QUALITY ASSURANCE PROJECT PLAN

HOOKER CHEMICAL/RUCO POLYMER SUPERFUND SITE
HICKSVILLE, NEW YORK
U.S. EPA INDEX NO: CERCLA-02-2001-2018

Prepared for:

Glenn Springs Holdings, Inc.

DECEMBER 2011
REF. NO. 006883 (57)
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ATTACHMENTS

QAPP Worksheet #1
(UFP-QAPP Section 2.1)
Title and Approval Page

Site Name/Project Name:	Hooker Chemical/Ruco Polymer Superfund Site
Site Location:	Hicksville, New York
Document Title:	Quality Assurance Project Plan Hooker Chemical/Ruco Polymer Superfund Site in Hicksville, New York
Lead Organization:	Conestoga-Rovers & Associates, Inc. (CRA)
Preparer's Name and Organizational Affiliation:	Angela Bown, Conestoga-Rovers & Associates, Inc. (CRA)
Preparer's Address:	9033 Meridian Way, West Chester, Ohio 45069
Telephone Number:	513-942-4750
E-mail Address:	abown@craworld.com
Preparation Date:	October 14, 2011

Roger Smith — 12/27/2011
Project Director
(Roger Smith – Glenn Springs Holdings, Inc.) — Date

Jim Kay — Dec 21/11
Project Manager
(Jim Kay – Conestoga-Rovers & Associates, Inc.) — Date

Diane Schmidtke — Dec 21, 2011
Project Coordinator
(Diane Schmidtke - Conestoga-Rovers & Associates, Inc.) — Date

12/23/2011
Project QA/QC Officer – Analytical Activities
(Denise R. Anderson - Conestoga-Rovers & Associates, Inc) — Date

12/21/2011
Project QA/QC Officer – Field Activities
(Victoria Whelan – C. A. Rich, Inc) — Date

12/23/11
Laboratory Project Manager
(Shirley Ng - Spectrum Analytical, Inc.) — Date

Sharyn Lawler — 12/23/11
Laboratory QA/QC Officer
(Sharyn Lawler - Spectrum Analytical, Inc.) — Date

Remedial Project Manager
(Thomas Tascone - USEPA Region II) — Date

Document Control Numbering System: 006883-UFP QAPP-001

QAPP Worksheet #2
(UFP-QAPP Section 2.2.4)
QAPP Identifying Information

Site Name/Project Name: Hooker Chemical/Ruco Superfund Site
Site Location: Hicksville, New York
Site Number/Code: NA
Operable Unit: OU-3
Contractor Name: Conestoga-Rovers & Associates, Inc.
Contractor Number: NA
Contract Title: NA
Work Assignment Number: NA

Title: Hooker/Ruco Site QAPP
Revision Number: 0
Revision Date: 10/21/11

1. Identify regulatory program: Comprehensive Environmental Response, Compensation and Liability Act of 1980

2. Identify approval entity: USEPA Region II

3. The QAPP is (select one): ☐Generic ☒Project Specific

4. List dates of scoping sessions that were held: None

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

Title	Approval Date
Quality Assurance Project Plan-OU-3 Biosparge Remedy	May 2005

6. List organizational partners (stakeholders) and connection with lead organization:
USEPA Region II, Glenn Springs Holdings, Inc.(GSHI)

7. List data users:
USEPA Region II, CRA, GSHI, NYSDEC, C.A. Rich, Langan, Northrop Grumman, and Sleepys

8. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusions below:
N/A

QAPP Worksheet #2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
Heading4Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	Worksheet (WS) #1
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	Table of Contents List of Attachments WS #2
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	WS #3 WS #4
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	WS #5 WS #6 WS #7 WS #8
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	WS #9 WS #10
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria Table	WS #11 WS #12 Attachment D - Laboratory Deliverables Requirements

QAPP Worksheet #2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
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2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table	2.8.1 - Attachment C - Analytical Data Flow WS #14 WS #15 WS #16
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3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	- Analytical SOPs - Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Attachment A - List of Spectrum Lab's Applicable Standard Operating Procedures WS #23 WS #24 WS #25

QAPP Worksheet #2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Required Documents
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QAPP Worksheet #2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
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5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment	WS #34 WS #35 WS #36 WS #37
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining		

QAPP Worksheet #3
(UFP-QAPP Manual Section 2.3.1)

Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Thomas Taccone	Remedial Project Manager USEPA Region II	USEPA Region II	212-637-4281		Taccone.tom@epamail.epa.gov	
Roger Smith	Project Director	GSHI	972-687-7516	972-687-7524	roger_smith@oxy.com	
Jim Kay	Project Manager	Conestoga-Rovers & Associates, Inc.	519-884-0510	519-884-0525	jkay@craworld.com	
Klaus Schmidtke	Project Coordinator	Conestoga-Rovers & Associates, Inc.	519-884-0510	519-884-0525	kschmidtke@craworld.com	
Denise R. Anderson	QA/QC Officer-Analytical Activities	Conestoga-Rovers & Associates, Inc.	716-297-2160	716-297-2265	Danderson@craworld.com	
Victoria Whelan	QA/QC Officer-Field Activities	C. A. Rich, Inc.	516-576-8844	516-576-0093	vwhelan@carichinc.com	
Kathy Willy	Data Validator	Conestoga-Rovers & Associates, Inc.	716-297-2160	716-297-2265	kwilly@craworld.com	
Shirley Ng	Laboratory Project Manager	Spectrum Analytical, Inc.	401-732-3400		sng@mitkem.com	
Sharyn Lawler	Laboratory QA/QC Manager	Spectrum Analytical, Inc.	401-732-3400		Slawler@mitkem.com	
Daniel McKenna	Laboratory Sample Custodian	Spectrum Analytical, Inc.	40-1732-3400		dmckenna@mitkem.com	
Sophia Dore	HPC Analyst	Conestoga-Rovers & Associates, Inc.	716-297-2160	716-297-2265	sdore@craworld.com	
Steven Scharf	Project Manager – Northrop Site	NYSDEC	518-402-9620		sxscharf@gw.dec.state.ny.us	
Ryan Andersen	QA/QC Officer, Langan	Langan	201-794-6900		randersen@langan.com	

QAPP Worksheet #4
(UFP-QAPP Manual Section 2.3.2)

Project Personnel Sign-Off Sheet

Organization: USEPA Region II

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Thomas Taccone	Remedial Project Manager USEPA Region II	212-637-4281		

QAPP Worksheet #4
(UFP-QAPP Manual Section 2.3.2)

Project Personnel Sign-Off Sheet

Organization: GSHI

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Roger Smith	Project Director	(972) 687 7516	Roger Smith	12/21/2011

QAPP Worksheet #4
(UFP-QAPP Manual Section 2.3.2)

Project Personnel Sign-Off Sheet

Organization: C.A. Rich, Inc

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Victoria Whelan	QA/QC Officer - Field Activities	516-576-8844		12/23/2011

QAPP Worksheet #4
(UFP-QAPP Manual Section 2.3.2)

Project Personnel Sign-Off Sheet

Organization: Conestoga-Rovers & Associates, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Jim Kay	Project Manager	519-884-0510	Jim Kay	Dec 21/11
Klaus Schmidtke	Project Coordinator	519-884-0510	Klaus Schmidtke	Dec 21, 2011
Denise R. Anderson	QA/QC Officer – Analytical Activites	716-297-2160	Denise R. Anderson	12/23/2011
Sophia Dore	HPC Analyst	716-297-2160	Sophia Dore	01/03/2012
Kathy Willy	Data Validator	716-297-2160	Kathy Willy	12/23/11

QAPP Worksheet #4
(UFP-QAPP Manual Section 2.3.2)

Project Personnel Sign-Off Sheet

Organization: Spectrum Analytical, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Shirley Ng	Laboratory Project Manager	401-732-3400		
Sharyn Lawler	Laboratory QA/QC Officer	401-732-3400		

QAPP Worksheet #4
(UFP-QAPP Manual Section 2.3.2)

Project Personnel Sign-Off Sheet

Organization: NYSDEC

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Steven Scharf	Project Manager – Northrop Site	518-409-9620		

QAPP Worksheet #4
(UFP-QAPP Manual Section 2.3.2)

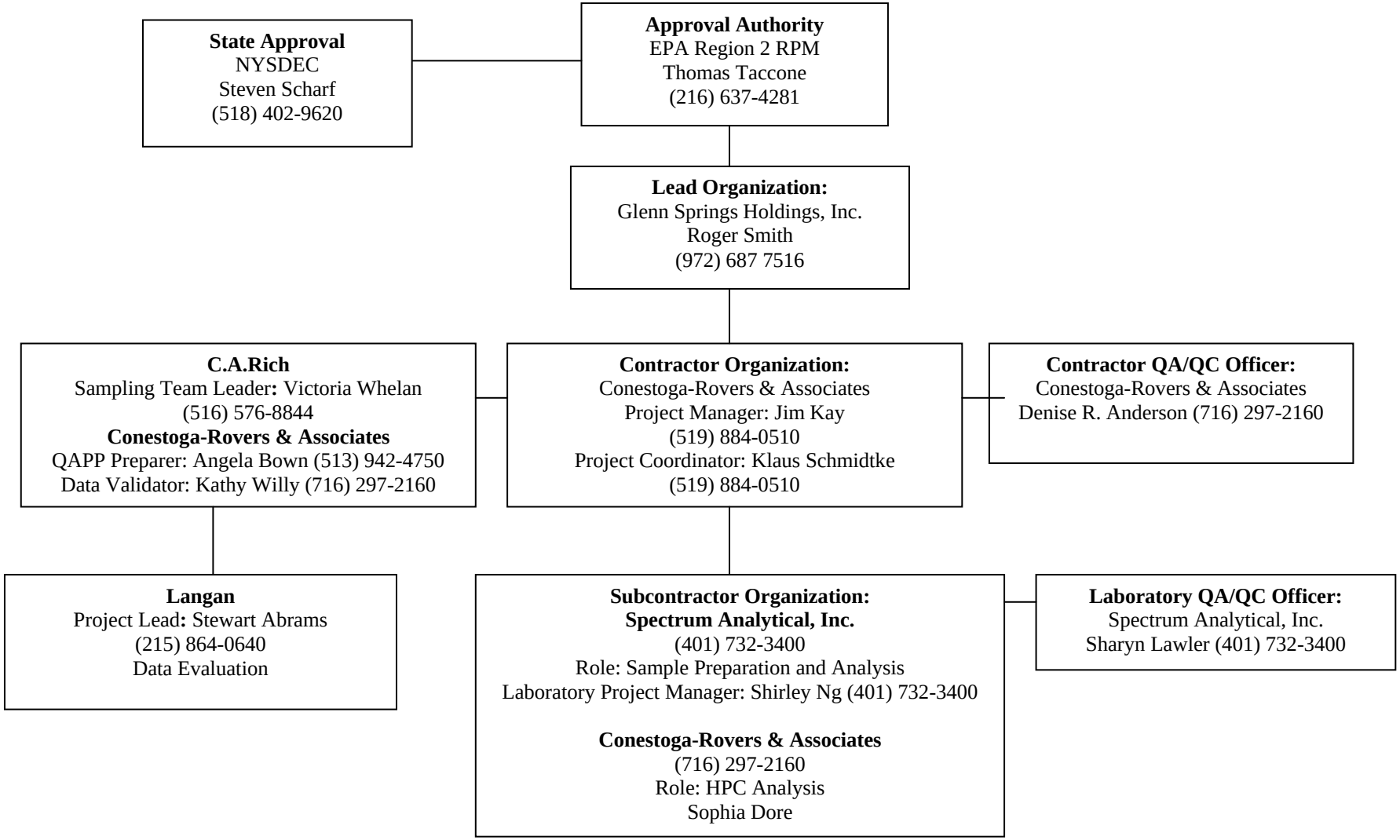
Project Personnel Sign-Off Sheet

Organization: Langan

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Ryan Andersen	QA/QC Officer – Langan	201-794-6900		12/23/11
Stewart Abrams	Data Validator	215-864-0640		12/29/11

QAPP Worksheet #5
(UFP-QAPP Manual Section 2.4.1)

QAPP Worksheet #5
Project Organizational Chart



QAPP Worksheet #6
(UFP-QAPP Manual Section 2.4.2)

Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Point of contact with USEPA and GSHI	Project Director	Roger Smith	972-687 7516	Data and information about the project will be forwarded to USEPA by Project Director.
Manage all project phases	Project Manager	Jim Kay	519-884-0510	Notify Project Director and Project Coordinator of field related problem by phone, email or fax by COB the next business day or earlier as required by the Consent Decree.
QAPP changes in the field Initiate work stoppage	Field QA/QC Officer	Victoria Whelan	516-576-8844	Notify Project Manager by phone/email of changes to QAPP and work stoppages made in the field and the reasons as soon as practicable. All QAPP changes in the field will be forwarded to USEPA by Project Director.
Daily Field Progress Reports	Field QA/QC Officer	Victoria Whelan	516-576-8844	Daily field progress reports will be verbal, e-mailed, hand delivered or faxed to Project Manager, as required by the tasks.
Reporting lab data quality issues	Laboratory QA/QC Officer	Sharyn Lawler	401-732-3400	All QA/QC issues with the project field samples will be reported to Data Validator and Project Managers within 2 business days.
Field and analytical corrective actions	Project Manager	Jim Kay	519-884-0510	The need for corrective action for field and analytical issues will be determined by Project Manager. All field and analytical corrective actions will be forwarded to Project Director by Project Manager.
Release of analytical data	Project Coordinator	Klaus Schmidtke	519-884-0510	Analytical data will be transmitted to USEPA following data validation unless otherwise directed by USEPA.
QAPP Amendments	Remedial Project Manager Region II	Thomas Taccone	212-637-4281	Any major changes to the QAPP must be approved by USEPA before the changes can be implemented. All QAPP amendments will be forwarded to USEPA by Project Coordinator.

QAPP Worksheet #7
(UFP-QAPP Manual Section 2.4.3)

Personnel Responsibilities and Qualification Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Roger Smith	Project Director	GSHI	Oversees project and corresponds with USEPA and Performing Parties	B.B., Chemical Engineering, 15+ years experience in environmental site management and coordination.
Jim Kay	Project Manager	Conestoga-Rovers & Associates	Oversees project and corresponds with project coordinator, project manager, and USEPA	B.A.Sc., Civil Engineering, 30+ years experience in environmental site assessment and remediation/project management.
Klaus Schmidtke	Project Coordinator	Conestoga-Rovers & Associates	Oversees project and corresponds with project director, project coordinator, field personnel, subcontractors, and USEPA	B.A.Sc., M.A.Sc., Ph.D, Civil Engineering, 25+ years experience in environmental site assessment and remediation/project management.
Denise R. Anderson	QA/QC Officer – Analytical Activities	Conestoga-Rovers & Associates	Review field and laboratory QA/QC procedures. Oversee corrective action process. Conduct external audits	B.S., Chemistry, 25+ years experience in analytical chemistry, data validation, and analytical support services.
Kathy Willy	Data Validator	Conestoga-Rovers & Associates	Conduct data validation and prepare validation reports. Assist QA/QC Officer with external audits	B.S., Chemistry, 26+ years experience in analytical chemistry, laboratory operations, and data validation.
Victoria Whelan	QA/QC Officer - Field Activities	C.A.Rich	Schedule and oversee field work.	B.S., Geology, 5+ years environmental experience.
Shirley Ng	Laboratory Project Manager	Spectrum Analytical, Inc.	Sample receipt, analysis, and reporting.	B.S., Computer Oriented Mathematics, 7+ years experience in the environmental laboratory industry.
Sharyn Lawler	Laboratory QA/QC Officer	Spectrum Analytical, Inc.	Oversee and implement QA program	B.S., Coastal Plant Ecology, 20+ years experience in the environmental laboratory industry.
Daniel McKenna	Laboratory Sample Custodian	Spectrum Analytical, Inc.	Sample receipt, handling, documentation, and log-in of incoming samples.	High School Diploma, 1 year experience as sample receiving technician.
Sophia Dore	Laboratory Analyst	Conestoga-Rovers & Associates	Sample receipt, analysis, and reporting.	Ph.D., Biology, 15+ years environmental experience

Personnel Responsibilities and Qualification Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Stewart Abrams	Project Lead	Langan	Assist with data validation.	B.S. Civil Engineering; M.S. Environmental Science; 29+ years experience in environmental remediation and management

QAPP Worksheet #8
(UFP-QAPP Manual Section 2.4.4)

Special Personnel Training Requirements Table

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Groundwater/Soil Gas Sampling/Site Work	OSHA HAZWOPER (also refer to HASP)	Various	Varies	Groundwater/Soil Gas Sampling Staff	CRA/C.A.Rich	CRA Waterloo office/field vehicle or on site control building
Groundwater /Soil Gas Sampling/Supervision	OSHA HAZWOPER and 8-Hour Supervisor Training (also refer to HASP)	Various	Varies	Supervisor/Site Representative	CRA/C.A.Rich	CRA Waterloo office/field vehicle or on site control building
Drilling/Soil Sampling and Well Installation	OSHA HAZWOPER (also refer to HASP)	Various	Varies	Drillers/Helpers	TBD	CRA Waterloo office/field vehicle or on site control building
Agency Oversight	OSHA HAZWOPER (also refer to HASP)	Various	Varies	On-Site Representatives	USEPA	Varies (must be available and up to date to enter site)

QAPP Worksheet #9
(UFP-QAPP Manual Section 2.5.1)

☑ Worksheet Not Applicable (State Reason)

No scoping session was conducted

Project Scoping Session Participants Sheet

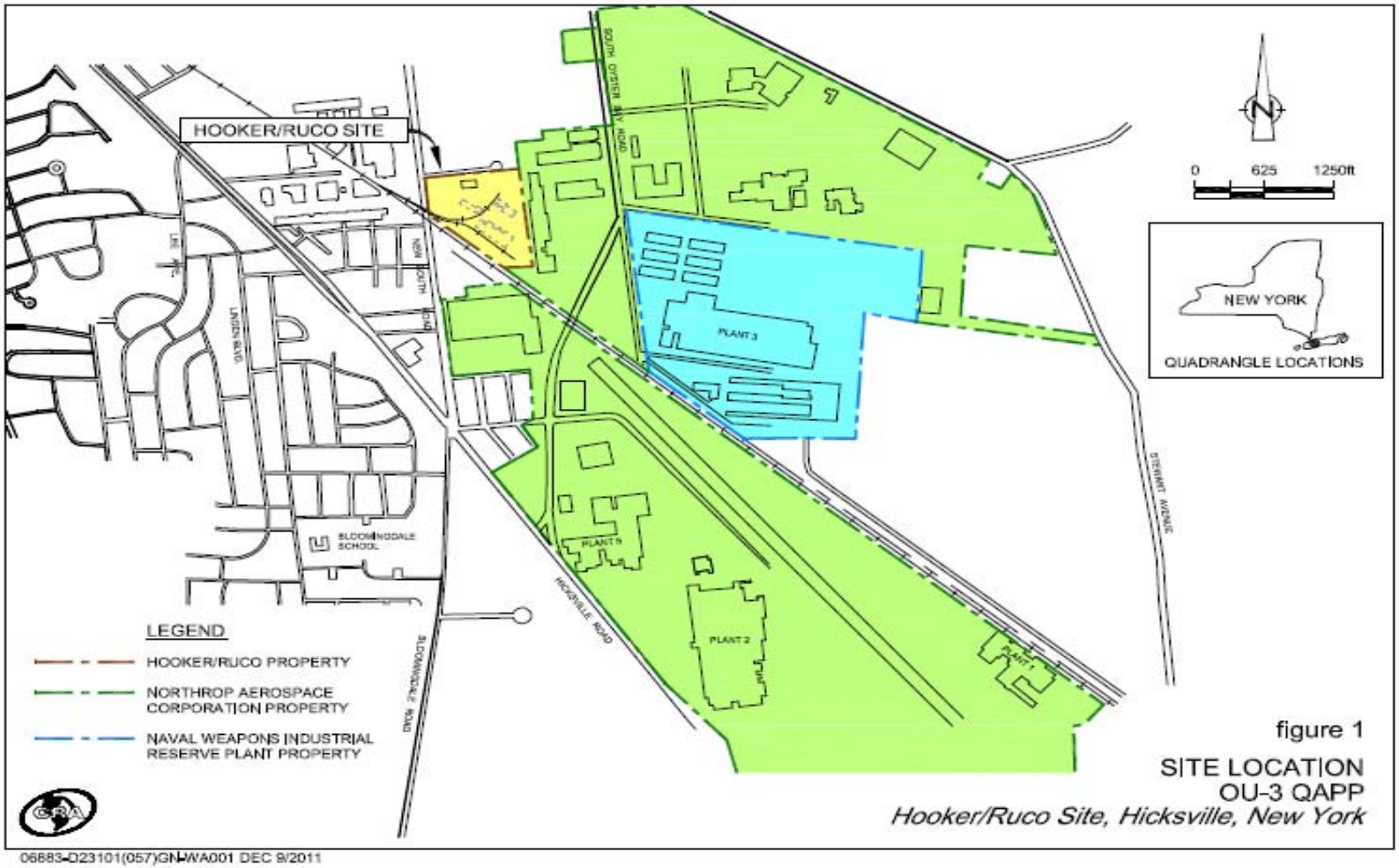
Project Name: Hooker/Ruco OU-3 Biosparge Remedy **Projected Date(s) of Sampling:** **Project Manager:** Jim Kay	**Site Name:** Hooker/Ruco Superfund Site **Site Location:** Hicksville, New York
Date of Session: **Scoping Session Purpose:** Overall project discussion.	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role

Site Description: The Hooker/Ruco Site is a 14 acre former polymer manufacturing facility located in a heavily industrialized section of Hicksville, Long Island, New York. The facility is being closed by Bayer under RCRA with oversight provided by the NYSDEC. Historically, the major industrial facility in the area was the Northrop manufacturing facility and airport. The Northrop plant is now shut down and Northrop is in the process of selling parcels of their property to other parties. There are many other small industries, commercial operations, residential areas, utilities, transportation corridors, and storm-water management basins in the area. Figure 1 (below) shows the Hooker/Ruco Site and its surroundings. Commerce Street and adjacent industrial development comprise the 880-foot northern site boundary. Along the site's 1000-foot eastern side is a large warehouse building formerly owned by Northrop. A small portion of undeveloped land abuts the site's 250-foot southern property boundary. Two active tracks of the Long Island Railroad parallel the site's 940-foot southwestern property boundary. The Hooker/Ruco Site is bounded on the 270-foot western boundary by New South Road. The property is enclosed by a chain link fence, which completely encompasses the site. The area surrounding the site is comprised of an industrialized corridor and residential complexes. Residential dwellings comprise approximately 22 percent of the area and are located southwest of the site. Approximately 65 percent of the area land is industrial or commercial.

