taken (yes/no)
Observations and Comments:		
Completed by: _____ Site Health and Safety Officer		Date
(a) Only the Site Health and Safety Officer may change personal protective equipment levels, using only criteria specified in the Health and Safety Plan.		