Comments/Decisions: Sampling is to be performed for an anticipated period of 12 years after the entire biosparge system becomes operational. Operation of the entire biosparge system is anticipated to start in the summer/fall of 2012.

Action Items: Submittal


HOOKER/RUCO SITE
SOUTH OYSTER BAY ROAD
NEW SOUTH ROAD
LINDEN BLVD
BLOOMINGDALE SCHOOL
PLANT 3
PLANT 5
PLANT 2
PLANT 1
HICKSVILLE ROAD
STEWART AVENUE
BLOOMINGDALE ROAD
N
0 625 1250ft
NEW YORK
QUADRANGLE LOCATIONS
LEGEND
HOOKER/RUCO PROPERTY
NORTHROP AEROSPACE CORPORATION PROPERTY
NAVAL WEAPONS INDUSTRIAL RESERVE PLANT PROPERTY
CRA
figure 1
SITE LOCATION
OU-3 QAPP
Hooker/Ruco Site, Hicksville, New York
06683-023101(057)GN-WA001 DEC 9/2011

QAPP Worksheet #10
(UFP-QAPP Manual Section 2.5.2)

Problem Definition

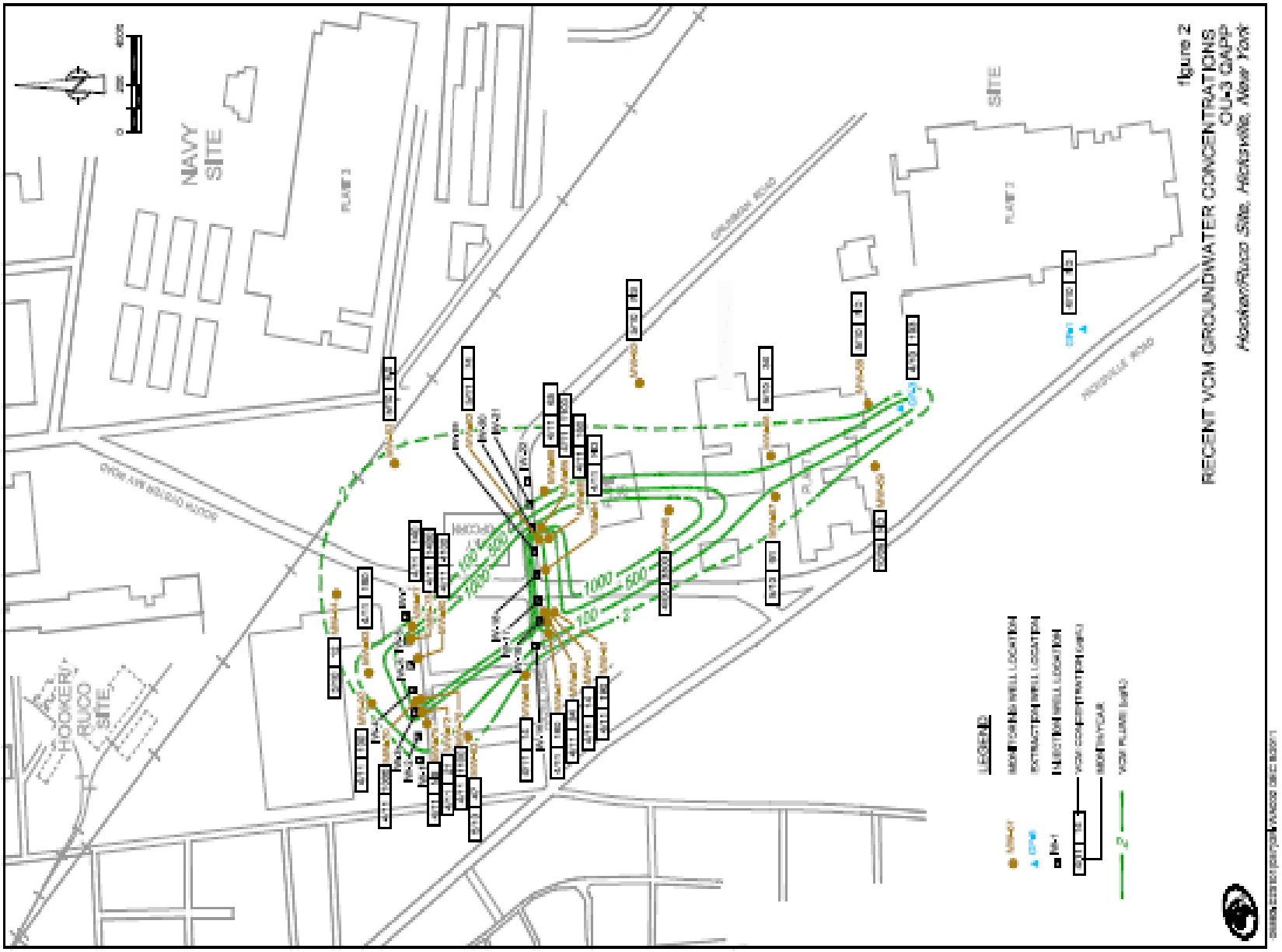
The problem to be addressed by the project: The site operated between 1945 and 2002 during which time some chemical releases into the hydrogeologic environment occurred. Some impacts due to the historic chemical releases persist in the groundwater. The groundwater impact has migrated off Site and is now commingled within the regional groundwater. The studies that have been performed over the years have defined the horizontal and vertical extent of the chemical plume emanating from the Site which is primarily characterized as a vinyl chloride monomer (VCM) plume. The Record of Decision (ROD) that was issued for the Site in 2000 determined that the appropriate remedy for the off-site groundwater plume of VCM (i.e., OU-3) would incorporate the use of in-situ biosparge. Considering that the VCM has migrated to well GP-3, the remedy has evolved into a dual remediation program involving both biosparge and pump/treat technologies for the northern and southern portions of the VCM plume, respectively. GSHI has joined efforts with Northrop Grumman Corporation to address the leading (southernmost) portion of the VCM plume using Northrop Grumman's existing GP-1/GP-3 pump and treat facility. Northrop Grumman's facility is being operated to address Northrop Grumman's groundwater plume of volatile organic chemicals (VOCs). With some modification of the Northrop Grumman treatment facility, the facility capably addresses the commingled Hooker/Ruco Site VCM plume and the Northrop Grumman VOC plume. These modifications are complete. Due to the relative size difference between the regional VOC plume and the VCM plume and the fact that the VCM plume is entirely encompassed within the lateral limits of the regional VOC plume, the VCM plume is often referred to as a subplume. The estimated areal extent of the VCM subplume in 2011 is shown in Figure 2 (below). The VCM concentrations on Figure 2 show the effectiveness of the Biosparge Pilot System in reducing VCM concentrations. The Biosparge Pilot System has been operational since October 2006.
The environmental questions being asked: Is the remedy reducing groundwater concentrations to meet USEPA/NYSDEC criteria?
Observations from any site reconnaissance reports: N/A
A synopsis of secondary data or information from site reports: Data uased to delineate areal and vertical extent of VCM subplume
The possible classes of contaminants and the affected matrices: VOCs in groundwater and soil gas. The primary contaminates of concern are: cis-1,2-dichloroethene, tetrachloroethene, trichloroethene, and vinyl chloride.
The rationale for inclusion of chemical and non-chemical analyses: To monitor the effectiveness of the biosparge system and groundwater quality.
Information concerning various environmental indicators: N/A
Project decision conditions ("If..., then..." statements): 1) If groundwater meets USEPA/NYSDEC groundwater standards, then the biosparge system can be shut down at the corresponding fence. **2)** If post shutdown monitoring shows a rebound in VOC concentration, then operation at that fence of the biosparge system will be restarted.

NOTE: Additional problem definition and biosparge system design details can be found in the 100% Final Design Report dated May 2005.

QAPP Worksheet #11
(UFP-QAPP Manual Section 2.6.1)

Project Quality Objectives /Systematic Planning Process Statements

Who will use the data? Conestoga-Rovers & Associates, Inc., GSHI, USEPA, NYSDEC, C.A. Rich, Langan, Northrop, Sleepys
What will the data be used for? The data will be used to support the effectiveness of the biosparge remedy for Operable Unit-3 (OU-3) at the Hooker Chemicals/Ruco Polymer Superfund Site located in Hicksville, New York.
What types of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques) Groundwater samples will be collected and analyzed for VOCs with Tentatively Identified Compounds (TICs), Ammonia as Nitrogen, Nitrate/Nitrite as Nitrogen, Phosphorus, Total Organic Carbon (TOC), and heterotrophic plate counts (HPC). Soil Gas samples will be collected and analyzed for VOCs by method TO-15 and Methane. Field parameters will include Fe2+ (by Hach kit), pH, DO, ORP, temperature, conductivity and turbidity.
How "good" do the data need to be in order to support the environmental decision? Data rejected based on the data validation process will not be used in the decision-making process.
How much data are needed? (number of samples for each analytical group, matrix, and concentration) Groundwater samples will be collected from up to 59 monitoring wells (as available) on a quarterly, semi-annual, and annual basis and will be analyzed for VOCs with TICs, Ammonia as Nitrogen, Nitrate/Nitrite as Nitrogen, Phosphorus, TOC, and HPC. Soil gas samples will be collected from 14 locations on a semi-annual basis and will be analyzed for VOCs by method TO-15 and Methane. Any subsequent sampling events that fall within the scope of the 100% Final Design Report shall also follow the requirements of this QAPP.
Where, when, and how should the data be collected/generated? Requirements are outlined in the 100% Final Design Report. See Worksheet 18 and Figures 2, 3 & 4 below for details.
Who will collect and generate the data? C.A. Rich will collect the samples and submit to Spectrum Laboratories for analyses.
How will the data be reported? The laboratory shall submit two copies of a final complete analytical report within 21 calendar days of sample receipt. The analytical report submitted by the laboratory shall conform to all reporting and deliverable requirements in a format as similar to CLP as possible. See Attachment D for Laboratory Deliverables Requirements.
How will the data be archived? Evidentiary files for the data shall be inventoried and maintained by CRA and shall consist of the following: Off-Site Groundwater Predesign Information Report, 100% Final Design Report; and supporting plans; project logbooks; field data records; sample identification documents; chain-of-custody records; laboratory data; correspondence; report notes, calculations, etc; references, copies of pertinent literature; miscellaneous photos, maps, drawings, etc; and final report. The evidentiary file material shall be the responsibility of the CRA Project Manager with respect to archiving.


NAVY SITE
PLANT 1
CRIMSON ROAD
NIAGARA FALLS BOULEVARD
HOOKER RUCO SITE
SITE
PLANT 1
ROBERTS ROAD
LEGEND
VOC PLUME (ug/L)
Figure 2
RECENT VOM GROUNDWATER CONCENTRATIONS
OU-3 QAPP
Hooker/Ruco Site, Niagara Falls, New York

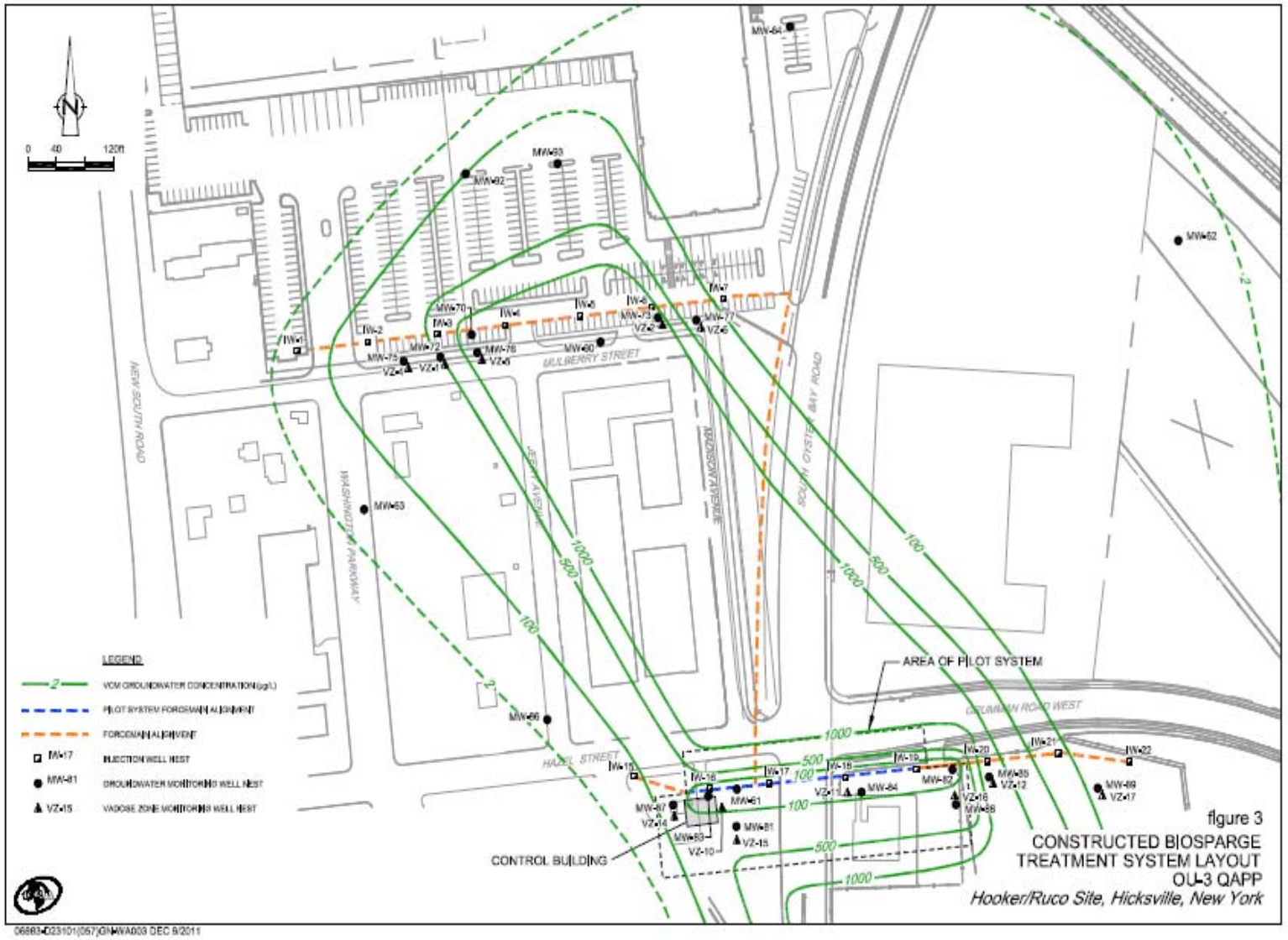


figure 3
CONSTRUCTED BIOSPARGE TREATMENT SYSTEM LAYOUT
OU-3 QAPP
Hooker/Ruco Site, Hicksville, New York

DURATION / ACTIVITY	2012			2013				2014				2015 to 2025			
	APR	JUL	OCT	JAN	APR	JUL	OCT	JAN	APR	JUL	OCT	JAN	APR	JUL	OCT
PILOT SYSTEM	*														
REMAINDER OF SYSTEM (1)				*			*				*				*
ENTIRE SYSTEM (1)			*			*				*				*				*

NOTE:
(1) SCHEDULE ASSUMES REMAINDER OF SYSTEM BECOMES OPERATIONAL IN LATE SUMMER/EARLY FALL 2012.

figure 4

ANTICIPATED BIOSPARGE SYSTEM PERFORMANCE MONITORING SCHEDULE
OU-3 QAPP
Hooker/Ruco Site, Hicksville, New York

06883-D23101(057)GN-WA004 DEC 9/2011

QAPP Worksheet #12
(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group	Volatiles plus TICs (8260C)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
001	A1	System Performance/Sensitivity	BFB Tune Criteria must be met per 8260C	BFB Tune	A
001	A1	Accuracy/Bias	%RSD and %D for each individual Calibration Check Compound (CCC) must be met per 8260C	Calibration Standards	A
001	A1	Sensitivity	Minimum Mean Response Factors for SPCCs must be met per 8260C	Calibration Standards	A
001	A1	Precision	RPD≤ 35%	Field Duplicates	S & A
001	A1	Accuracy/Bias	50-200% Recovery	Internal Standards	A
001	A1	Precision	Laboratory Limits	Matrix Spikes (in duplicate)	A
001	A1	Accuracy/Bias	Laboratory Limits	Surrogates	A
001	A1	Accuracy/Bias	Laboratory Limits	Laboratory Control Sample-2nd Source	A
001	A1	Contamination	<QL or <5 x QL for common lab contaminants	Laboratory Blanks	A
001	A1	Contamination	<QL or <5 x QL for common lab contaminants	Field QA/QC Blanks	S

QAPP Worksheet #12
(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria
(continued)

Matrix	Soil Gas
Analytical Group	VOCs (TO-15)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
002	A2	System Performance/Sensitivity	BFB Tune Criteria must be met per TO-15.	BFB Tune	A
002	A2	Accuracy/Bias	%RSD and %D for each individual Calibration Check Compound (CCC) must be met per TO-15	Calibration Standards	A
002	A2	Sensitivity	Minimum Mean Response Factors for SPCCs must be met per TO-15	Calibration Standards	A
002	A2	Accuracy/Bias	50-200% Recovery	Internal Standards	A
002	A2	Accuracy/Bias	Laboratory Limits	Laboratory Control Sample-2nd Source	A
002	A2	Accuracy/Bias	Laboratory Limits	Surrogates	A
002	A2	Contamination	<QL or <5 x QL for common lab contaminants	Laboratory Blanks	A

QAPP Worksheet #12
(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table
(continued)

Matrix	Soil Gas
Analytical Group	Methane (Modified EPA Method 3C)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
002	A3	Accuracy/Bias	$\leq$ 30%RSD or R $\geq$ 0.99	Calibration Standards	A
002	A3	Sensitivity	70-130% Recovery for ICV & CCV	Calibration Standards	A
002	A3	Precision	RPD $\leq$ 30%	Lab Duplicate	S & A
002	A3	Accuracy/Bias	70-130% Recovery	Laboratory Control Sample-2nd Source	A
002	A3	Contamination	< MDL	Laboratory Blanks	A

QAPP Worksheet #12
(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table
(continued)

Matrix	Groundwater
Analytical Group	General Chemistry (Ammonia-N, Nitrate/Nitrite-N, Phosphorus, TOC, HPC)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
001	A4,A5,A6,A7,A8	Accuracy/Bias	90-110%	Calibration Check	A
001	A4,A5,A6,A7,A8	Accuracy/Bias	90-100% 80-120%-TOC	Laboratory Control Sample-2nd Source	A
001	A4,A5,A6,A7,A8	Precision	RPD $\leq$ 20%	Laboratory Duplicates	A
001	A4,A5,A6,A7,A8	Contamination	<QL	Laboratory Blanks	A
001	A4,A5,A6,A7,A8	Contamination	<QL	Field QA/QC Blanks	S

QAPP Worksheet #13
(UFP-QAPP Manual Section 2.7)

Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
Historical data from the remedial investigation, pre-design studies, performance monitoring of the biosparge pilot system	CRA, Inc.	**H2M Lab**-Organic & Inorganic Data, October 2006 through May 2011 **Mitkem/Spectrum Labs**-Organic & Inorganic Data, April 2009 through May 2011	To assess existing contamination and the effectiveness of the biosparge pilot system.	Data that were rejected during validation was not/will not be used for decision making.
	CRA, Inc. "Off-Site Groundwater Predesign Information Report" November 2002. "100% Final Design Report, Off-Site Groundwater Biosparge Phase I Treatment System" May 2005	The data listed above is limited to October 2006 through 2011. Historical data is available that dates back to the 1980s.	Historical data was used to guide the investigations performed to define the horizontal and vertical extent of the VCM subplume emanating from the site.	Data that were rejected during validation was not/will not be used for decision making.

QAPP Worksheet #14
(UFP-QAPP Manual Section 2.8.1)

Summary of Project Tasks

Sampling Tasks: Performance Monitoring includes the collection and analysis of groundwater and soil gas samples. Sampling procedures and protocols are provided in the 100% Final Design Report. The groundwater and soil gas samples collected will be analyzed by Spectrum Analytical, Inc. of Warwick, RI with the exception of heterotrophic plate count (HPC) which will be performed by the CRA Laboratory in Niagara Falls, NY.
Analysis Tasks: Spectrum Laboratories of Warwick, RI will process, prepare, and analyze the groundwater and soil gas samples for the list of analytes in Worksheet #23 with the exception of heterotrophic plate count (HPC) which will be performed by the CRA Laboratory in Niagara Falls, NY.
Quality Control Tasks: The following QA/QC samples will be collected and analyzed for groundwater samples: trip blanks for VOCs, field duplicates (VOCs), matrix spikes (VOCs), and matrix spike duplicates (VOCs) or laboratory duplicates. The following QA/QC samples will be collected and analyzed for soil gas samples: trip blanks for VOCs. All the following QC checks will be performed as applicable to the specific method: tuning, initial calibration, continuing calibration checks, laboratory control samples (LCS), surrogates, method blanks, instrument blanks, and all other applicable QC as defined in the analytical methods.
Secondary Data: See WS 13
Data Management Tasks: Analytical data will be maintained in a database after validation.
Documentation and Records: 1. All samples collected will have location documented, records of each sample collected in notebooks, and all field measurements documented in notebooks. CoCs, airbills, and sample logs will be prepared and retained for each sample. (see Worksheet #11) 2. A controlled copy of the finalized QAPP will be retained by CRA. 3. Document retention is to be until 10 years after the USEPA provides notice of termination of Administrative Order, Index No. II-CERCLA-02-2001-2018.
Assessment/Audit Tasks: For the purpose of external evaluation, performance evaluation check samples from the USEPA will be analyzed by the analytical laboratory at the request of the USEPA. Internally, the evaluation of data from these samples is done on a continuing basis over the duration of a given project. The CRA QA/QC Officer may carry out performance and/or systems audits to insure that data of known and defensible quality consistently are produced during a program. Systems audits are qualitative evaluations of all components of field and laboratory quality control measurement systems. They determine if the measurement systems are being used appropriately. The audits may be carried out before all systems are operational, during the program, or after the completion of the program. Such audits typically involve a comparison of the activities given in the QA/QC plan described herein, with activities actually scheduled or performed. A special type of systems audit is the data management audit. This audit addresses only data collection and management activities. The performance audit is a quantitative evaluation of the measurement systems used for the analysis of actual environmental samples. It requires testing the measurement systems with samples of known composition or behavior to evaluate precision and accuracy. The CRA QA/QC Officer will conduct a performance audit if the project laboratory has not successfully analyzed a performance evaluation sample for the constituents of concern within 6 months of the project start date.

QAPP Worksheet #14
(UFP-QAPP Manual Section 2.8.1)

Summary of Project Tasks

Data Review Tasks:
Spectrum of Warwick will verifiy that all data packages are complete for samples received. All data package deliverable requirements will be met. Validation of the analytical data will be performed by CRA. Data validation will be performed utilizing the following documents for guidance:
1. "Organic Data Review for Low Concentration Water" CLP/SOW, OLC03.2 (SOP #HW-13, Revision 3), September 2006; and
2. "Validation of Metals for the Contract Laboratory Program (CLP) based on SOW ILMO5.3 (SOP Revision 13)" (SOP #HW-2, Revision 13), September 2006.

Data obtained using methods not covered in these documents will be validated using the general princlples used in these documents and the analytical requirements specified in the methods.
Validated data and all related field logs/notes/records will be reviewed to assess total measurement error and determine overall usability of the data for project purposes. Data limitations will be determined and data will be compared to Project Quality Objectives and required Action Limits. Corrective action is initiated as necessary. Final data will be placed in database, with any necessary qualifiers, and tables, charts, and graphs are generated.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOC (A1)

Concentration Level: Low-Level

Analyte	CAS Number	Project Action Limit (ug/L) NYSDEC Class GA Standards	Project Quantitation Limit (ug/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (ug/L)	Method CRQLs (ug/L)	MDLs (ug/L)	QLs (ug/L)
1,1,1-Trichloroethane	71-55-6	5	5	0.50	5.0	0.50	5.0
1,1,2,2-Tetrachloroethane	79-34-5	5	5	0.42	5.0	0.42	5.0
1,1,2-Trichloroethane	79-00-5	1	1	0.38	5.0	0.38	5.0
1,1-Dichloroethane	75-35-3	5	5	0.25	5.0	0.25	5.0
1,1-Dichloroethene	75-35-4	5	5	0.39	5.0	0.39	5.0
1,2-Dichloroethane	107-06-2	0.6	0.6	0.41	5.0	0.41	5.0
1,2-Dichloropropane	78-87-5	1	1	0.61	5.0	0.61	5.0
2-Butanone	78-93-3	50	50	2.10	5.0	2.10	5.0
2-Hexanone	591-78-6	10	10	1.70	5.0	1.70	5.0
4-Methyl-2-pentanone	108-10-1	10	10	0.82	5.0	0.82	5.0
Acetone	67-64-1	50	50	2.20	5.0	2.20	5.0
Benzene	71-43-2	0.7	0.7	0.33	5.0	0.33	5.0
Bromodichloromethane	75-27-4	10	10	0.26	5.0	0.26	5.0
Bromoform	75-25-2	10	10	0.77	5.0	0.77	5.0
Bromomethane	74-83-9	5	5	0.80	5.0	0.80	5.0
Carbon disulfide	75-15-0	60	60	0.34	5.0	0.34	5.0
Carbon tetrachloride	56-23-5	5	5	0.54	5.0	0.54	5.0
Chlorobenzene	108-90-7	5	5	0.26	5.0	0.26	5.0
Chloroethane	75-00-3	5	5	0.48	5.0	0.48	5.0

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: VOC (A1)

Concentration Level: Low-Level

Analyte	CAS Number	Project Action Limit (ug/L) NYSDEC Class GA Standards	Project Quantitation Limit (ug/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (ug/L)	Method CRQLs (ug/L)	MDLs (ug/L)	QLs (ug/L)
Chloroform	67-66-3	7	7	0.33	5.0	0.33	5.0
Chloromethane	74-87-3	5	5	0.26	5.0	0.26	5.0
1,2-Dichloroethene (Total)	156-59-2	5	5	0.65	5.0	0.65	5.0
Cis-1,2-dichloroethene	156-59-2	5	5	0.48	5.0	0.48	5.0
Trans-1,2-dichloroethene	156-60-5	5	5	0.65	5.0	0.65	5.0
Cis-1,3-Dichloropropene	10061-01-5	5	5	0.45	5.0	0.45	5.0
Dibromochloromethane	124-48-1	10	10	0.57	5.0	0.57	5.0
Ethyl benzene	100-41-4	5	5	0.35	5.0	0.35	5.0
Methylene chloride	75-09-2	5	5	0.41	5.0	0.41	5.0
Styrene	100-42-5	5	5	0.50	5.0	0.50	5.0
Tetrachloroethene	127-18-4	5	5	0.65	5.0	0.65	5.0
Toluene	108-88-3	5	5	0.32	5.0	0.32	5.0
Xylenes (total)	1330-20-7	5	5	0.77	5.0	0.77	5.0
m/p-Xylene	108-38-3 106-42-3	5	5	0.77	5.0	0.77	5.0
o-Xylene	95-47-6	5	5	0.36	5.0	0.36	5.0
Trans-1,3-Dichloropropene	10061-02-6	5	5	0.48	5.0	0.48	5.0
Trichloroethene	79-01-6	5	5	0.36	5.0	0.36	5.0
Vinyl chloride	75-01-4	2	2	0.50	5.0	0.50	5.0

- Quantitation limits are provided for guidance purposes only as they may not always be technically achievable due to such factors as elevated analyte concentrations and matrix interferences, which would require sample dilution.
- Compounds highlighted in yellow shall be reported at the MDL.
- The laboratory will make every effort possible to achieve target Quantitation limits.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: SOIL GAS

Analytical Group: VOC (A2)

Concentration Level: LOW

Analyte	CAS Number	Project Action Limit (ug/m3) DAR-1 AGC/SGC Tables	Project Quantitation Limit (ug/m3)	Analytical Method		Achievable Laboratory Limits	
				MDLs (ug/m3)	Method CRQLs (ug/m3)	MDLs (ug/m3)	QLs (ug/m3)
1,1,1,2-Tetrachloroethane	630-20-6	NA	NA	1.56	3.44	1.56	3.44
1,1,1-Trichloroethane	71-55-6	NA	NA	1.07	2.73	1.07	2.73
1,1,2,2-Tetrachloroethane	79-34-5	NA	NA	1.87	3.43	1.87	3.43
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	960000	NA	2.83	3.83	2.83	3.83
1,1,2-Trichloroethane	79-00-5	NA	NA	1.43	2.73	1.43	2.73
1,1-Dichloroethane	75-35-3	NA	NA	0.81	2.02	0.81	2.02
1,1-Dichloroethene	75-35-4	NA	NA	1.48	1.98	1.48	1.98
1,2,4-Trichlorobenzene	120-82-1	3700	NA	1.37	3.71	1.37	3.71
1,2,4-Trimethylbenzene	95-63-6	NA	NA	0.82	2.46	0.82	2.46
1,2-Dibromoethane	106-93-4	NA	NA	2.34	3.84	2.34	3.84
1,2-Dichlorobenzene	95-50-1	30000	NA	1.39	3.01	1.39	3.01
1,2-Dichloroethane	107-06-2	NA	NA	1.03	2.02	1.03	2.02
1,2-Dichloropropane	78-87-5	NA	NA	0.91	2.31	0.91	2.31
1,2-Dichlorotetrafluoroethane (CFC114)	76-14-2	NA	NA	2.55	3.49	2.55	3.49
1,3,5-Trimethylbenzene	108-67-8	NA	NA	1.44	2.46	1.44	2.46
1,3-Butadiene	106-99-0	NA	NA	0.83	1.10	0.83	1.10
1,3-Dichlorobenzene	541-73-1	NA	NA	1.64	3.01	1.64	3.01
1,4-Dichlorobenzene	106-46-7	NA	NA	1.29	3.01	1.29	3.01
1,4-Dioxane	123-91-1	3000	NA	0.95	1.80	0.95	1.80
2-Butanone	78-93-3	13000	NA	1.06	1.47	1.06	1.47
2-Hexanone	591-78-6	NA	NA	0.63	2.05	0.63	2.05
2-Phenylbutane (sec-Butylbenzene)	135-98-8	NA	NA	1.33	2.74	1.33	2.74
4-Ethyl toluene	622-96-8	NA	NA	1.17	2.46	1.17	2.46

Reference Limits and Evaluation Table

Matrix: SOIL GAS

Analytical Group: VOC (A2)

Concentration Level: LOW

Analyte	CAS Number	Project Action Limit (ug/m3) DAR-1 AGC/SGC Tables	Project Quantitation Limit (ug/m3)	Analytical Method		Achievable Laboratory Limits	
				MDLs (ug/m3)	Method CRQLs (ug/m3)	MDLs (ug/m3)	QLs (ug/m3)
4-Methyl-2-pentanone	108-10-1	31000	NA	1.02	2.05	1.02	2.05
Acetone	67-64-1	180000	NA	1.06	1.19	1.06	1.19
Acrylonitrile	107-13-1	NA	NA	0.83	1.08	0.83	1.08
Benzene	71-43-2	1300	NA	0.51	1.60	0.51	1.60
Benzyl chloride	100-44-7	240	NA	0.92	2.58	0.92	2.58
Bromodichloromethane	75-27-4	NA	NA	1.41	3.35	1.41	3.35
Bromoform	75-25-2	NA	NA	2.29	5.17	2.29	5.17
Bromomethane	74-83-9	NA	NA	1.16	1.94	1.16	1.94
Carbon disulfide	75-15-0	6200	NA	1.16	1.56	1.16	1.56
Carbon tetrachloride	56-23-5	1900	NA	1.31	3.15	1.31	3.15
Chlorobenzene	108-90-7	NA	NA	1.34	2.30	1.34	2.30
Chloroethane	75-00-3	NA	NA	1.18	1.32	1.18	1.32
Chloroform	67-66-3	150	NA	1.38	2.43	1.38	2.43
Chloromethane	74-87-3	22000	NA	0.77	1.03	0.77	1.03
Cis-1,2-Dichloroethene	156-59-2	NA	NA	0.65	1.98	0.65	1.98
Cis-1,3-Dichloropropene	10061-01-5	NA	NA	0.77	2.27	0.77	2.27
Cyclohexane	110-82-7	NA	NA	0.60	1.72	0.60	1.72
Cymene (p-Isopropyltoluene)	99-87-6	NA	NA	1.28	2.68	1.28	2.68
Dibromochloromethane	124-48-1	NA	NA	1.57	4.26	1.57	4.26
Dichlorodifluoromethane (CFC-12)	75-71-8	NA	NA	1.63	2.47	1.63	2.47
Ethanol	64-17-5	NA	NA	0.76	0.94	0.76	0.94
Ethyl acetate	141-78-6	NA	NA	0.99	1.80	0.99	1.80
Ethyl benzene	100-41-4	54000	NA	0.85	2.17	0.85	2.17
Hexachlorobutadiene	87-68-3	NA	NA	2.50	5.33	2.50	5.33
Hexane	110-54-3	NA	NA	0.77	1.76	0.77	1.76
Isopropyl Alcohol	67-63-0	98000	NA	0.56	1.23	0.56	1.23
Isopropylbenzene	98-82-8	NA	NA	1.24	2.46	1.24	2.46
m&p-Xylene	108-38-3 106-42-3	4300	NA	2.14	2.17	2.14	2.17

Reference Limits and Evaluation Table

Matrix: SOIL GAS

Analytical Group: VOC (A2)

Concentration Level: LOW

Analyte	CAS Number	Project Action Limit (ug/m3) DAR-1 AGC/SGC Tables	Project Quantitation Limit (ug/m3)	Analytical Method		Achievable Laboratory Limits	
				MDLs (ug/m3)	Method CRQLs (ug/m3)	MDLs (ug/m3)	QLs (ug/m3)
Methyl tert-Butyl Ether	1634-04-4	NA	NA	0.61	1.80	0.61	1.80
Methylene chloride	75-09-2	14000	NA	1.54	1.74	1.54	1.74
Naphthalene	91-20-3	7900	NA	0.91	2.62	0.91	2.62
n-Butylbenzene	104-51-8	NA	NA	1.34	2.74	1.34	2.74
n-Heptane	142-82-5	210000	NA	0.75	2.05	0.75	2.05
o-Xylene	95-47-6	4300	NA	1.32	2.17	1.32	2.17
Propylene (Propene)	115-07-1	NA	NA	0.37	0.86	0.37	0.86
Styrene	100-42-5	17000	NA	1.05	2.13	1.05	2.13
Tetrachloroethene	127-18-4	1000	NA	1.36	3.39	1.36	3.39
Tetrahydrofuran	109-99-9	30000	NA	0.65	1.47	0.65	1.47
Toluene	108-88-3	37000	NA	0.71	1.88	0.71	1.88
Trans-1,2-Dichloroethene	156-60-5	NA	NA	0.84	1.98	0.84	1.98
Trans-1,3-Dichloropropene	10061-02-6	NA	NA	0.68	2.27	0.68	2.27
Trichloroethene	79-01-6	14000	NA	0.96	2.69	0.96	2.69
Trichlorofluoromethane (CFC-11)	75-69-4	9000	NA	2.51	2.81	2.51	2.81
Vinyl chloride	75-01-4	180000	NA	1.01	1.28	1.01	1.28

- Quantitation limits are provided for guidance purposes only as they may not always be technically achievable due to such factors as elevated analyte concentrations and matrix interferences, which would require sample dilution.
- The laboratory will make every effort possible to achieve target Quantitation limits.

Reference Limits and Evaluation Table

Matrix: Soil Gas

Analytical Group: Methane (A3)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (ppmv)	Project Quantitation Limit (ppmv)	Analytical Method		Achievable Laboratory Limits	
				MDLs (ppmv)	Method QLs (ppmv)	MDLs (ppmv)	QLs (ppmv)
Methane	74-82-8	NA	NA	9.16	10.0	9.16	10.0

- Quantitation limits are provided for guidance purposes only as they may not always be technically achievable due to such factors as elevated analyte concentrations and matrix interferences, which would require sample dilution.
- The laboratory will make every effort possible to achieve target Quantitation limits.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: General Chemistry (A4, A5, A6, A7)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/L)	Project Quantitation Limit (mg/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (mg/L)	Method QLs (mg/L)	MDLs (mg/L)	QLs (mg/L)
Ammonia-N	7664-41-7	NA	NA	0.151	0.200	0.151	0.200
Nitrate-N	14797-55-8	NA	NA	0.00628	0.0100	0.00628	0.0100
Nitrite-N	14797-65-0	NA	NA	0.00562	0.0100	0.00562	0.0100
Phosphorus	7723-14-0	NA	NA	0.030	0.030	0.030	0.030
Total Organic Carbon	NA	NA	NA	2.0	10.0	2.0	10.0

- Quantitation limits are provided for guidance purposes only as they may not always be technically achievable due to such factors as elevated analyte concentrations and matrix interferences, which would require sample dilution.
- The laboratory will make every effort possible to achieve target Quantitation limits.

QAPP Worksheet #16
(UFP-QAPP Manual Section 2.8.2)

Project Schedule Timeline Table

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
See Figure 4 below					

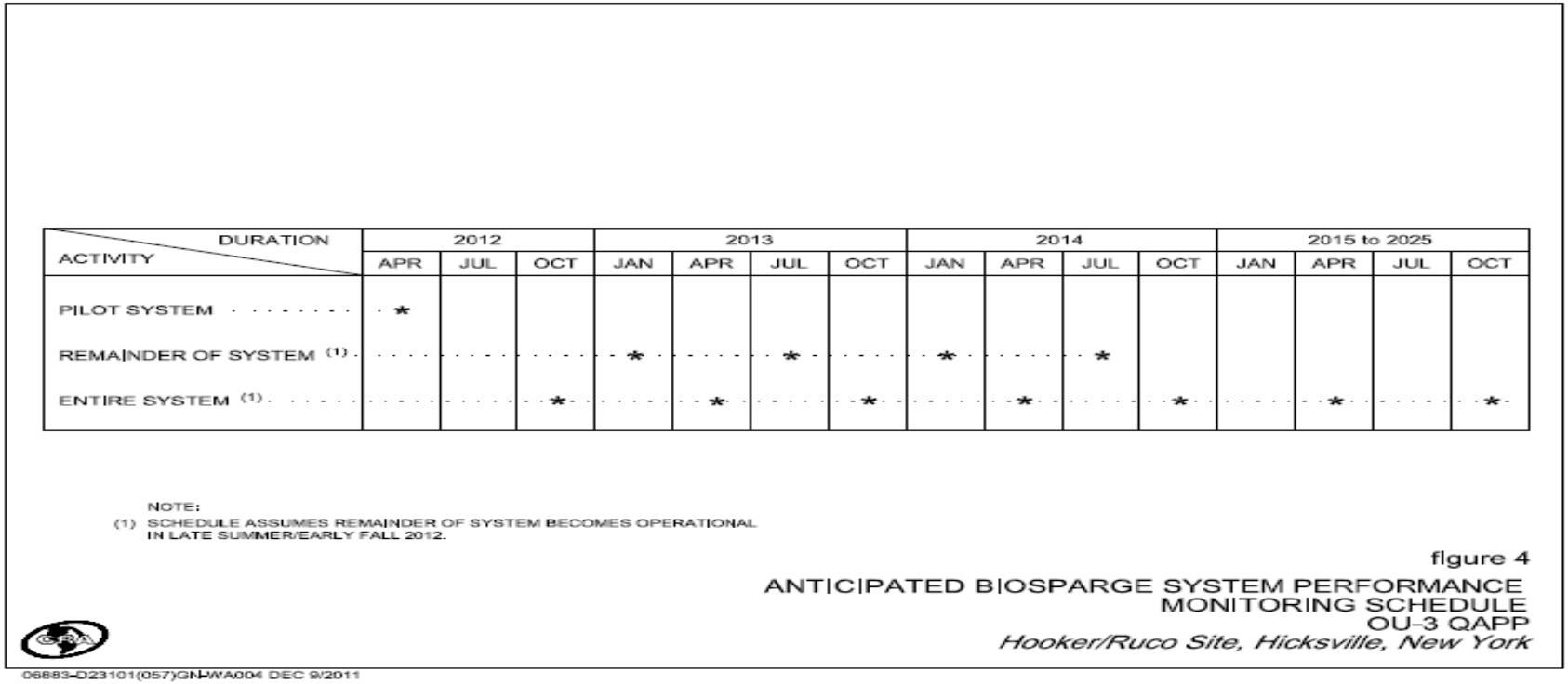


figure 4

ANTICIPATED BIOSPARGE SYSTEM PERFORMANCE MONITORING SCHEDULE
OU-3 QAPP
Hooker/Ruco Site, Hicksville, New York

06883-D23101(057)GN-WA004 DEC 9/2011

QAPP Worksheet #17
(UFP-QAPP Section 3.1.1)

☑ Worksheet Not Applicable (State Reason)

This information can be found in the 100% Final Design Report dated May 2005.

Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):
Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [May refer Worksheet #18 for details]

QAPP Worksheet #18
(UFP-QAPP Manual Section 3.1.1)

Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number	Matrix	Depth* ( )	Analytical Group	Concentration Level	Number of Samples (identify field duplicates	Sampling SOP Reference[1]	Rationale for Sampling Location
QUARTERLY LIST (2 Years)							
MW-70D1	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-70D2	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-72D1	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-72D2	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-73D1	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-73D2	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-75D1	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-75D2	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-76S	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-76I	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-76D1	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-76D2	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-77D1	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-77D2	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-85S	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-85I	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-85D1	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-85D2	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-86D1	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-86D2	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-89D1	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-89D2	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-87D1 (One Year)	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-87D2	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-90D1	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report
MW-90D2	Groundwater	NA	VOCs/Gen Chem	Low	8	001	See 100% Final Design Report

Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number	Matrix	Depth* ( )	Analytical Group	Concentration Level	Number of Samples (identify field duplicates	Sampling SOP Reference[1]	Rationale for Sampling Location
SEMI-ANNUAL LIST							
Quarterly list plus:							
MW-61I	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-61D1	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-61D2	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-81D1	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-81D2	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-82D1	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-82D2	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-83D1	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-83D2	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-84D1	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-84D2	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-87D1	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-87D2	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-88D1	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
MW-88D2	Groundwater	NA	VOCs/Gen Chem	Low	4	001	See 100% Final Design Report
VZ-1S	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-1D	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-2S	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-2D	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-4S	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-4D	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-5S	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-5D	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-6S	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-6D	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-12S	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-12D	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-17S	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report
VZ-17D	Soil Gas	NA	VOCs/Methane	Low	4	002	See 100% Final Design Report

Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number	Matrix	Depth* ( )	Analytical Group	Concentration Level	Number of Samples (identify field duplicates	Sampling SOP Reference[1]	Rationale for Sampling Location
ANNUAL FOR 2 YEARS			**Analysis to be performed by CRA In-House Lab**				
MW-70D1	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-70D2	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-72D1	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-72D2	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-73D1	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-73D2	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-75D1	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-75D2	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-76S	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-76I	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-76D1	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-76D2	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-77D1	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-77D2	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-85S	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-85I	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-85D1	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-85D2	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-86D1	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-86D2	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-89D1	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-89D2	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-90D1	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report
MW-90D2	Groundwater	NA	Heterotrophic Plate Count	Low	2	001	See 100% Final Design Report

Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number	Matrix	Depth* ( )	Analytical Group	Concentration Level	Number of Samples (identify field duplicates	Sampling SOP Reference[1]	Rationale for Sampling Location
PERIODICALLY SAMPLED							
MW-58D1	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-58D2	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-59D1	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-59D2	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-62I	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-62D	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-63I	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-63D1	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-63D2	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-64I	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-64D	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-67S	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-67D	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-68S	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-68D	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-92D1	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-92D2	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-93D1	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report
MW-93D2	Groundwater	NA	VOC/Gen Chem	Low	TBD	001	See 100% Final Design Report

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

*** See Attachment H, Well Installation Details, for proposed sample depth and well screen interval.**

QAPP Worksheet #19
(UFP-QAPP Manual Section 3.1.1)

Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference [1]	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Groundwater	VOCs plus TICs	Low	A1	4 x 40 mL	40 mL glass Teflon septum vials	HCl to pH <2 Cool 2 to 6 C	14 days from collection to analysis; 7 days from collection to analysis for unpreserved samples
Soil Gas	VOCs	Low	A2	6L SUMMA Canister	6L SUMMA Canister	NA	30 days from collection to analysis
Soil Gas	Methane	Low	A3	1L SUMMA Canister	1L SUMMA Canister	NA	30 days from collection to analysis
Groundwater	Ammonia-N	Low	A4	1 x 250 mL	HD Polyethylene Bottle	H_2SO_4 to pH <2 Cool 2 to 6 C	28 days from collection to analysis
Groundwater	Nitrate/Nitrite-N	Low	A5	1 x 250 mL	HD Polyethylene Bottle	H_2SO_4 to pH <2 Cool 2 to 6 C	28 days from collection to analysis
Groundwater	Phosphorus	Low	A6	1 x 150 mL	HD Polyethylene Bottle	H_2SO_4 to pH <2 Cool 2 to 6 C	28 days from collection to analysis
Groundwater	TOC	Low	A7	1 x 250 mL	Amber glass bottles	HCl or H_2SO_4 to pH <2 Cool 2 to 6 C	28 days from collection to analysis
Groundwater	HPC	Low	A8	1 x 100 mL	Amber glass bottles	Cool 2 to 6 C	24 hours from collection to analysis

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

- The number of sample containers will be kept to a minimum to reduce sample preservation requirements.
- The pH of the VOC samples will be adjusted to <2 by carefully adding 1:1 HCl drop-wise to the two 40 mL sample vials. The number of drops of HCl required will be determined on a third 40 mL vial of sample. If acidification causes effervescence, the sample will be submitted without acid preservation

QAPP Worksheet #20
(UFP-QAPP Manual Section 3.1.1)

Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Field Duplicate Pairs	No. of MS/MSD & DUP	No. of Field Blanks	No. of Equip. Blanks	No. of PT Samples	Total No. of Samples to Lab
Groundwater	VOCs with TICs	Low	A1	59	One pair per 20 investigative samples	One MS/MSD pair per 20 investigative samples	One trip blank in every cooler	NA	NA	TBD-Multiple sampling events
Soil Gas	VOCs	Low	A2	14	NA	NA	NA	NA	NA	TBD-Multiple sampling events
Soil Gas	Methane	Low	A3	14	NA	NA	NA	NA	NA	TBD-Multiple sampling events
Groundwater	Ammonia-N	Low	A4	59	NA	NA	NA	NA	NA	TBD-Multiple sampling events
Groundwater	Nitrate/Nitrite-N	Low	A5	59	NA	NA	NA	NA	NA	TBD-Multiple sampling events
Groundwater	Phosphorus	Low	A6	59	NA	NA	NA	NA	NA	TBD-Multiple sampling events
Groundwater	TOC	Low	A7	59	NA	NA	NA	NA	NA	TBD-Multiple sampling events
Groundwater	HPC	Low	A8	59	NA	NA	NA	NA	NA	TBD-Multiple sampling events

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

QAPP Worksheet #21
(UFP-QAPP Manual Section 3.1.2)

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Check if yes)	Comments
001	SOP# 001 - Passive Groundwater Sampling, Revision 0, October 10, 2011	Langan	Various	☐	
002	Soil Vapor Sampling Using SUMMA Canisters per Method TO-15	C.A.Rich	Various	☐	

QAPP Worksheet #22
(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Field Meters (water level, pH/temperature, conductivity, DO, turbidity, and eH meters)	Per manufacturers instructions.	Check/replace batteries. Clean electrode with soap and rinse thoroughly with water. Immerse the lower 3rd of the electrode in diluted HCl (1:9) solution for 10 minutes to remove any film. Rinse with water Keep electrode properly filled with appropriate solution	Check/replace batteries.	Check/replace batteries.	Daily	Per manufacturers instructions.	Replace batteries. Clean electrode. Consult manufacturer's instruction manual.	Victoria Whelan	001

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

QAPP Worksheet #23
(UFP-QAPP Manual Section 3.2.1)

Analytical SOP References Table

Reference Number (1)	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
A1	Standard Operating Procedure for Determination of Volatile Organic Compounds by GC/MS Analysis by SW846 Method 8260C	Definitive	VOCs with TICs	GC/MS	Spectrum Analytical, Inc.	☒
A2	Standard Operating Procedure for Method TO-15 – Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters and Analyzed by GC/MS	Definitive	VOCs	GC/MS	Spectrum Analytical, Inc.	☒
A3	Standard Operating Procedure for Modified EPA 3C/RSKSOP-175 – Determination of Fixed Gases and Dissolved Gases in Gaseous and Aqueous Phase Matrixes by TCD	Definitive	Organic	GC	Spectrum Analytical, Inc.	☒
A4	Standard Operating Procedure for Ammonia Nitrogen as N (mg/L) SM4500-NH3-B,C, 20th Edition	Definitive	General Chemistry	Colorimeter	Spectrum Analytical, Inc.	☒
A5	Standard Operating Procedure for Nitrate/Nitrite-N by Flow Injection Analysis – Lachat QuikChem Method 10-107-04-1-B, Nitrate via EPA 353.2, Nitrite via EPA 353.2	Definitive	General Chemistry	Colorimeter	Spectrum Analytical, Inc.	☒
A6	Standard Operating Procedure for Total Phosphorus and Orthophosphate Analysis for Aqueous Samples by Standard Method 4500-P B (5) & E, Ascorbic Acid Method	Definitive	General Chemistry	Spectrophotometer	Spectrum Analytical, Inc.	☒
A7	Standard Operating Procedure for Total Organic Carbon by Methods SW-846 9060A and/or SM5310B	Definitive	General Chemistry	TOC Analyzer	Spectrum Analytical, Inc.	☒
A8	Heterotrophic Plate Count, Standard Methods 9215 (Data will not be validated)	Definitive	Microbiological	NA	CRA Lab	☒

Notes:
(1) See Attachment A for further laboratory SOP information

QAPP Worksheet #24
(UFP-QAPP Manual Section 3.2.2)

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/MS	See A1, A2	Initial calibration after initial instrument set-up, then when daily 12-hour calibration verification criteria is not met.	See SW-846 8260B See TO-15	Inspect system; correct problem and/or re-calibrate; re-analyze all affected investigative samples.	Shirley Ng	A1, A2
GC	See A3	Initial calibration after initial instrument set-up, then when daily calibration verification criteria is not met.	See 3C	Inspect system; correct problem and/or re-calibrate; re-analyze all affected investigative samples.	Shirley Ng	A3
Colorimeter	See A4 & A5	Initial calibration after initial instrument set-up, then when daily calibration verification criteria is not met.	See EPA 353.2 & 350.1	Inspect system; correct problem and/or re-calibrate; re-analyze all affected investigative samples.	Shirley Ng	A4, A5
Spectrophotometer	See A6	Initial calibration after initial instrument set-up, then when daily calibration verification criteria is not met.	See EPA 365.2	Inspect system; correct problem and/or re-calibrate; re-analyze all affected investigative samples.	Shirley Ng	A6
TOC Analyzer	See A7	Initial calibration after initial instrument set-up, then when daily calibration verification criteria is not met, but at least monthly..	See SM 5310B	Inspect system; correct problem and/or re-calibrate; re-analyze all affected investigative samples	Shirley Ng	A7

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

QAPP Worksheet #25
(UFP-QAPP Manual Section 3.2.3)

Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity[1]	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference [2]
GC/MS	Replace consumables, bake out column and trap, recondition column.	Perform BFB tune. Perform calibration check.	Check connections, replace consumables, bake out column and trap, recondition column, and perform leak test.	Every 12 hours	See A1, A2	Correct problem and/or re-calibrate; re-analyze all affected investigative samples.	Shirley Ng	A1, A2
GC	Replace consumables, bake out column and trap, recondition column.	Perform calibration check.	Check connections, replace consumables, bake out column and trap, recondition column, and perform leak test.	Every 12 hours	See A3	Correct problem and/or re-calibrate; re-analyze all affected investigative samples.	Shirley Ng	A3
Colorimeter	Replace pump tubing, replace consumables, clean instrument trap.	Perform calibration check.	Inspect pump tubing, check connections and instrument cleanliness.	Every 10 samples	See A4 & A5	Correct problem and/or re-calibrate; re-analyze all affected investigative samples.	Shirley Ng	A4 & A5
Spectrophotometer	Replace pump tubing, replace consumables, clean instrument trap.	Perform calibration check.	Inspect pump tubing, check connections and instrument cleanliness.	Every 10 samples	See A6	Correct problem and/or re-calibrate; re-analyze all affected investigative samples.	Shirley Ng	A6
TOC Analyzer	Replace pump tubing, replace consumables, replace all reagents with fresh.	Perform calibration check	Inspect pump tubing, check connections and instrument cleanliness.	Every 10 samples	See A7	Correct problem and/or re-calibrate; re-analyze all affected investigative samples.	Shirley Ng	A7

[1]Maintenance activity, inspection activity, and corrective action performed as needed based on results of testing activity.
[2]Specify the appropriate reference letter or number from Analytical SOP References table (Worksheet #23).
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QAPP Worksheet #26
(UFP-QAPP Manual Appendix A)

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Victoria Whelan/C. A. Rich
Sample Packaging (Personnel/Organization): Victoria Whelan/C. A. Rich
Coordination of Shipment (Personnel/Organization): Victoria Whelan/C. A. Rich
Type of Shipment/Carrier: Overnight Courier/FedEx
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): TBD by Shirley Ng/Spectrum Analytical, Inc./ Sophia Dore-CRA, Inc.
Sample Custody and Storage (Personnel/Organization): TBD by Shirley Ng/Spectrum Analytical, Inc./Sophia Dore-CRA, Inc.
Sample Preparation (Personnel/Organization): TBD by Shirley Ng/Spectrum Analytical, Inc. /Sophia Dore-CRA, Inc.
Sample Determinative Analysis (Personnel/Organization): TBD by Shirley Ng/Spectrum Analytical, Inc. /Sophia Dore-CRA, Inc.
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Up to 48 hours
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 40 days
Biological Sample Storage (No. of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: TBD by Shirley Ng/Spectrum Lab /Sophia Dore-CRA, Inc.
Number of Days from Analysis: 30 days after reports have been submitted to agency.

QAPP Worksheet #27
(UFP-QAPP Manual Section 3.3.3)

Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Field personnel are required to keep a written record of field activities in applicable field logbooks. The field logbooks provide the means of recording data collecting activities. These records will be written legibly in ink and will contain pertinent field data and observations. Entry errors or changes will be crossed out with a single line, dated, and initialed by the person making the correction. Field logbooks will be periodically reviewed by the Field QA/QC Officer. Each member of the field team will be assigned a field logbook. Each field logbook title page should include field team member's name, project name, activity start date, activity end date, and unique log number. Upon collection, samples are placed in the appropriate pre-cleaned containers. The samples will be assigned a unique sample number and will be affixed to a sample label. The information to be placed on the sample label will include the sample ID number, sample type, sampler's name, date collected, preservation technique, and analytical parameter and method to be performed. Information on the labels will be completed with a ballpoint pen or indelible marker. A chain of custody (CoC) record (see Attachment B) will be completed during sample collection and will accompany each shipment identifying the contents of the shipment. The field personnel collecting the samples will be responsible for the custody of the samples until the samples are relinquished to the laboratory. Sample transfer will require the individuals relinquishing and receiving the samples to sign, date and note the time of sample transfer on the CoC record. As few people as possible should handle the samples. Samples will be shipped or delivered in a timely fashion to the laboratory so that holding times and/or analysis times as prescribed by the methodology can be met. Samples will be transported in containers (coolers) packed with ice to maintain the proper temperature range for shipment (0-6 degrees Celsius). Samples will be packaged for shipment and shipped to the appropriate laboratory for analysis with a separate signed CoC record enclosed in each sample cooler. Water VOC samples will be placed in bubble wrap bags that contain three vials per bag. All samples will be placed in an upright position and limited to one layer of samples per each cooler. Additional bubble wrap or packing material will be added to fill the cooler. Shipping containers will be secured with strapping tape and custody tape for shipment to the lab. If samples are sent by a commercial carrier, a bill of lading will be used. A copy of the bill of lading will be retained as part of permanent documentation. Commercial carriers are not required to sign the CoC record as long as the custody record is sealed inside the sample cooler and the custody tape remains intact. Samples will be hand delivered or picked up by a laboratory courier within 48 hours of collection unless collected on a weekend or holiday. In these cases, the samples will be stored in a secure location until delivery to the lab. Additional ice will be added to the cooler as needed to maintain proper preservation temperature.
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): A full time sample custodian will be assigned the responsibility of sample control. It will be the responsibility of the sample custodian to receive all incoming samples. Once received, the custodian will document that the custody tape on the coolers is unbroken, that each sample is received in good condition (i.e., unbroken, cooled, etc.), that the associated paperwork, such as the CoC forms have been completed and will sign the CoC forms. In special cases, the custodian will document from appropriate sub-samples that CoC with proper preservation has been accomplished. The custodian will also document that sufficient sample volume has been received to complete the analytical program. The sample custodian will then place the samples into secure, limited access storage (refrigerated storage, if required). Consistent with the analyses requested on the CoC form, analyses by the laboratory analysts will begin in accordance with the appropriate methodologies. Samples will be removed from secure storage only after internal CoC sign-out procedures have been followed. Sample containers with volume remaining will be returned to secure and limited access storage. Upon completion of all laboratory analyses for each sample submittal and generation of the laboratory report, samples will be stored by the sample custodian. The length of time that samples are held will be at least thirty (30) days after reports have been submitted. Disposal of remaining samples will be completed in compliance with all Federal, State, and local requirements.

QAPP Worksheet #27
(UFP-QAPP Manual Section 3.3.3)

Sample Custody Requirements

Sample Identification Procedures: Each sample will be labeled with a unique sample number that will facilitate tracking and cross-referencing of sample information. Field blank and field duplicate samples also will be numbered with a unique sample number to prevent analytical bias of field QC samples. The sample numbering system to be used is described as follows:

Example: GW-092311-AA-YYY
Where:
GW: Designated sample type (GW=groundwater)
092311: Date of collection (mm/dd/yy)
AA: Sampler initials
YYY: Sequential number starting with 001 at the start of the program

Upon arrival at the lab, the sample custodian will assign a unique number to each incoming sample for use in the lab. The unique number will then be entered into the sample-receiving log. The laboratory date of receipt will also be noted.

Chain-of-custody Procedures: Custody is one of several factors necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Attachment B illustrates a typical chain-of-custody form. For samples to be shipped off Site for analyses, custody seals will be placed around each cooler. The cooler will then be sealed with packing tape. Sample container labels will include sample number, place of collection and date and time of collection. All samples will be shipped via courier or hand delivered to the laboratory from the Site within 48 hours of collection.

The chain-of-custody form, completed at the time of sampling, will include, but not be limited to, the sample number, date and time of sampling, designation of samples as grab or composite, parameters to be analyzed, preservatives used and the name of the sampler. The chain-of-custody form will be signed, timed, and dated by the sampler when transferring the samples. Custody transfers will be recorded for each individual sample. For example, if samples are split and sent to more than one laboratory, a chain-of-custody form will accompany each sample. The number of custodians in the chain of possession will be kept to a minimum. The fully executed chain-of-custody forms will be returned to CRA.

A chain-of-custody form will be prepared for each cooler of samples being shipped to the off-Site laboratory. The chain-of-custody form is a four-part carbonless copy form that is completed and distributed as follows:

- Sampling personnel, after including all required information on the form, will relinquish custody of the samples to Site personnel responsible for sample packaging and shipping and retain the bottom (pink) carbonless copy;
- Sample packaging and shipping personnel will properly package the samples for transport, relinquish custody to the courier service (by writing the courier's name in the "received by" line), and retain the third (goldenrod) carbonless copy;
- The original form (i.e., top copy) and remaining carbonless copy will be packaged in a waterproof enclosure within the cooler with the samples, and the cooler will be sealed for shipping by the courier service;
- The laboratory, upon receiving the shipping cooler, will accept custody of the samples and complete the laboratory-required information on the form and retain the second (yellow) carbonless copy for its records; and
- The fully executed top (white) copy will be returned as part of the data deliverables package.

Upon receipt of the cooler at the laboratory, the cooler and the seal will be inspected by the designated sample custodian. The laboratory will measure and record the cooler and sample temperatures on the chain-of-custody form. The condition of the cooler and the sample container custody seals will be noted on the chain-of-custody form by the sample custodian. If the cooler seals are intact, the sample containers will be accepted for analyses. The sample custodian will document the date and time of receipt of the cooler, and sign the chain-of-custody form. If damage or discrepancies are noticed, they will be recorded in the remarks column of the chain-of-custody form, dated and signed. Any damage or discrepancies will be reported to the laboratory supervisor who will inform the laboratory manager and QA/QC Officer, who will in turn notify CRA. Completed chain-of-custody forms describing the transport to and receipt at the laboratory are required to be returned by the off-Site laboratory to CRA with the hard copy of the analytical report in order to facilitate data validation.

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	VOCs Plus TICs
Concentration Level	Low
Sampling SOP	001
Analytical Method/ SOP Reference	A1
Sampler's Name	Victoria Whelan
Field Sampling Organization	C.A. Rich
Analytical Organization	Spectrum Analytical, Inc.
No. of Sample Locations	59

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	Every 12 hours or 1/20 samples, whichever is most frequent	Lab Limits	Re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	Lab Limits
Method Blank	Every 12 hours or 1/20 samples, whichever is most frequent	Target Compounds <QL or <5 x QL for common lab contaminants	Re-analyze affected samples. Flag data as needed.	Shirley Ng	Contamination	Target Compounds <QL or <5 x QL for common lab contaminants
Surrogate Spike	All samples, blanks, and QC samples	Laboratory Limits	Re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	Laboratory Limits
Internal Standards	All samples, blank, and QC samples	50-200% recovery	Re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	50-200% recovery

QC Samples Table

Matrix	Groundwater
Analytical Group	VOCs Plus TICs
Concentration Level	Low
Sampling SOP	001
Analytical Method/ SOP Reference	A1
Sampler's Name	Victoria Whelan
Field Sampling Organization	C.A. Rich
Analytical Organization	Spectrum Analytical, Inc.
No. of Sample Locations	59

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Trip Blank	1 per cooler	Target compounds <QL	None. Flag data as needed.	NA	Contamination	Target compounds <QL
Field Duplicates	1 per 20 samples	≤50 RPD	None. Flag data as needed.	NA	Precision	≤50 RPD

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Soil Gas
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	002
Analytical Method/ SOP Reference	A2
Sampler's Name	Victoria Whelan
Field Sampling Organization	C.A.Rich
Analytical Organization	Spectrum Analytical, Inc.
No. of Sample Locations	14

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	Every 24 hours or 1/20 samples, whichever is most frequent	Lab Limits	Re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	Lab Limits
Method Blank	Every 24 hours or 1/20 samples, whichever is most frequent	Target Compounds <QL or < 5x QL for common lab contaminants	Re-analyze affected samples. Flag data as needed.	Shirley Ng	Contamination	Target Compounds<QL or < 5x QL for common lab contaminants
Surrogate Spike	All samples, blanks, and QC samples	Lab Limits	Re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	Lab Limits

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Soil Gas
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	002
Analytical Method/ SOP Reference	A2
Sampler's Name	Victoria Whelan
Field Sampling Organization	C.A. Rich
Analytical Organization	Spectrum Analytical, Inc.
No. of Sample Locations	14

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standards	All samples, blank, and QC samples	50-200% recovery	Re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	50-200% recovery

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Soil Gas
Analytical Group	Methane
Concentration Level	Low
Sampling SOP	002
Analytical Method/ SOP Reference	A3
Sampler's Name	Victoria Whelan
Field Sampling Organization	C.A. Rich
Analytical Organization	Spectrum Analytical, Inc.
No. of Sample Locations	14

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	Every 24 hours or 1/20 samples, whichever is most frequent	Laboratory Limits	Re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	Laboratory Limits
Method Blank	1 per 20 samples	Target Compound <QL	Re-analyze affected samples. Flag data as needed.	Shirley Ng	Contamination	Target Compound <QL
Surrogate Spike	All samples, blanks, and QC samples	Laboratory Limits	Re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	Laboratory Limits

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	Ammonia-N
Concentration Level	Low
Sampling SOP	001
Analytical Method/ SOP Reference	A4
Sampler's Name	Victoria Whelan
Field Sampling Organization	C.A. Rich
Analytical Organization	Spectrum Analytical, Inc.
No. of Sample Locations	59

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	1 per 20 samples	90-110% recovery or Method Criteria	Re-extract and re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	90-110% recovery or Method Criteria
Method Blank	1 per 20 samples	<QL	Re-extract and re-analyze affected samples. Flag data as needed.	Shirley Ng	Contamination	<QL
Lab Duplicate	1 per 20 samples	$\leq$ 20 RPD	None. Flag data as needed.	Shirley Ng	Precision	$\leq$ 20 RPD

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	Nitrate/Nitrite-N
Concentration Level	Low
Sampling SOP	001
Analytical Method/ SOP Reference	A5
Sampler's Name	Victoria Whelan
Field Sampling Organization	C.A. Rich
Analytical Organization	Spectrum Analytical, Inc.
No. of Sample Locations	59

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	1 per 20 samples	90-110% Recovery or Method Criteria	Re-extract and re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	90-110% recovery or Method Criteria
Method Blank	1 per 20 samples	<QL	Re-extract and re-analyze affected samples. Flag data as needed.	Shirley Ng	Contamination	<QL
Lab Duplicate	1 per 20 samples	$\leq$ 20 RPD	None. Flag data as needed.	Shirley Ng	Precision	$\leq$ 20 RPD

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	Phosphorus
Concentration Level	Low
Sampling SOP	001
Analytical Method/ SOP Reference	A6
Sampler's Name	Victoria Whelan
Field Sampling Organization	C.A. Rich
Analytical Organization	Spectrum Analytical, Inc.
No. of Sample Locations	59

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	1 per 20 samples	90-110% Recovery or Method Criteria	Re-extract and re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	90-110% recovery or Method Criteria
Method Blank	1 per 20 samples	<QL	Re-extract and re-analyze affected samples. Flag data as needed.	Shirley Ng	Contamination	<QL
Lab Duplicate	1 per 20 samples	$\leq$ 20 RPD	None. Flag data as needed.	Shirley Ng	Precision	$\leq$ 20 RPD

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	TOC
Concentration Level	Low
Sampling SOP	001
Analytical Method/ SOP Reference	A7
Sampler's Name	Victoria Whelan
Field Sampling Organization	C.A. Rich
Analytical Organization	Spectrum Analytical, Inc.
No. of Sample Locations	59

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	1 per 20 samples	80-120% Recovery or Method Criteria	Re-prep/re-analyze affected samples. Flag data as needed.	Shirley Ng	Accuracy/Bias	80-120% Recovery or Method Criteria
Method Blank	1 per 20 samples	<QL	Re-prep/re-analyze affected samples. Flag data as needed.	Shirley Ng	Contamination	<QL
Lab Duplicate	1 per 20 samples	≤ 20 RPD	None. Flag data as needed.	Shirley Ng	Precision	≤ 20 RPD

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	HPC
Concentration Level	Low
Sampling SOP	001
Analytical Method/ SOP Reference	A8
Sampler's Name	Victoria Whelan
Field Sampling Organization	C.A. Rich
Analytical Organization	CRA, Inc
No. of Sample Locations	59

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	1 per 20 samples	80-120% Recovery or Method Criteria	Re-prep/re-analyze affected samples. Flag data as needed.	Sophia Dore	Accuracy/Bias	80-120% Recovery or Method Criteria
Method Blank	1 per 20 samples	<QL	Re-prep/re-analyze affected samples. Flag data as needed.	Sophia Dore	Contamination	<QL
Lab Duplicate	1 per 20 samples	$\leq$ 20 RPD	None. Flag data as needed.	Sophia Dore	Precision	$\leq$ 20 RPD

QAPP Worksheet #29
(UFP-QAPP Manual Section 3.5.1)

Project Documents and Records Table

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Logbooks Chain of Custody Records Air Bills Telephone Logs Deviation Reports & Corrective Action Forms E-mail & written Correspondence	Equipment calibration logs Field measured parameter logs Sample collection details	Sample Receipt, Custody, and Tracking Records Standard Traceability Logs Equipment Calibration Logs Sample Prep Logs Run Logs Equipment Maintenance, Testing and Inspection Logs Deviation Reports Corrective Action Forms Reported Sample Results Reported Results for Standards, QC Checks, and QC Samples Instrument Printouts (raw data) for Field Samples, QC Checks, and QC Samples Laboratory Case Narrative Lab Qualifier Definitions MDL Study Results Data Package Completeness Checklists Extraction/Clean-up Records Raw Data (stored on electronic media) Sample Disposal Records Telephone Logs E-mail & written Correspondence	Field Sampling Audit Checklists Fixed Laboratory Audit Checklists Data Validation Reports Corrective Action Forms Telephone Logs E-mail & written Correspondence Laboratory QA Plan	Photos, maps, drawings Reports associated with work

QAPP Worksheet #30
(UFP-QAPP Manual Section 3.5.2.3)

Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number
Groundwater	All	Low	59	A1, A4, A5, A6, A7	Within 30 calendar days after sample receipt	Shirley Ng, Spectrum Analytical, Inc. 175 Metro Center Blvd. Warwick, RI 02886-1755 401-732-3400	NA
Groundwater	All	Low	59	A8	Within 30 calendar days after sample receipt	Sophia Dore, CRA 2055 Niagara Falls Blvd Suite 3 Niagara Falls, NY 14604 716-297-2160	NA
Soil Gas	All	Low	14	A2, A3	Within 30 calendar days after sample receipt	Shirley Ng, Spectrum Analytical, Inc. 175 Metro Center Blvd. Warwick, RI 02886-1755 401-732-3400	NA

QAPP Worksheet #31
(UFP-QAPP Manual Section 4.1.1)

Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Offsite Laboratory Performance Audit	A performance audit will be conducted if the project laboratory has not successfully analyzed a performance evaluation sample for the constituents of concern within 6 months of the project start date.	External	CRA.	Denise Anderson QA/QC Officer, CRA	Denise Anderson QA/QC Officer, CRA	Denise Anderson QA/QC Officer, CRA	Denise Anderson QA/QC Officer, CRA

QAPP Worksheet #32
(UFP-QAPP Manual Section 4.1.2)

Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Org.)	Timeframe for Response
Offsite Laboratory Performance Audit	Written Audit Report	Sharyn Lawler, QA/QC Officer, Spectrum Analytical, Inc. Jim Kay, Project Manager, CRA Klaus Schmidtke, Project Coordinator, CRA Roger Smith, GSHI	15 days after audit	Letter	Denise Anderson, QA/QC Officer, CRA .	24 hours after notification

QAPP Worksheet #33
(UFP-QAPP Manual Section 4.2)

QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Data Validation Report	One report after each sampling event. HPC data will not be validated.	20 Business days after the validation of analytical data.	Kathy Willy, CRA	Jim Kay, Project Manager, CRA
Periodic Performance and Data Quality Reports	Varies	Varies	Denise Anderson, QA/QC Officer, CRA.	Jim Kay, Project Manager, CRA Roger Smith, GSHI

QAPP Worksheet #34
(UFP-QAPP Manual Section 5.2.1)

Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain of Custody (CoC) and Shipping Forms	CoC forms and shipping documents will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the CoC should be initialed by the reviewer, a copy of the CoC retained in the site file, and the original and remaining copies taped inside the cooler for shipment.	Internal	Victoria Whelan, C.A. Rich
Field Notes	Field notes will be reviewed internally and placed in the site file.	Internal	Victoria Whelan, C.A. Rich
Laboratory Data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal	Internal	Shirley Ng, Spectrum Analytical, Inc.
Laboratory Data	All received data packages will be verified externally according to the data validation procedures specified in Worksheet # 35 using the guidance specified in Worksheet #36.	External	Kathy Willy, CRA

QAPP Worksheet #35
(UFP-QAPP Manual Section 5.2.2)

Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	Data Deliverables	Ensure that all required documentation on sampling and analysis was provided	Kathy Willy, CRA
IIa	Analytes	Ensure that the required list of analytes was reported as specified in governing documents	Kathy Willy, CRA
IIa	Chain of Custody	Examine the traceability of the data from the time of sample collection until reporting of data. Examine CoC records against contract, method, and QAPP	Kathy Willy, CRA
IIa	Holding Times	Identify holding time criteria and determine if they were met. If holding times were not met, confirm that deviations were documented.	Kathy Willy, CRA
IIa	Sample Handling	Ensure that required sample handling, receipt, and storage procedures were followed and that any deviations were documented.	Denise Anderson, CRA
IIb	Sampling Methods and Procedures	Ensure that all sampling SOPs were followed and that any deviations were noted.	Denise Anderson, CRA
IIa	Field Transcription	Authenticate transcription accuracy for sampling data (i.e., from field notebook to reports.)	Denise Anderson, CRA
IIa	Analytical Methods and Procedures	Establish that required analytical methods were used and that any deviations were noted. Determine if the QC samples met performance criteria and ensure that any deviations were noted.	Kathy Willy, CRA
IIa	Data Qualifiers	Determine that the laboratory qualifiers were defined and applied as specified in methods, procedures, or contracts.	Kathy Willy, CRA
IIa	Laboratory Transcription	Authenticate accuracy of the transcription of analytical data (i.e. lab notebook to report form, or instrument to LIMS)	Kathy Willy, CRA
IIa	Verification of Calculations	Verify 5% of all calculations summarized on the laboratory's QA/QC summary sheets.	Kathy Willy, CRA
IIa	Standards	Determine if standards are traceable and meet contract, method, or procedural requirements.	Kathy Willy, CRA
IIb	Deviations	Determine the impacts of any deviations from sampling or analytical methods and SOPs.	Kathy Willy, CRA Denise Anderson, CRA
IIa	Documentation of Method QC Results	Determine if all method required QC samples were analyzed and met required acceptance limits.	Kathy Willy, CRA
IIb	Project Quantitation Limits	Determine if all sample results met the project quantitation limits specified in the QAPP	Kathy Willy, CRA
IIb	Field Duplicates	Compare results of field duplicates with criteria established in the QAPP	Kathy Willy, CRA

QAPP Worksheet #35
(UFP-QAPP Manual Section 5.2.2)

Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIb	Performance Criteria	Evaluate QC data against project-specific performance criteria in the QAPP	Kathy Willy, CRA
IIb	Data Qualifiers	Determine that the data qualifiers are appropriate and justified.	Kathy Willy, CRA
IIb	Data Validation Report	Summarize deviations from the methods, procedures, or contracts. Summarize the outcome of comparison of data to performance criteria in the QAPP. Include qualified data and explanation of all data qualifiers.	Kathy Willy, CRA

QAPP Worksheet #36
(UFP-QAPP Manual Section 5.2.2)

Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa	Groundwater	VOCs plus TICs	Low	"Organic Data Review for Low Concentration Water" CLP/SOW, OLC03.2 (SOP #HW-13, Revision 3), September 2006	Kathy Willy, CRA
IIa	Soil Gas	VOCs	Low	"Organic Data Review for Low Concentration Water" CLP/SOW, OLC03.2 (SOP #HW-13, Revision 3), September 2006	Kathy Willy, CRA
IIa	Soil Gas	Methane	Low	"Organic Data Review for Low Concentration Water" CLP/SOW, OLC03.2 (SOP #HW-13, Revision 3), September 2006	Kathy Willy, CRA
IIa	Groundwater	Ammonia-N	Low	"Validation of Metals for the Contract Laboratory Program (CLP) based on SOW ILMO5.3 (SOP Revision 13)" (SOP #HW-2, Revision 13), September 2006	Kathy Willy, CRA
IIa	Groundwater	Nitrate/Nitrite-N	Low	"Validation of Metals for the Contract Laboratory Program (CLP) based on SOW ILMO5.3 (SOP Revision 13)" (SOP #HW-2, Revision 13), September 2006	Kathy Willy, CRA
IIa	Groundwater	Phosphorus	Low	"Validation of Metals for the Contract Laboratory Program (CLP) based on SOW ILMO5.3 (SOP Revision 13)" (SOP #HW-2, Revision 13), September 2006	Kathy Willy, CRA
IIa	Groundwater	TOC	Low	"Validation of Metals for the Contract Laboratory Program (CLP) based on SOW ILMO5.3 (SOP Revision 13)" (SOP #HW-2, Revision 13), September 2006	Kathy Willy, CRA

QAPP Worksheet #37
(UFP-QAPP Manual Section 5.2.3)

Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:
Data will be validated using the guidelines referenced on Worksheet #36 and the laboratory SOPs. In order to assess data quality, these documents specify the consideration of statistical values such as percent recovery, relative percent difference, percent relative standard deviation, and percent difference and the use of equations such as those used to calculate response and calibration factors, quantitation limits, and analyte concentrations.

Describe the evaluative procedures used to assess overall measurement error associated with the project:
The sensitivities required for samples submitted for the chemical analyses will be at least the targeted quantitation limits listed in Worksheet #15 barring any chemical interferences or dilutions required due to elevated concentrations of the subject parameter(s). In these cases, the sample-specific quantitation limits will be reported in accordance with the method(s) protocols.
Accuracy/bias will be assessed by comparing a set of analytical results for spiked QC samples to the accepted or "true" values that would be expected. In general, MS, MSD, LCS, and surrogate recoveries and blank data will be used to assess accuracy. Accuracy/bias is typically calculated by percent recovery, but for some methods, %RSD and %D are used to determine the accuracy of calibration standards.

$$\text{Accuracy} = \frac{A-B}{C} \times 100$$

A = The analyte determined experimentally from the spike sample
B = The background level determined by a separate analysis of the unspiked sample
C = The amount of spike added

Precision will be assessed by comparing the analytical results between duplicate samples, which include field duplicates, laboratory duplicates, and paired MS and MSD samples. Precision is calculated by determining the RPD between the paired measurements.

Matrix Spike/Matrix Spike Duplicate

$$\text{Precision} = \frac{\{D2-D1\}}{\{D1+D2/2\} \times 100}$$

D1 = matrix spike recovery
D2 = matrix spike duplicate spike recovery

Sample Duplicates

$$\text{Precision} = \frac{\{D2-D1\}}{\{D1+D2/2\} \times 100}$$

D1 = original sample result
D2 = duplicate sample result

For results near the associated detection limits, precision will be assessed based on the following criteria:
Precision =|original result - duplicate result| <CRDL

Data with measurement error outside the established control limits will be qualified as necessary. Final validated results will be reported as usable without qualifications, usable with qualifications (e.g., "J," "UJ"), or unusable due to severe QA/QC deficiencies ("R").
An objective of this program is the collection of samples that are representative of the matrix from which they were collected. Achievement of this objective will rely on the use of sampling procedures, as described in the 100% Final Design Report that have been designed with the goal of obtaining representative samples.
The QA objective for comparability is the generation of Site characterization data that can be used to make valid comparisons with other analytical data that may be generated in the future at this site. This objective also involves the analysis of environmental samples in a manner that produces results comparable to the results that would be obtained by another laboratory using the same analytical procedure. This objective is achieved by the use of standard materials traceable to the National Institute of Standards and Technology, the use of accepted standard procedures for sample collection and sample analysis, and the analysis of quality control samples to validate the analytical results. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as documented in the QA/QC Plan, are expected to provide comparable data.

Completeness is defined as the ratio of the number of valid measurements to the total number of measurements necessary to achieve a specified level of confidence in decision making. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the database is sufficient. The QA objective for completeness is to collect and analyze all environmental samples in a manner such that valid data are obtained from a minimum of 90 percent of the samples.

When possible, the percent completeness for each set of samples will be calculated as follows:

$$\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100 \text{ percent}$$

Identify the personnel responsible for performing the usability assessment: Kathy Willy

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:
A data validation report will be written for each sampling event. The report will summarize any identified trends, correlations, or anomalies so that the data user can make informed decisions on the use of the data.
Completeness is defined as the ratio of the number of valid measurements to the total number of measurements necessary to achieve a specified level of confidence in decision making. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the database is sufficient. The QA objective for completeness is to collect and analyze all environmental samples in a manner such that

ATTACHMENT A

LIST OF STANDARD OPERATING PROCEDURES
SPECTRUM LABORATORY

LIST OF STANDARD OPERATING PROCEDURES
SPECTRUM LABORATORY

SOP No.	*Title*	*Rev. No.*
90.0012	Standard Operating Procedure for Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8260C	12
TO-15 11-15-10 Rev.9	Standard Operating Procedure for Method TO-15 – Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry GC/MS	9
11/20/09.doc	Standard Operating Procedures for Modified EPA 3C/RSKSOP-175 – Determination of Fixed Gases and Dissolved Gases in Gaseous and Aqueous Phase Matrixes by TCD	1
100.0025	Standard Operating Procedure for Total Organic Carbon by Methods SW-846 9060A and/or SM5310B	8
100-0013	Standard Operating Procedure for Total Phosphorus and Orthophosphate Analysis for Aqueous Samples by Standard Method 4500-P B (5) & E, Ascorbic Acid Method	9
1-21-11.doc	Standard Operating Procedure for Nitrate/Nitrite-N by Flow Injection Analysis – Lachat QuikChem Method 10-107-04-1-B, Nitrate Via EPA 353.2, Nitrite via EPA 353.2	3
07-18-06.doc	Standard Operating Procedure for Ammonia Nitrogen as N (mg/L) SM4500-NH3-B,C, 20th Edition	6
30.0003	Standard Operating Procedure for Sample Receipt, Storage, Tracking and Disposal	16

ATTACHMENT B

SAMPLE CRA CHAIN-OF-CUSTODY FORM

CHAIN OF CUSTODY RECORD

CONESTOGA-ROVERS & ASSOCIATES	SHIPPED TO (Laboratory Name):	REFERENCE NUMBER:

SAMPLER'S SIGNATURE: _____ PRINTED NAME: _____

SEQ. No.	DATE	TIME	SAMPLE No.	SAMPLE TYPE	No. of Containers	PARAMETERS	REMARKS
TOTAL NUMBER OF CONTAINERS						HEALTH/CHEMICAL HAZARDS	

RELINQUISHED BY:	DATE: / TIME:	RECEIVED BY:	DATE: / TIME:
① _____		① _____	
RELINQUISHED BY:	DATE: / TIME:	RECEIVED BY:	DATE: / TIME:
② _____		② _____	
RELINQUISHED BY:	DATE: / TIME:	RECEIVED BY:	DATE: / TIME:
③ _____		③ _____	

METHOD OF SHIPMENT: WAY BILL No.

White —Fully Executed Copy
Yellow —Receiving Laboratory Copy
Pink —Shipper Copy
Goldenrod —Sampler Copy

SAMPLE TEAM:

_____

_____

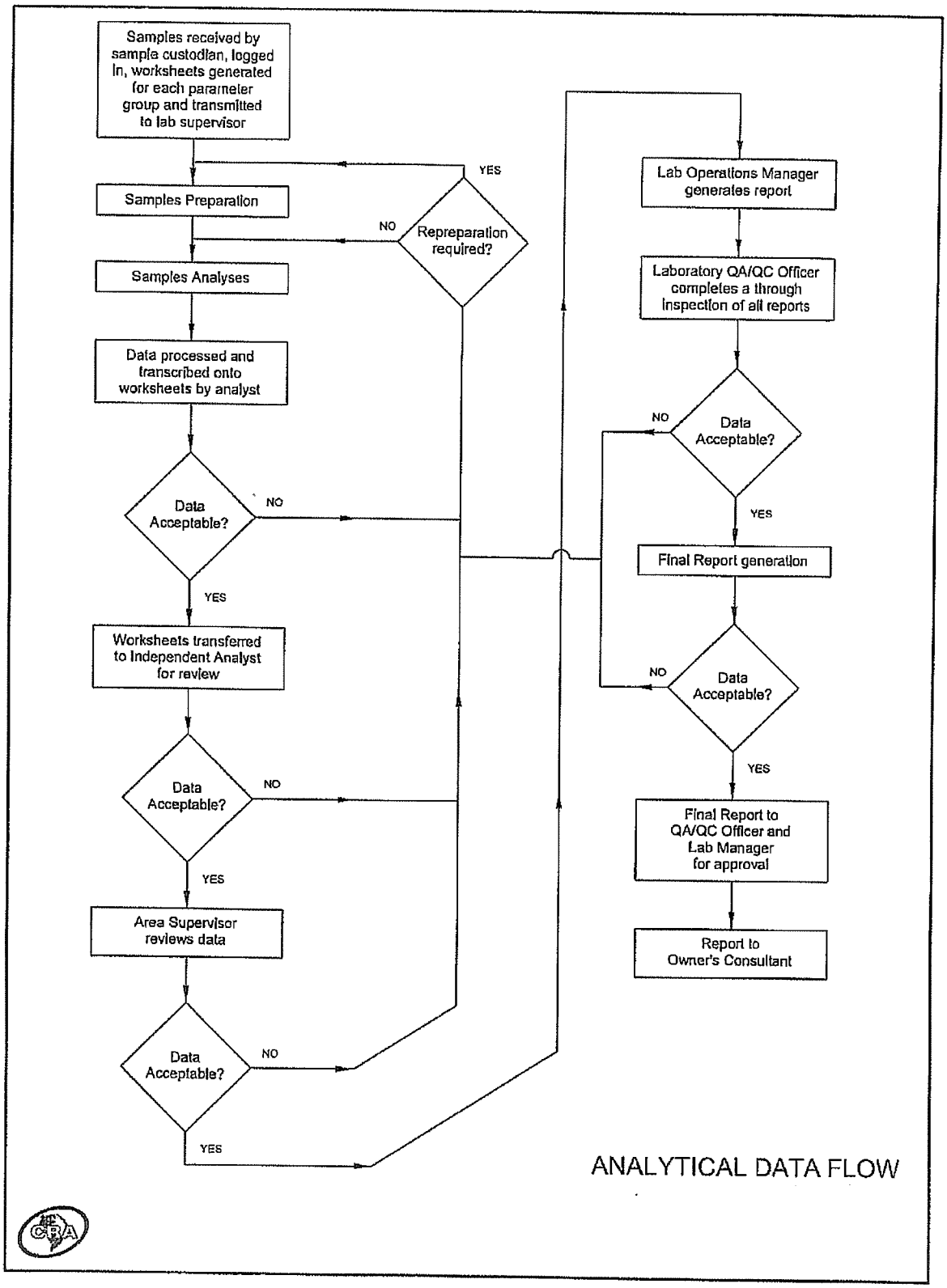
RECEIVED FOR LABORATORY BY:

_____

DATE: _____ TIME: _____

ATTACHMENT C

ANALYTICAL DATA FLOW


Samples received by sample custodian, logged in, worksheets generated for each parameter group and transmitted to lab supervisor
Samples Preparation
Repreparation required?
YES
NO
Samples Analyses
Data processed and transcribed onto worksheets by analyst
Data Acceptable?
NO
YES
Worksheets transferred to Independent Analyst for review
Data Acceptable?
NO
YES
Area Supervisor reviews data
Data Acceptable?
NO
YES
Lab Operations Manager generates report
Laboratory QA/QC Officer completes a through inspection of all reports
Data Acceptable?
NO
YES
Final Report generation
Data Acceptable?
NO
YES
Final Report to QA/QC Officer and Lab Manager for approval
Report to Owner's Consultant
ANALYTICAL DATA FLOW

ATTACHMENT D

LABORATORY DELIVERABLES REQUIREMENTS

LABORATORY DELIVERABLES REQUIREMENTS

The analytical report shall include, for each sample:

- Copies of fully executed chain-of-custody documents;
- Date collected;
- Date of sample receipt;
- Cooler temperature measurements;
- Sample pH measurements (where applicable);
- Date extracted or digested;
- Preparation logs;
- Date analyzed;
- Raw data including logbook pages and instrument printouts;
- Method for sample preparation;
- Analytical methodology;
- Method of sample cleanup;
- Method quantitation limits;
- Sample results for all samples;
- Sample dilution factor; and
- A case narrative including a discussion of all QC problems and corrective actions taken.

QC data that shall be submitted with each report shall include:

- Definition of surrogates;
- Recovery of surrogates;
- Method blanks;
- Instrument blanks;
- MS/MSD results;
- LCS data; and
- A comparison of all QC data to QC acceptance criteria.

The case narrative to each analytical report shall describe, in lay terms, any and all QA/QC problems encountered during analysis of the samples. For each sample for which QA/QC problems are encountered, the following specific information shall be reported in the case narrative:

- CRA sample number;
- Laboratory sample number;
- Date of sample collection;
- Date of sample receipt at the laboratory;
- Date of sample analysis;
- Sample matrix;
- Parameters analyzed;
- Data with outlying quality control;
- Specific analytical problems that occurred; and
- The corrective action that was taken or attempted to resolve the problems.

ATTACHMENT E

LIST OF ACRONYMS/ABBREVIATIONS

LIST OF ACRONYMS/ABBREVIATIONS

ASTM	American Society for Testing and Materials
B.A.Sc.	Bachelor of Applied Science
B.S.	Bachelor of Science
BFB	Bromofluorobenzene
CA	Corrective Actions
CCC	Calibration Check Compounds
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
COB	Close of Business
CoC	Chain of Custody
COC	Contaminant of Concern
CRA	Conestoga-Rovers & Associates
DO	Dissolved Oxygen
DQI	Data Quality Indicators
DUP	Duplicate
DVC	Data Validation Criteria
EE/CA	Engineering Evaluation Cost Analysis
EPA	Environmental Protection Agency
ft. amsl	Feet above mean sea level
GC	Gas Chromatograph
GC/MS	Gas Chromatograph/Mass Spectrometer
HAZWOPER	Hazardous Waste Operations and Emergency Response
HCl	Hydrochloric Acid
HNO_3	Nitric Acid
H_2SO_4	Sulfuric Acid
HSP	Health and Safety Plan
IDL	Instrument Detection Limit
LIMS	Laboratory Information Management System
LCS	Laboratory Control Sample
MDL	Method Detection Limit
mL	milliliter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
NaOH	Sodium Hydroxide
NTU	Nephelometric Turbidity Unit
NYSDEC	New York State Department of Environmental Conservation
OSHA	Occupational Safety and Health Administration
OL	Operating Limit
P.E.	Professional Engineer
PQO	Project Quality Objective
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
RD	Remedial Design
RL	Reporting Limit
RPD	Relative Percent Difference
RPM	Regional Project Manager
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SM	"Standard Methods for the Examination of Water and Waste Water", 19th Edition, 1995
SOP	Standard Operating Procedure
SPCC	System Performance Check Compounds

LIST OF ACRONYMS/ABBREVIATIONS

SW-846	"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", 3rd Edition, November 1986, with all updates
TB	Trip Blanks
TBD	To Be Determined
TOC	Total Organic Carbon
µg/L	Microgram per Liter
UAO	Unilateral Administrative Order
USEPA	United States Environmental Protection Agency
UFP	Uniform Federal Policy
VOC	Volatile Organic Compounds

ATTACHMENT F

LABORATORY STANDARD OPERATING PROCEDURES

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		9/9/11
Quality Assurance Director		9/9/11
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

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. 12

	Signature	Date
QA Director:	Shannon Sawle	9/9/11
Lab Director:	[signature]	9/9/11
Effective Date:	9/16/11	

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. 12

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 High purity helium (99.999%) is used both as GC carrier gas and Purge and Trap purge gas

6.1.4 The instruments scan from amu 35 to 300 at EM voltage similar to those of the tunes.

6.1.5 The BFB data acquisition method is V1TUNE, V2TUNE, V5TUNE, V6TUNE and V10TUNE for each of the five instruments.

6.1.6 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.

6.1.7 The purge and trap systems operating conditions are as follows:

Aqueous (including both 5mL and 25mL samples) and medium soil samples

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 (containing 8cm each of Tanax and silica gel and carbon molecular sieve)
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 (containing 8cm each of Tanax and silica gel and carbon molecular sieve)

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.

6.1.8 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".

6.1.9 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 ≤ 6° C or as recommended by the vendor. The lab stores standards in a separate freezer which is maintained between -10 and -20 ° 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 Tuning:

8.1.1 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.

8.1.1.1 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.**

8.1.1.2 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.

8.2 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.

8.2.1 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.

8.2.2 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:

$$\frac{A_x}{} \quad \frac{C_{is}}{}$$

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

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

8.2.3 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 $\mathbf{r \geq 0.995}$.

(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.

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**.

8.2.4 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.

8.2.5 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.

8.2.6 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.

8.2.7 If necessary, the reference spectra in Target are updated from mid-point calibration (50 ppb standard), or from the continuing calibration standard.

8.2.8 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.

8.2.9 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.

8.3 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.

8.3.1 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.

8.3.2 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

8.3.3 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.

8.3.4 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).

8.3.5 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.

8.4 Sample Analysis - Samples are allowed to warm to ambient temperature before analysis.

8.4.1 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.

8.4.2 Soil samples are analyzed using heated purge for low level analysis and methanol extraction approach for medium level analysis.

8.4.2.1 Low level soil analysis (method SW5035) – 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.

8.4.2.2 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.

8.4.2.3 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.

8.4.2.4 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.

8.4.2.5 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.

8.4.3 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.

8.4.3.1 Low level aqueous sample – Dilutions for aqueous samples are prepared in 40 mL 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; 4 mL for 10X dilution, 1 mL for 40X dilution, 400 uL 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 to the lip with analyte free water and closed. The amount of sample used to

prepare the dilution must be noted on the logbook 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.

8.4.3.2 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.

8.4.3.3 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 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.

8.4.3.4 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.

8.4.3.5 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.

8.4.3.6 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.

8.4.3.7 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.

8.4.3.8 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.

8.4.4 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 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 mL
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 5035, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, Revision 0, December 1996.

5. Environmental Protection Agency. Purge-and-Trap for Aqueous Samples Method 5030, 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)

Figure 1

LIMS standard/spike Logbook
Main page

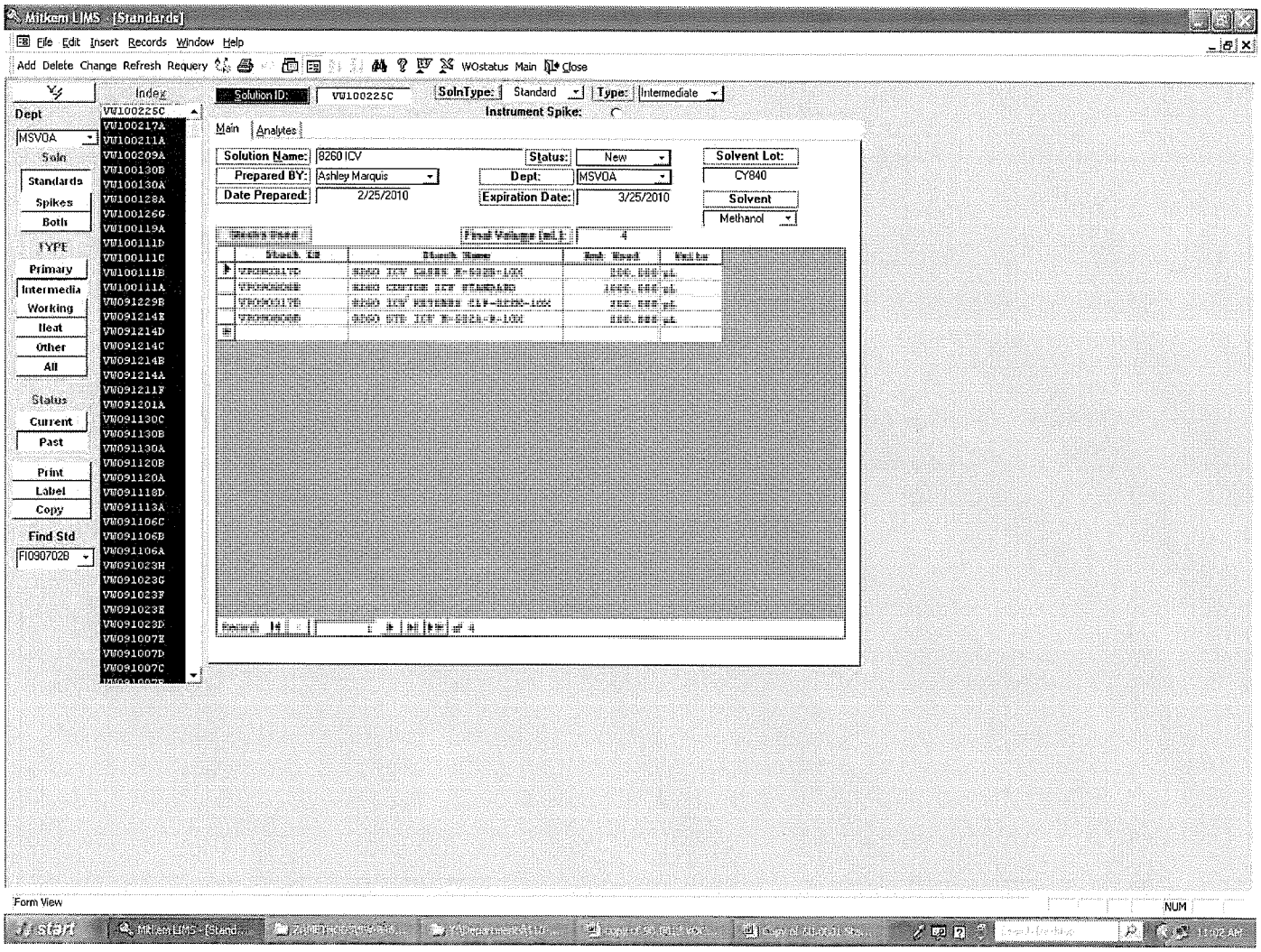


Figure 2

LIMS standard/spike Logbook
Analyte page

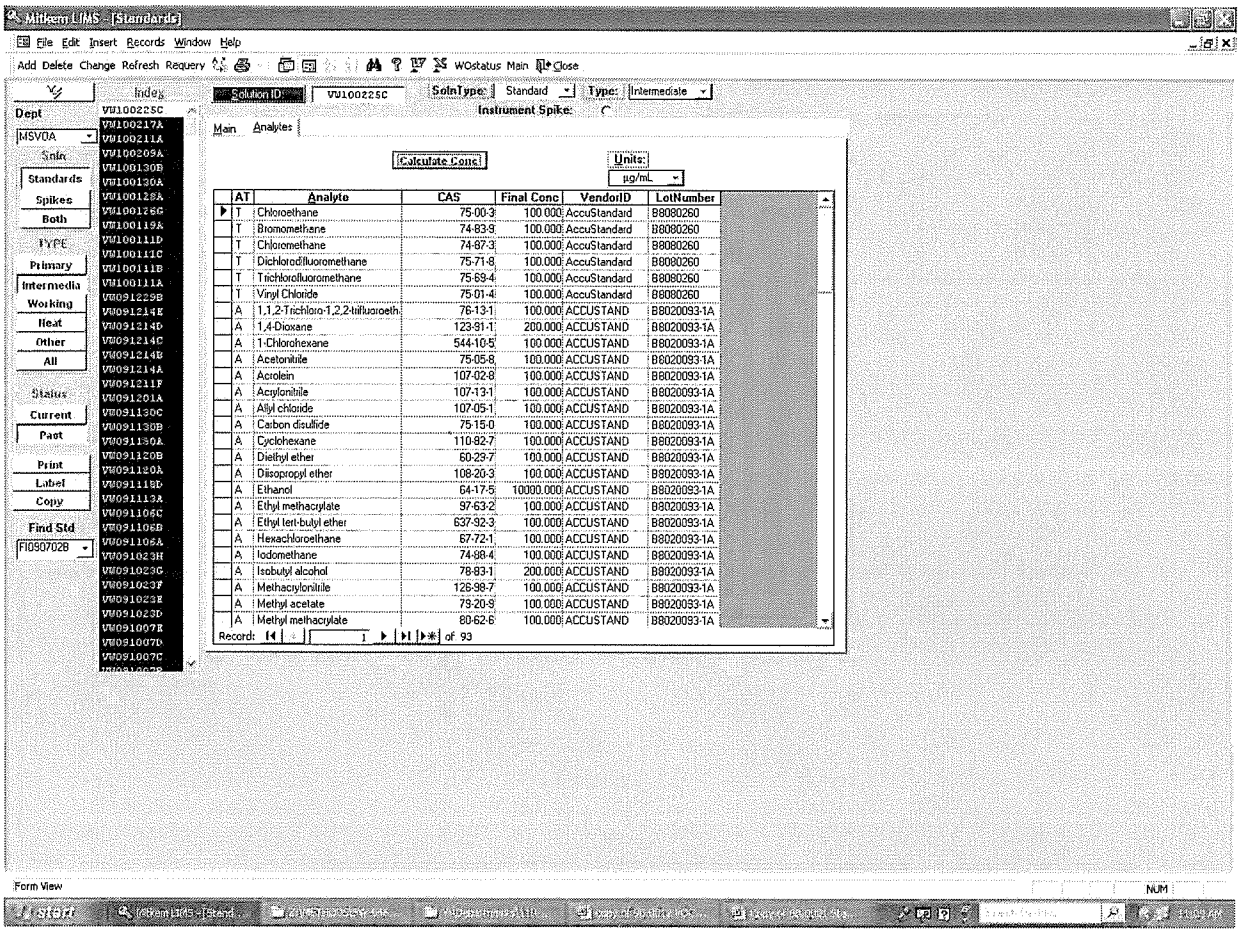


Figure 3

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\V2.I\110614A.B\V2M1580.D

Date : 14-JUN-2011 13:02

Client ID: BFB2F

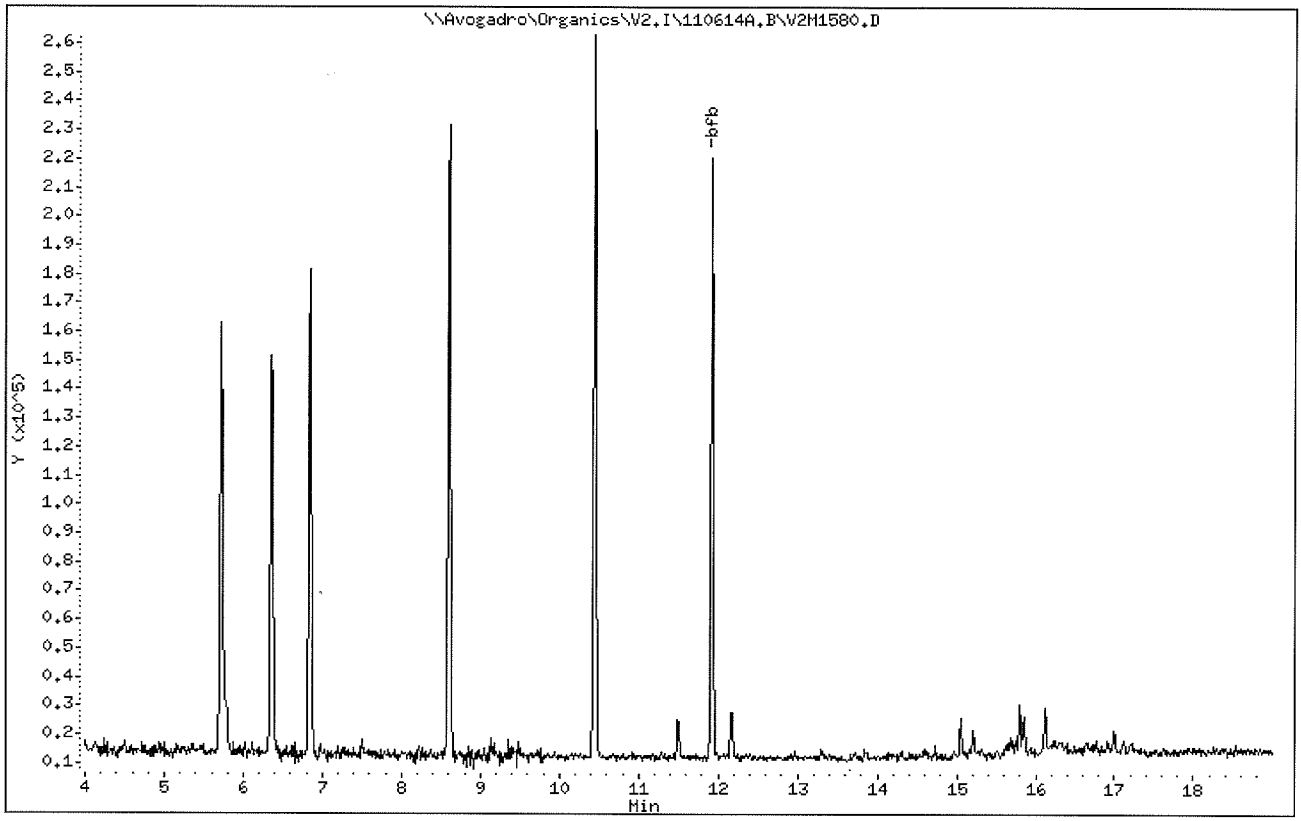
Instrument: V2.i

Sample Info: 2UL,BFB2F,BFB2F

Operator: SRC:

Column phase: DB-624

Column diameter: 0.25



Attachment 3
BFB Tune Mass Spectrum and Ion Abundance Criteria

Data File: \\Avogadro\Organics\V2.I\110614A.B\V2M1580.D

Date : 14-JUN-2011 13:02

Client ID: BFB2F

Instrument: V2.i

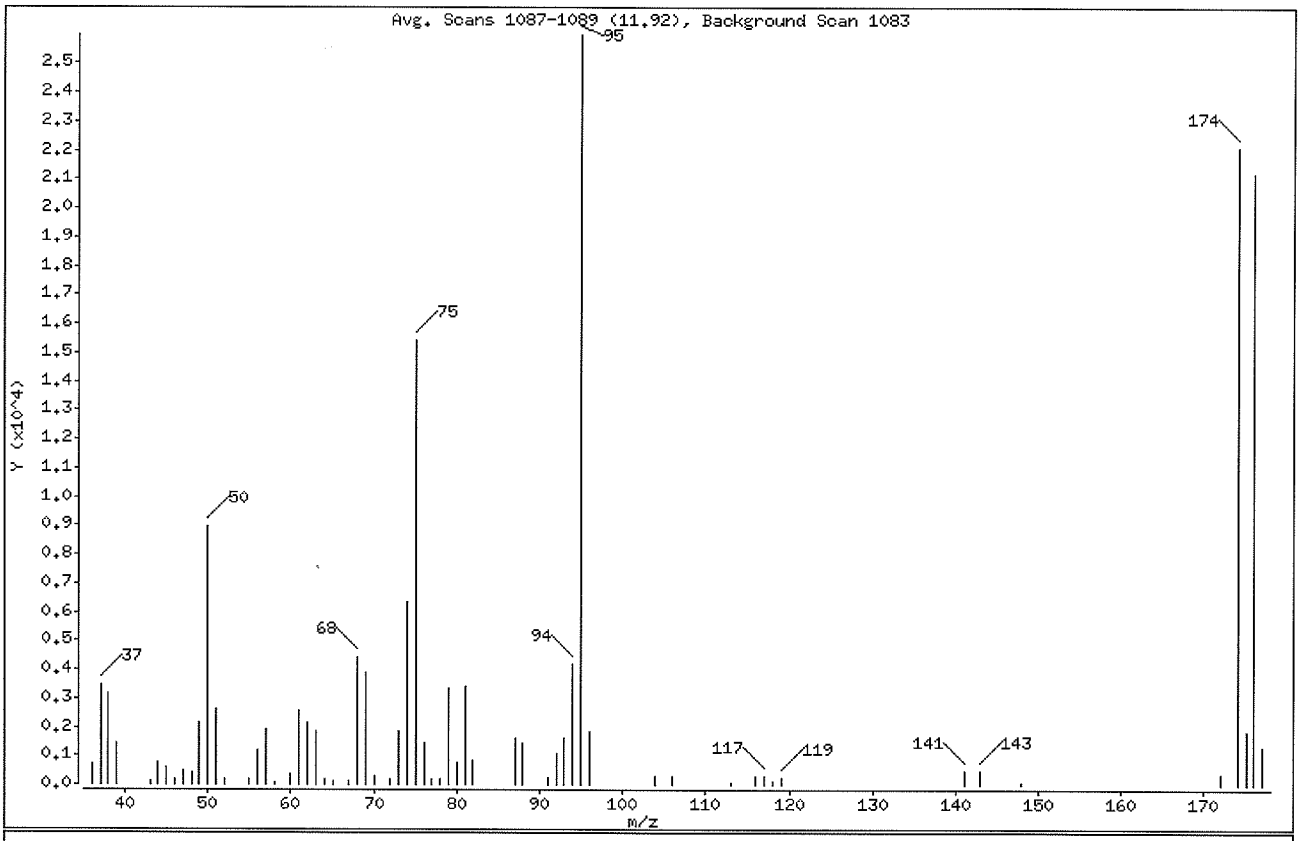
Sample Info: 2UL,BFB2F,BFB2F

Operator: SRC:

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	34.58
75	30.00 - 60.00% of mass 95	59.23
96	5.00 - 9.00% of mass 95	7.04
173	Less than 2.00% of mass 174	0.00 (0.00)
174	50.00 - 100.00% of mass 95	84.98
175	5.00 - 9.00% of mass 174	7.10 (8.36)
176	95.00 - 101.00% of mass 174	81.62 (96.05)
177	5.00 - 9.00% of mass 176	4.95 (6.06)

Data File: \\Avogadro\Organics\V2.I\110614A.B\V2M1580.D

Date : 14-JUN-2011 13:02

Client ID: BFB2F

Instrument: V2.i

Sample Info: 2UL,BFB2F,BFB2F

Operator: SRC:

Column phase: DB-624

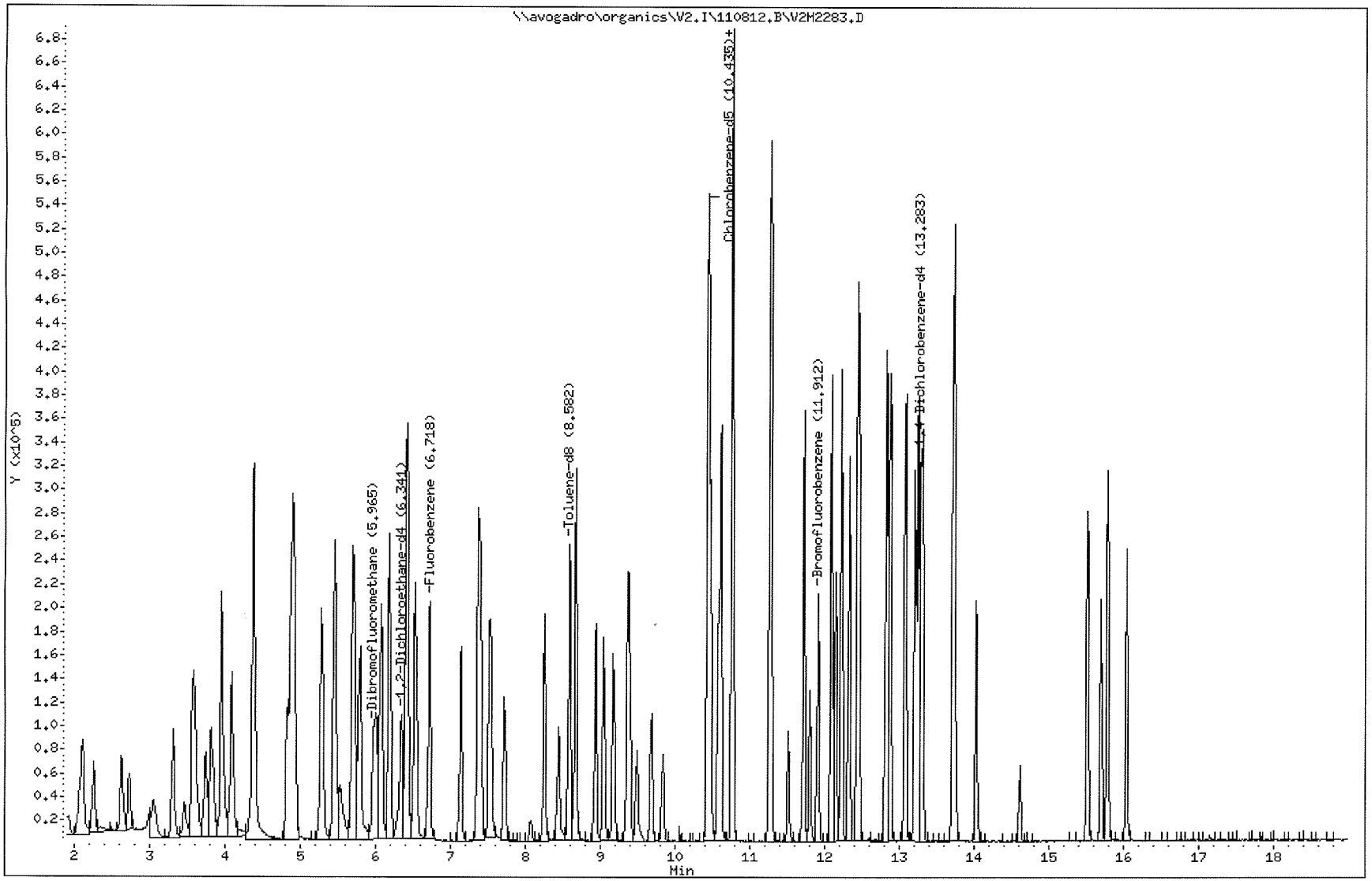
Column diameter: 0.25

Data File: V2M1580.D
Spectrum: Avg. Scans 1087-1089 (11.92), Background Scan 1083
Location of Maximum: 95.00
Number of points: 62

m/z	Y	m/z	Y	m/z	Y	m/z	Y
36.00	741	57.00	1915	76.00	1461	106.00	284
37.00	3444	58.00	67	77.00	177	113.00	72
38.00	3158	60.00	336	78.00	201	116.00	303
39.00	1426	61.00	2564	79.00	3360	117.00	312
43.00	96	62.00	2130	80.00	759	118.00	111
44.00	786	63.00	1850	81.00	3425	119.00	264
45.00	606	64.00	196	82.00	825	141.00	473
46.00	157	65.00	146	87.00	1604	143.00	496
47.00	502	67.00	102	88.00	1432	148.00	67
48.00	419	68.00	4391	91.00	222	172.00	356
49.00	2151	69.00	3900	92.00	1077	174.00	22080
50.00	8985	70.00	306	93.00	1615	175.00	1845
51.00	2619	72.00	203	94.00	4202	176.00	21208
52.00	153	73.00	1866	95.00	25984	177.00	1286
55.00	193	74.00	6319	96.00	1828		
56.00	1221	75.00	15390	104.00	323		

Attachment 4
Chromatogram and Quantitation Report of 50 µg/L Standard

Data File: \\avogadro\organics\V2.I\110812.B\V2M2283.D
Date : 12-AUG-2011 09:23
Client ID: VSTD0502P
Instrument: V2.i
Sample Info: 5ML,VSTD0502P,VSTD0502P
Purge Volume: 5.0
Operator: SRC:
Column phase: DB-624
Column diameter: 0.25



Data File: \\avogadro\organics\V2.I\110812.B\V2M2283.D
Report Date: 15-Aug-2011 15:58

Spectrum Analytical, Inc. RI Division

Method 8260 Water and Medium Soil

Data file : \\avogadro\organics\V2.I\110812.B\V2M2283.D
Lab Smp Id: VSTD0502P Client Smp ID: VSTD0502P
Inj Date : 12-AUG-2011 09:23
Operator : SRC: Inst ID: V2.i
Smp Info : 5ML,VSTD0502P,VSTD0502P
Misc Info :
Comment :
Method : \\Avogadro\Organics\V2.I\110812.B\v28260Gadd-6lvl.m
Meth Date : 15-Aug-2011 15:56 adatta Quant Type: ISTD
Cal Date : 10-AUG-2011 13:58 Cal File: V2M2258.D
Als bottle: 17 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: HP RTE Compound Sublist: all.sub
Target Version: 4.14
Processing Host: TARGET115

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	1.912	1.920	(0.285)	40478	50.0000	40
2	Freon114	85	2.069	2.067	(0.308)	71549	50.0000	47
3	Chloromethane	50	2.101	2.098	(0.313)	94534	50.0000	50
4	Vinyl Chloride	62	2.247	2.245	(0.335)	88093	50.0000	52
5	Bromomethane	94	2.614	2.611	(0.389)	22776	50.0000	48
6	Chloroethane	64	2.718	2.716	(0.405)	60602	50.0000	53
7	Trichlorofluoromethane	101	3.043	3.041	(0.453)	61029	50.0000	56
126	Ethanol	46	3.294	3.302	(0.490)	2589	5000.00	1200(AQ)
8	Ether	59	3.305	3.302	(0.492)	72469	50.0000	52(Q)
9	Acrolein	56	3.451	3.449	(0.514)	60816	250.000	260(A)
10	1,1-Dichloroethene	96	3.566	3.564	(0.531)	58934	50.0000	48
11	1,1,2-Trichloro-1,2,2-Trifluo	101	3.587	3.595	(0.534)	65663	50.0000	54
12	Acetone	58	3.619	3.637	(0.539)	7833	50.0000	36
13	Iodomethane	142	3.734	3.732	(0.556)	117340	50.0000	52
14	Carbon Disulfide	76	3.807	3.805	(0.567)	247838	50.0000	49
15	Acetonitrile	41	3.943	3.941	(0.587)	246106	500.000	530(A)
16	Allyl Chloride	39	3.943	3.941	(0.587)	115372	50.0000	60
17	Methyl Acetate	43	3.964	3.972	(0.590)	93835	50.0000	44
18	Methylene Chloride	84	4.079	4.077	(0.607)	75127	50.0000	44
19	tert-Butanol	59	4.216	4.245	(0.628)	6045	100.000	98
20	Acrylonitrile	53	4.341	4.349	(0.646)	34674	50.0000	50
21	trans-1,2-Dichloroethene	96	4.373	4.370	(0.651)	70615	50.0000	50

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Report Date: 15-Aug-2011 15:58

			QUANT SIG					AMOUNTS	
		Compounds	MASS	RT	EXP RT	REL RT	RESPONSE	CAL-AMT (ug/L)	ON-COL (ug/L)
	22	Methyl tert-butyl ether	73	4.373	4.370	(0.651)	229041	50.0000	55
	23	1,1-Dichloroethane	63	4.823	4.820	(0.718)	136284	50.0000	57
	24	Vinyl acetate	43	4.865	4.862	(0.724)	323901	50.0000	56
	25	Diisopropyl Ether	45	4.886	4.883	(0.727)	330208	50.0000	52
	26	2-Chloro-1,3-Butadiene	53	4.917	4.915	(0.732)	122820	50.0000	58
	27	Ethyl tert-butyl ether	59	5.273	5.271	(0.785)	258209	50.0000	57
	29	2,2-Dichloropropane	77	5.451	5.449	(0.811)	103619	50.0000	59
	28	cis-1,2-Dichloroethene	96	5.441	5.438	(0.810)	75593	50.0000	52
	30	2-Butanone	72	5.451	5.459	(0.811)	10315	50.0000	53
	32	Propionitrile	54	5.524	5.532	(0.822)	124274	500.000	580(A)
	33	Methacrylonitrile	41	5.681	5.679	(0.846)	47169	100.000	100
	34	Bromochloromethane	128	5.702	5.700	(0.849)	37177	50.0000	55
	31	Tetrahydrofuran	72	5.755	5.752	(0.857)	25056	100.000	120
	35	Chloroform	83	5.786	5.784	(0.861)	121268	50.0000	51
$	36	Dibromofluoromethane	113	5.964	5.962	(0.888)	59070	50.0000	54
	37	1,1,1-Trichloroethane	97	6.006	6.004	(0.894)	87550	50.0000	55
	38	Cyclohexane	56	6.069	6.066	(0.903)	137175	50.0000	48
	39	1,1-Dichloropropene	110	6.173	6.171	(0.919)	32856	50.0000	56
	40	Carbon Tetrachloride	117	6.184	6.182	(0.921)	71949	50.0000	58
	41	Isobutyl Alcohol	43	6.289	6.307	(0.936)	52443	1000.00	900(A)
$	42	1,2-Dichloroethane-d4	102	6.341	6.339	(0.944)	17781	50.0000	55
	43	Benzene	78	6.404	6.401	(0.953)	292915	50.0000	53
	44	1,2-Dichloroethane	62	6.425	6.422	(0.956)	102380	50.0000	69
	45	tert-Amyl methyl ether	73	6.519	6.517	(0.970)	239176	50.0000	54
M	50	1,2-Dichloroethene (Total)	96				146208	100.000	100
*	46	Fluorobenzene	96	6.718	6.715	(1.000)	217124	50.0000	
	47	Trichloroethene	130	7.137	7.134	(1.062)	69819	50.0000	54
	48	Methylcyclohexane	83	7.367	7.365	(1.097)	122267	50.0000	48
	49	1,2-Dichloropropane	63	7.398	7.396	(1.101)	79512	50.0000	55
	51	Methyl Methacrylate	69	7.514	7.511	(1.118)	75713	50.0000	55
	52	Dibromomethane	93	7.535	7.532	(1.122)	48700	50.0000	59
	53	1,4-Dioxane	88	7.692	7.689	(1.145)	577	1000.00	820(A)
	54	Bromodichloromethane	83	7.713	7.710	(1.148)	95928	50.0000	52
	55	2-Chloroethyl vinyl ether	63	8.069	8.066	(1.201)	9034	50.0000	55
	56	cis-1,3-Dichloropropene	75	8.247	8.244	(1.228)	133610	50.0000	57
	57	4-Methyl-2-pentanone	43	8.435	8.433	(1.256)	111638	50.0000	58
$	58	Toluene-d8	98	8.582	8.579	(0.822)	199114	50.0000	49
	59	Toluene	91	8.665	8.663	(1.290)	277707	50.0000	52
	60	trans-1,3-Dichloropropene	75	8.938	8.925	(1.330)	120926	50.0000	59
	61	Ethyl Methacrylate	69	9.042	9.040	(1.346)	108874	50.0000	55
	62	1,1,2-Trichloroethane	97	9.168	9.165	(1.365)	62702	50.0000	50
	63	Tetrachloroethene	164	9.356	9.354	(0.897)	54604	50.0000	51
	64	1,3-Dichloropropane	76	9.388	9.385	(0.900)	125004	50.0000	55
	65	2-Hexanone	43	9.482	9.480	(0.909)	84387	50.0000	54
	66	Dibromochloromethane	129	9.681	9.678	(0.928)	72570	50.0000	54
	67	1,2-Dibromoethane	107	9.827	9.825	(0.942)	70654	50.0000	54
	69	1-Chlorohexane	91	10.435	10.432	(1.000)	114706	50.0000	51
*	68	Chlorobenzene-d5	117	10.435	10.432	(1.000)	158606	50.0000	
	70	Chlorobenzene	112	10.466	10.464	(1.003)	174796	50.0000	51
	71	1,1,1,2-Tetrachloroethane	131	10.571	10.568	(1.013)	62039	50.0000	54
	72	Ethylbenzene	106	10.613	10.610	(1.017)	90261	50.0000	50
	73	m,p-Xylene	106	10.759	10.757	(1.031)	229958	100.000	100
	74	o-Xylene	106	11.262	11.259	(1.079)	109994	50.0000	50
	75	Styrene	104	11.283	11.280	(1.081)	202616	50.0000	50

Data File: \\avogadro\organics\V2.I\110812.B\V2M2283.D
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		Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ug/L)	AMOUNTS ON-COL (ug/L)
	76	Bromoform	173	11.513	11.511	(1.103)	52617	50.0000	52
	77	Isopropylbenzene	105	11.723	11.720	(1.123)	282855	50.0000	49
	78	trans-1,4-Dichloro-2-butene	75	11.806	11.804	(1.131)	39236	50.0000	56
$	79	Bromofluorobenzene	95	11.911	11.909	(1.141)	80841	50.0000	52
	80	1,1,2,2-Tetrachloroethane	83	12.089	12.087	(0.910)	101225	50.0000	52
	81	Bromobenzene	156	12.099	12.097	(0.911)	76749	50.0000	52
	82	1,2,3-Trichloropropane	75	12.141	12.139	(0.914)	119689	50.0000	56
	83	n-Propylbenzene	120	12.225	12.223	(0.920)	73025	50.0000	51
	84	2-Chlorotoluene	126	12.330	12.327	(0.928)	67535	50.0000	52
	85	1,3,5-Trimethylbenzene	105	12.435	12.432	(0.936)	227145	50.0000	50
	86	4-Chlorotoluene	126	12.455	12.453	(0.938)	70673	50.0000	52
M	94	Xylene (Total)	106				339952	150.000	150
	87	tert-Butylbenzene	119	12.822	12.819	(0.965)	266016	50.0000	52
	88	1,2,4-Trimethylbenzene	105	12.874	12.872	(0.969)	233798	50.0000	50
	89	sec-Butylbenzene	105	13.084	13.081	(0.985)	321985	50.0000	50
	90	1,3-Dichlorobenzene	146	13.209	13.207	(0.994)	142434	50.0000	51
	91	4-Isopropyltoluene	119	13.251	13.249	(0.998)	245227	50.0000	51
*	92	1,4-Dichlorobenzene-d4	152	13.283	13.280	(1.000)	84513	50.0000	
	93	1,4-Dichlorobenzene	146	13.314	13.312	(1.002)	148368	50.0000	52
	95	n-Butylbenzene	91	13.722	13.720	(1.033)	266585	50.0000	51
	96	1,2-Dichlorobenzene	146	13.743	13.741	(1.035)	137554	50.0000	50
	97	Hexachloroethane	117	14.026	14.024	(1.056)	57885	50.0000	54
	98	1,2-Dibromo-3-chloropropane	75	14.612	14.610	(1.100)	16816	50.0000	68
	99	1,2,4-Trichlorobenzene	180	15.523	15.521	(1.169)	95441	50.0000	47
	100	Hexachlorobutadiene	225	15.701	15.699	(1.182)	44204	50.0000	49
	101	Naphthalene	128	15.785	15.782	(1.188)	258457	50.0000	47
	102	1,2,3-Trichlorobenzene	180	16.047	16.044	(1.208)	88854	50.0000	45

QC Flag Legend

A - Target compound detected but, quantitated amount exceeded maximum amount.
Q - Qualifier signal failed the ratio test.

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

Title: Table II A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8260B

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		

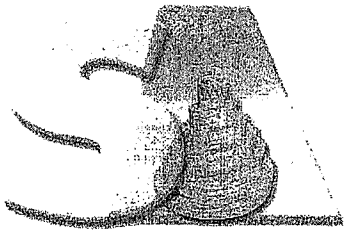
compound above reporting limit.	5. Investigate source of problem. Reanalyze all effected samples. If reanalysis is within holding time, report only these analyses. If they are beyond holding time, report both sets and notify project manager. If contaminant is not present in samples, data may be released 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.	

NY Lab #11393/11840
FL Lab #E87600/E87936



SPECTRUM ANALYTICAL, INC
Featuring
Hanibal Technology
11 Almgren Drive
Agawam, MA 01001

Standard Operating Procedure
For
METHOD TO-15
Determination of Volatile Organic Compounds (VOCs) in Air collected in Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)

Prepared by	11/15/10 Date
Reviewed by	11/15/10 Date
Lab Director	11/15/10 Effective Date

STANDARD OPERATING PROCEDURE
for
Air Analysis Method TO-15

I. SCOPE AND APPLICATION

This method is applicable to the determination of VOC's in whole air sampling. It is for the sampling, analysis and measurement of subsets of the 97 VOCs that are included in the 189 hazardous pollutants (HAPs) listed in Title III of the Clean Air Act Amendments of 1990. The samples are collected in either Tedlar bags or Summa Canisters (specially prepared canisters such as silcosteel or siloniate). The VOC's are separated by gas chromatography and detected by a mass spectrometer. The VOC's in this method have been tested and determined to be stable in pressurized and sub-ambient canisters. These compounds are detected at low PPBv levels. This SOP covers the TO-14 method also. TO-14 specifies a nafion dryer, which is outdated technology. For our intents and purposes, TO-14 specifies a subset of TO-15 target analytes, which are specified in the compounds of interest table in section XX.

II. SUMMARY OF METHOD

A. Samples are introduced into specially prepared canisters under vacuum either by sub-atmospheric pressure or by pressurizing canister. After collection the canister valve is closed, labeled and sent to the laboratory for analysis. Samples are logged in and the canister pressure is recorded from the gauge on the cans (if canister does not have a gauge, attach a vacuum gauge to valve and record pressure). Tedlar bags do not have the pressure recorded. If the canister is below -10 psig, the can must be pressurized to 0 psig. The client information on the canister is then verified comparing the COC to the canister and is then shelved to wait for analysis.

B. Analysis consists of placing the sample on a 16 position Entech autosampler/ concentrator. An aliquot of sample (mls) is drawn from the canister and measured by a mass flow controller (MFC). The sample is preconcentrated removing water and CO2 from the sample. It is then transferred to the GC where separation of the compounds occurs. The compounds are detected by a quadrapole low-resolution mass spectrometer in full scan mode. After analysis, data is reviewed and approved. The canister is removed from the autosampler and put onto the canister cleaner for cleaning, then storage ready for reuse.

C. Method TO-15 uses a water removal system which employs a technique of trapping the sample on a multisorbent /dry purge trap using a cryogenic cooling system to bring the sample to –190 degrees C. This is the most effective technique for water removal. This technique allows for a more extensive compound list then method TO-14A due to this water management system (method TO-14A uses a naphion dryer).

III. DEFINITIONS

A. Whole Air Sample – An air technique that does not selectively sample certain components onto a specific sampling media. Instead, an aliquot of air is drawn into a passivated sampling canister and a measured amount of air is used as the sample.

B. PPBv – Parts Per Billion by Volume

C. PPMv – Parts Per Million by Volume

D. Subatmospheric Sampling – collection of an air sample in an evacuated canister at a (final) canister pressure below atmospheric pressure, without the assistance of a sampling pump. The canister is filled as the internal canister pressure increases to ambient or near ambient pressure.

E. Pressurized Sampling – collection of an air sample in a canister with a (final) Canister pressure above atmospheric pressure, using a sample pump.

F. Cryogenic Cooling – A refrigerant used to obtain very low temperature in the cryogenic trap of the analytical system. A typical gas used for cryogenic cooling is liquid Nitrogen (bp – 195.8 Celcius) or liquid Argon (bp – 185.7 Celcius).

1. Mass Flow Controller – A device that is able to measure the flow of gases in milliliters.

2. Summa Canister – Summa is a trade name for the sampling container of a whole air Sample. The container is either made from electropolished stainless steel or an ineret coating of the surface. Canisters with the inert coating are called Passivated Sampling Canisters.

3. Flow Controllers – Passivated devices that attach to the top of the canister and regulate the flow of air into the canisters. Controllers can be set from 0.5 hrs to week for a sampling time duration.

4. Grab sample – Sample taken without the use of a flow regulator.

IV. INTERFERENCES

A. Interference can occur from fuel pattern areas, saturated targets/ non-targets, or an excess of moisture that is not removed by the instrument. Moisture buildup is also possible if many saturated samples are analyzed consecutively.

B. Improper cleaning of Summa Canisters and Passive Flow Controllers will result in contaminated equipment.

V. APPARATUS AND MATERIALS

A. Summa Canisters - 6 Liters (silcosteel or silonite)

B. Passive Flow Controllers (silcosteel or silonite)

C. Entech autosampler and concentrator

D. Hewlett Packard 6890 Gas Chromatograph

E. Hewlett Packard 5973 MSD

F. Restek RTX-1, 60 M, 0.32mm ID, 1.0 um df chromatographic column

G. Entech 4620 Dynamic Diluter, 5 channel

H. Entech 3100 Canister Cleaner, 16 position

VI. REAGENTS

A. Liquid and Vapor N2

B. Chromatographic grade He

C. Internal Standards (4 cmpds) -1PPMv Spectra Gases

D. TO-15 Calibration Mix (62 cmpds) -1 PPMv Spectra Gases

E. Quality Control Std. (62 cmpds) –1PPMv Spectras Gases (independent formulation from calibration TO-15 std)

F. 4-Bromofluorobenzene Tuning Solution (part of internal standard mix)

VII. SAFETY

To maintain the application of OSHA regulations regarding the safe handling of the chemicals specified in this method, the laboratory must follow proper safety procedures:

A. All chemicals should be transported on a cart when moved from room to room.

VIII. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

A. Whole air sampling can be performed with either Summa Canisters or Tedlar bags. Summa Canister – sampling can occur either by taking Grab samples (10 sec – 30 sec) or can be sampled over a period of time by using a passive flow controller. The canister is under vacuum (-30 in Hg). This vacuum draws the sample (air) into the canister when the valve is open. The sample is drawn into the canister based on the pressure difference. A grab sample is taken immediately without the use of a flow controller. It takes 10 – 30 seconds to fill a 6 Liter without the controller. This is used in acute situations and data should be interpreted as screen data only. Another technique is using passive flow controllers. Passive flow controllers restrict the flow of air drawn into the canister. They can be regulated at 15min, 30min, and between 1hr through 1 week. The longer time duration of sampling allows for a more representative sample. Any time interval less then 2 hrs is considered a screen.

B. Passive flow controllers are cleaned with nitrogen between uses and recalibrated prior to returning it to the field.

IX. QUALITY CONTROL

The quality control associated with this method is modified for additional QC then required by method. The additional quality control implemented is as follows:

1. Independent laboratory fortified blank
2. Internal standard criteria
3. Addition and reporting of a surrogate standard
4. Method blank required before analytical sequence

X. CALIBRATION

A. 4-Bromofluorobenzene(BFB) – Tuning Std.
100 ng BFB is injected into instrument prior to running initial calibration or continuing calibration. BFB criteria is as follows:

MASS	ION TUNING CRITERIA
50	8.0 to 40% of mass 95
75	30 to 66^ of mass 95
95	Base peak, 100% relative abundance
96	5 to 9 % of mass 95
173	<2% of mass 174
174	>50% to 120% of mass 95
175	4 to 9% of mass 95
176	>93% but <101
177	5 to 9% of mass 174

Standard and sample analysis is valid for 24 hours following valid BFB.
The calibration mixes are blended using the Entech 4600 Dynamic Diluter. Two standards are prepared. The lower concentration is a 10 PPBv and the other is a 50 PPBv. The calibration consists of at least a five-point curve. Stds are at concentration of 0.5PPBv, 2PPBv, 5PPBv, 10 PPBv, 20PPBv, 25PPBv, 50 PPBv and 100 PPBv. The 0.5PPBv, 2 PPBv, 5 PPBv, 10 PPBV, and 20PPBv stds are analyzed from the 10 PPBv std and the 25 PPBv, 50 PPBV and 100 PPBv. Stds are analyzed by the 50 PPBv std. The different concentration is based on volume adjustment of the working std.

A 10 PPBv std is used as a daily continuing std. The QC std is prepared and analyzed at 10 PPBv.

Initial calibration requires %RSD of 30% for all cmpds, with two exceptions up to a limit of 40%.

Continuing calibration requires a %D of 30.

Method Blanks are analyzed before samples and after stds. A blank consists of a Summa Canister pressurized with N2 and carried through the same analytical process as the samples. Any components detected in the blanks are required to be below the MDL before analyzing samples.

B. Internal Standards are compounds added to sample that are unlikely to be found in environmental samples (duterated compounds are commonly used). The response is similar to the compounds detected in the samples. The internal standards are as follows:

Bromochloromethane
1,4-Diflurobenzene
Chlorobenzene-d5

The IS criteria is + or – 50% of the midpoint area response from the most recent calibration for the daily CCV. Samples must be within 50% of the daily CCV. The retention time must be + or – 0.33 min from the retention time of the most recent calibration.

XI. PROCEDURE & CALCULATIONS

A. Canister pressure is recorded from gauge(psig) on cans or manually attach a vacuum gauge to valve and open it. If canister is < -10 psig, the canister will need to be pressurized to 0 psig (14.0 psia). This will be performed by the dynamic diluter and a dilution factor will result. The canister(or Tedlar Bag) is attached to the 16 position autosampler. All valves are keep closed and a leak check and line flush are performed for each sample line. This procedure verifies that no leaks are present and that the lines are clean. After this procedure the valves are opened.

B. Analysis of samples begin after tuning std, continuing calibration, blanks and QC pass criteria. A sequence is set up on both the Entech system and on the HP MSD. An aliquot is withdrawn from each canister, preconcentrated, separated and analyzed by the MSD.

C. After analysis all samples are checked for surrogate, IS criteria and saturation of compounds. If target compounds exceed the linear range of the curve, a dilution of the sample is necessary. This position on the autosampler will require flushing, possibly several times due to the type and concentration of contamination.

D. Calculations are based on the internal standard technique.

Cx = AxCisDF/AisRRF
Cx = compound concentration, ppbv
Ax = Area of the characteristic ion for the compound to be measured, area counts
Ais = Area of the characteristic ion for the specific internal standard, area counts
Cis = Concentration of the internal standard spiking mixture, ppbv
RRF= Relative response factor from the continuing calibration level of the initial curve
DF = Dilution factor

E. Canister Cleaning.

All canisters are cleaned after sample analysis and prior to reusing. They are cleaned on a 16 position Entech Canister Cleaner. Canister with pressurized samples should be vented into a hood prior to placing in cleaner. Canisters are attached and fittings are tightened. A leak check is performed and the can heaters are heated to 100 degrees C. The valves are opened and a roughing pump evacuates the canisters.

Each canister is evacuated and then pressurized with synthetic air. All 16 cans will cycle through this process 3 or more times. Each cycle will roughly take 1.5 hrs. At the completion of the third cycle, the molecular drag pump is activated and all canisters are brought to a vacuum of < 50 millitorr. The pump down with the molecular drag pump is about 1.5 hrs – 2 hrs. The total canister cleaning time can be 6-7 hrs.

At the completion of the process, one canister is chosen for batch analysis. It is pressurized to 30 psig with N2 and then analyzed. If all compounds are below the MDL of the method it is considered clean. Upon completion of analysis the canister is put back on the cleaner and brought to < 50 millitorr. All canisters from that cleaning batch are labeled, documented in a cleaning logbook and placed in storage. The canister will then stay there until prepped prior to being sent out.

XII. METHOD PERORMANCE

The method for determining the MDL is to analyze 7 samples of concentration at 2 times the expected limit of detection. The standard deviation is then determined. The one-sided t distribution is determined and multiplied versus the determined standard deviation. For seven samples (with six degrees of freedom) the t value for a 99% confidence interval is 3.14.

XIII. POLLUTION PREVENTION

The air laboratory is designed to eliminate any pollution that may be generated by the laboratory. Procedures are in place to deal with pollution from either samples or standards. The laboratory air is recirculated through additional air scrubbers to maintain a volatile free atmosphere within the laboratory.

All instrumentation for standards formulation and sample analysis are based on closed systems to eliminate exposure to the atmosphere. If a neat std is used to formulate a std, a hood is used to prevent any possible contamination or pollution. High level samples are flushed in a hood prior to placing on the canister cleaner to avert any contamination from this source.

Additional laboratory procedures for pollution prevention can be found in the chemical hygiene plan.

XIV. DATA ASSESSMENT ACCEPTANCE

The criteria for acceptable data is as follows:

A. BFB passes performance std criteria (opens a 24 hr window)
B. Initial calibration < 30% RSD with two exceptions limited to <40% RSD.
C. Continuing calibration + or – 30% D.
D. Surrogate recovery for all samples 70% - 130%
E. Laboratory Control Sample limits 70% - 130%.
F. Contamination free method blanks.

XV. CORRECTIVE ACTION

Any data that is not within acceptable criteria is considered out-of-control. Each is evaluated on a case by case basis to determine the cause of the problem ant the corrective action for the data. Generally, reanalysis occurs, but occasionally data is flagged.

XVI. CONTINGENCIES FOR OUT-OF-CONTROL DATA

The procedure for handling out-of-control data begins at the analyst level. If data of this quality is generated it is brought to the attention of the laboratory manager. The laboratory manager reviews the data, the cause and the different options available. The laboratory manager may discuss the data with the quality control dept and the laboratory director. A final decision is made and documented.

XVII. WASTE MANAGEMENT

Refer to the Laboratory Chemical Hygiene Plan.

XVIII. REFERENCES

EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-15, Determination of Volatile Compounds (VOCs) in Air Collected in Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)

XVIV. APPENDIX

APPENDIX A

FORMALDEHYDE

In order to run formaldehyde, the amu scan range must be lowered to 29-180, and the solvent delay must be removed. The temperature ramp is changed to 35 degrees to 5 minutes, 10 degrees to 85, then 25 degrees to 210 and hold for 3 minutes. These changes are set in the acquisition method 'FORM'.
Module 1 on the 7100 must be switched to an empty trap (Entech #1 stamp), and the method must be switched to cold trap dehydration (CTD).

LEAD

In order to run lead – Tetramethyl and Tetraethyl (TML, TEL), the amu scan range must be raised to run up to 310. This is set in the acquisition method 'LEAD'.

APPENDIX B

TO-15 LOW LEVEL ANALYSIS

The low-level calibration is based on a volume of 1000mls. Calibration standards are run from a prepared 10ppbv standard at levels of 0.1, 0.2, 0.5, 1.0, 2.0, 5.0, and 10ppbv. This method can only be run from 6l cans where the sample is not expected to have target concentrations greater than 10ppbv. Duplicate samples must be collected in separate cans, as there is not enough sample volume to run duplicate's from the same can. All canisters must be individually certified clean; batch cleaning is not acceptable for the low-level analysis.

TO-15 LOW LEVEL ANALYSIS VIA SIM

Along with the above listed parameters, a standard of 0.04ppbv is run for 5 compounds via

SIM during the full scan analysis. The primary and secondary ions are as follows: (TCE 95,132), (PCE 166,129), (1,1-DCE 96,61), (1,1-DCA 63,65), (CTET 119,117) and (VC 62,64).

APPENDIX C

TO-15 SIM ANALYSIS

SIM analysis is utilized to achieve detection limits as low as 0.01ppbv (10pptv). All instrument settings are the same as full scan analysis. The MS is set to SIM and groups are set to analyze only for primary and secondary ions for each analytes that are determined by time reference from a full scan of a calibration standard. The current SIM group settings on AIR2 are:

START TIME	GROUP	IONS
0.00	1	39,42,50,52,85,87
4.8	2	39,45,46,50,52,53,54,62,64,66,85,94,96,135
6.15	3	43,45,49,58,61,76,84,96,101,103,151
8.17	4	43,45,57,61,63,65,72,73,83,85,86,96,98,128,130
10.08	5	43,62,64,71,72
10.65	6	56,57,63,71,78,83,84,85,95,97,99,112,114,117,119, 130,132
13.5	7	43,57,58,75,83,91,92,97,107,109,110,127,129
16.35	8	82,112,114,117,129,166
17.75	9	83,91,103,104,106,131,173,175
19.43	10	95,174
20.61	11	91,105,120,126,146,148
24.93	12	120,126,146,148

Start times, number of groups and ions in each group will vary from instrument to instrument and depend on the analyte list that is needed for SIM analysis.
The SIM standard is blended on the 4600 dynamic diluter. The 1ppm calibration standard is mixed with N2 at 3996ml N2 + 4ml standard into a 6 liter summa canister to make a 1ppbv working standard. The calibration is based on 1000mls and is run from this mixture at levels of 0.01, 0.02, 0.04, 0.1, 0.25, 0.5, and 1.0ppbv. Calibrations, standards, QC's, blanks, and samples are all processed, integrated, and reported in the same manner as full scan TO-15.

APPENDIX D

TVH ANALYSIS

TVH or Total Volatile Hydrocarbons is a calculation of the entire chromatographic run of a TO-15 sample from approximately 0.5-25 minutes and is based on the response of Toluene in the daily CCV. The final unit for TVH is PPBv, but due to varying molecular weights of all of the individual peaks it will be a closely estimated value.

Calculations for TVH are as follows:

To obtain the RF for samples from the daily CCV=

$$\frac{\text{TIC area of Toluene}}{\text{Ion area of 1,4-Difluorobenzene}} \times \frac{\text{Concentration of 1,4-Difluorobenzene}}{\text{Concentration of Toluene}}$$

Sample Calculation=

Total sample area (integrated from end of injection peak to end of chromatogram)
-TIC areas of internal standards and surrogate
(Bromochloromethane, 1,4-Difluoromethane, Chlorobenzene-D5, 4-Bromofluorobenzene)
= Adjusted sample area
÷ Ion area of 1,4-Difluorobenzene
X Concentration of 1,4-Difluorobenzene ÷ RF (From CCV)
X Dilution factor when applicable
= TVH

Note: Toluene area from client samples is not used in the calculation.

XX. ANALYTE LIST

Analytes marked with an * are TO-14 compounds.

PROPENE
*DICHLORODIFLUOROMETHANE
*CHLOROMETHANE
*FREON 114
*VINYL CHLORIDE
1,3-BUTADIENE
*BROMOMETHANE
*CHLOROETHANE
ACETONE
*TRICHLOROFLUOROMETHANE
ETHANOL
ACRYLONITRILE
*1,1-DICHLOROETHENE
*METHYLENE CHLORIDE
*FREON 113
CARBON DISULFIDE
*TRANS-1,2-DICHLOROETHENE
*1,1-DICHLOROETHANE
MTBE
IPA
2-BUTANONE(MEK)
*CIS-1,2-DICHLOROETHENE
HEXANE
1,4-DIOXANE
HEPTANE
MIBK
*CIS-1,3DICHLOROPROPENE
*TRANS-1,3-DICHLOROPROPENE
*1,1,2-TRICHLOROETHANE
*TOLUENE
2-HEXANONE(MBK)
DIBROMOCHLOROMETHANE
*1,2-DIBROMOETHANE
*TETRACHLOROETHENE
*CHLOROBENZENE
1,1,1,2-TETRACHLOROETHANE
*ETHYLBENZENE
*M/P-XYLENE
BROMOFORM
*STYRENE
*O-XYLENE
*1,1,2,2-TETRACHLOROETHANE
ISOPROPYLBENZENE
*1,3,5-TRIMETHYLBENZENE
*4-ETHYLTOLUENE
*1,2,4-TRIMETHYLBENZENE

ETHYL ACETATE

*CHLOROFORM
TETRAHYDROFURAN
*1,2-DICHLOROETHANE
*1,1,1-TRICHLOROETHANE
*BENZENE
*CARBON TETRACHLORIDE
CYCLOHEXANE
*1,2-DICHLOROPROPANE
BROMODICHLOROMETHANE
*TRICHLOROETHENE

*1,3-DICHLOROBENZENE

*BENZYL CHLORIDE
*1,4-DICHLOROBENZENE
SEC-BUTYLBENZENE
4-ISOPROPYLTOLUENE
*1,2-DICHLOROBENZENE
n-BUTYLBENZENE
*1,2,4-TRICHLOROBENZENE
*HEXACHLOROBUTADIENE

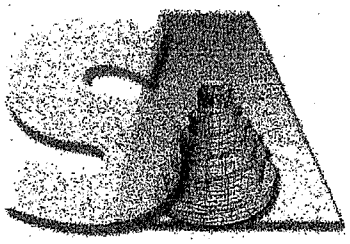
XXI. REVISIONS

Rev. 7
Section I: Updated to include the TO-14 method.
Section II: Corrected can pressures.
Section X: Updated calibration points and internal standard response criteria.
Section XII: Added correct formula for MDL requirements.
Section XX: Added correct analyte list for both TO-15 and TO-14.
Appendix B: Added to include low level analysis.
Appendix C: Added to include SIM analysis
Appendix D: Added to include TVH analysis

Rev. 8
Appendix B: Added low level SIM analysis

Rev. 9
Appendix B: Added 1,1-DCA and ions to Low level SIM

ACCREDITED IN ACCORDANCE WITH NELAC
NY Lab #11393/11840
FL Lab #E87600/E8793



SPECTRUM ANALYTICAL, INC.
Featuring
Hanibal Technology
11 Almgren Drive
Agawam, MA 01001

Standard Operating Procedures
For
Modified EPA 3C / RSKSOP-175
Determination of Fixed Gases and Dissolved Gases in Gaseous and Aqueous phase matrixes
By TCD

Prepared by: _____ Date: 11/20/09

Reviewed by: _____ Date: 11/20/09

Laboratory Director: _____ Date: 11/20/09

TABLE OF CONTENTS
STANDARD OPERATING PROCEDURES
FOR
Modified EPA 3C / RSKSOP-175
Determination of Fixed Gases and Dissolved Gases in Gaseous and Aqueous phase matrixes
By TCD

I. METHOD SCOPE AND APPLICATION

A. This method is designed to measure the gaseous-phase concentrations of Fixed gases in air and dissolved fixed gases in water. Fixed gases of interest are as follows:

1 Carbon Dioxide (CO2)
2 Carbon Monoxide (CO)
3 Methane (CH4)
4 Hydrogen (H)
5 Nitrogen (N)
6 Oxygen (O)
7 Ethene
8 Ethane
9 Acetylene

II. METHOD SUMMARY

A. Gaseous phase samples are introduced into specially prepared canisters under vacuum either by sub-atmospheric pressure or by pressurizing the canister. After collection, the valve is closed, labeled, and returned to the laboratory for analysis. Samples are logged in, client information is verified against the COC, and the canister pressure is recorded from the gauge on the can. If the canister is below 15 "Hg, the can may need to be pressurized to allow sampling.

B. Aqueous samples are collected in 40ml unpreserved amber vials without headspace. These samples follow the same procedure as the Gaseous phase samples once they have reached the laboratory.

C. Analysis consists of attaching the can or vial to the inlet of the Perkin-Elmer/Arnell GC where an aliquot of sample is loaded onto a loop with a pump from where it is desorbed onto the capillary column in the GC then analyzed by the TCD. After analysis, data is reviewed and approved, or rerun if it is rejected. Minimum reporting limit should be 10PPMv for Dissolved gasses and 1,000ppmv for Fixed gasses.

III. DEFINITIONS

A. Whole Air Sampling – An air sample technique that does not selectively sample specific compounds onto a specific sampling media. An aliquot of air is drawn into a passivated sampling canister and a measured amount of sample is used for analysis.
B. PPBv – Parts per Billion by Volume
C. PPMv – Parts per Million by Volume
D. Sub-atmospheric Sampling – Collection of an air sample in an evacuated canister with a final canister pressure below atmospheric pressure without the assistance of a sampling pump. The canister is filled as the internal pressure equilibrates with the external pressure to near ambient pressure.
E. Summa Can – Summa is a trade name for the sampling container for whole air sampling. The container is either made from Electro-polished stainless steel or has an inert coating applied to the inner surface. Canisters with inert coating are referred to as Passivated Sampling Containers.
F. GC – Gas Chromatograph.
G. TCD – Thermal Conductivity Detector.
H. Passive Flow Controller – A calibrated metering device that is attached to a Summa Canister to control flow into the container at a fixed rate (5 minutes to 7 days).

IV. HEALTH AND SAFTEY

A. To maintain the application of OSHA regulations regarding the safe handling of chemicals specified in the method, the laboratory must follow proper safety procedures as outlined in Spectrum Analyticals Chemical Hygiene Plan.

V. CAUTIONS

A. The operating temperature of the TCD is 200 degrees.

VI. INTERFERENCES

A. Improper cleaning of Summa Canisters and Passive Flow controllers may result in contaminated equipment.

VII. PERSONNEL QUALIFICATIONS

A. Analysts must have a working knowledge of the Perkin-Elmer GC equipped with a TCD.
B. Must have experience with the dilution of Air standards to generate a calibration curve.

VIII. APPARATUS AND MATERIALS

A. Summa Canisters – 6 or 3.2 Liter with SilcoSteel or Silonite coating.
B. Passive Flow Controllers - With SilcoSteel or Silonite coating.
C. Perkin-Elmer / Arnel Clarus 500 GC.
D. 7' HayeSep N 60/80, 1/8" SF column
E. 9' Molecular Sieve 13x45/60, 1/8" SF column
F. Barnant Company Vacuum Pressure Station. (Pump)

IX. REAGENTS AND STANDARDS

A. 6 Component Fixed gas calibration standard at 5%.
B. 6 Component Fixed gas calibration standard at 1%
C. 9 Component Dissolved gas calibration standard at 1000PPMv.
D. Chromatographic grade He.

X. STANDARD PREPARATION

A. Standard is drawn from the pressurized standard cylinders into clean Tedlar Bags. From there they are diluted into separate Tedlar Bags with He.

XI. CALIBRATION

A. For Dissolved Gasses: Standards are diluted at a minimum of 5 different levels and injected onto the sample loop to create the calibration curve.

Standard dilutions are usually: 100x, 50x, 10x, 2x, and 1x to create a calibration range from 10PPMv to 1000PPMv.

B. For Fixed gasses: Standards are diluted at a minimum of 3 levels to create a calibration range from 1,000ppm to 50,000ppmv. For Oxygen the range is typically 10,000 – 100,000ppmv, for Nitrogen 10,000 – 200,000ppmv.

C. Initial calibration criteria for both Fixed and Dissolved is 30%RSD or 0.99 linear regression.

D. ICV must pass at 30%

E. CCV must pass at 30%

XII. SAMPLE COLLECTION, PRESERVATION AND STORAGE

A. Tedlar bag collection for Fixed gas analysis has a holding time of 5 days.

B. Summa Canister – Sample collection can be done via grab sampling or by time weighted average. Grab sampling is simply opening the valve on the canister until the can is just under ambient pressure. This process usually takes 10-30 seconds. Time weighted average sampling is done by attaching a pre-regulated passive flow controller to the can prior to opening the valve. Regulators can be set for 5 minutes, ½ hour, or anywhere from 1 hour to 7 days. This method creates a more representative sample. Samples are then transported to the lab, logged in, client information checked, then run for analysis. Recommended holding time for Fixed gas compounds in Summa canisters is 28 days.

C. Aqueous samples are collected in VOA vials with no preservative added. Samples are then transported to the lab, logged in, client information checked then analyzed. Recommended holding time for Dissolved gas compounds in VOA vials is 14 days.

XIII. PROCEDURE - SAMPLE PREPARATION AND ANALYSIS

A. After a calibration has been run, a continuing calibration check is run along with a method blank. The CCV must be 30%; the blank must not have targets above the MDL.

B. Air Phase Matrix - The can is attached to the inlet of the Perkin-Elmer GC the vacuum pump is turned on, then the canister valve is opened. Sample is allowed to flow long enough to fill the 2ml injection loop,

about 10 seconds. The pump is shut off and the valve closed as quickly as possible.

C. For Aqueous Matrix – Samples should be in an unpreserved 40ml amber vial with no headspace. A syringe is inserted through the septa on the vial and 10mls of water are extracted without allowing ambient air into the vial creating a headspace vacuum. The vial is then allowed to equilibrate to room temperature for 2 hours. The sample is then attached to the inlet of the Perkin-Elmer GC via a specially prepared airtight syringe. The vacuum pump is turned on for 10 seconds to fill the 5ml loop then the pump is turned off.

D. Once the rotometer has returned to zero, the sample run is started via the start button on the GC interface screen.

E. After the sample has run (15minutes), the sample data is opened and compared against the initial calibration to obtain the PPMv amounts for each compound of interest.

F. All sample sequences must include a duplicate sample with an RPD $\leq$30%.

G. After all samples have run, a closing continuing calibration check must be analyzed with $\leq$30% recovery.

H. LCS must pass with 30% RPD.

XIV. TROUBLESHOOTING

A. Check the entire system for leakage.

XV. DATA ACQUISITION, CALCULATIONS AND DATA REDUCTION

A. Data from Air phase samples are reported directly from the data results.

B. For dissolved gasses from an aqueous matrix, results must be multiplied by factors based on the equation from Henry's Law. The raw results are multiplied by the following conversion factors specific to each individual compound :

	a (concentration on column)	H (Henry's constant at 25C)	MW (molecular weight)	Temp (C)	MD (Molar Density= (MW * 273) / (22.4 * T))	Headspace	Volume of sample	B=a * MD * Head Space (vol.) / Sample (vol.)	A=a * 55.504 * MW / H	TC (ppb)
CH4	0.000001	39769.000	16	25	0.654	10.000	0.032	0.0002045	0.0000223	0.227
CO2	0.000001	1543.000	44	23	1.812	10.000	0.030	0.0006039	0.0015827	2.187
H2	0.000001	70719.000	2	25	0.082	10.000	0.032	0.0000256	0.0000016	0.027
O2	0.000001	43414.000	32	25	1.309	10.000	0.032	0.0004090	0.0000409	0.450
Ethane	0.000001	29771.000	30	25	1.227	10.000	0.032	0.0003834	0.0000559	0.439
Ethene	0.000001	11616.000	28	25	1.145	10.000	0.032	0.0003579	0.0001338	0.492
N2	0.000001	95411	28	25	1.145	10	0.032	0.0003579	0.0000163	0.374

XVI. COMPUTER HARDWARE AND SOFTWARE

A. Computer – Hewlett Packard PC
B. Software – Windows 2000, Perkin-Elmer TotalChrom Version 6.2.1

XVII. DATA MANAGEMENT AND RECORDS

A. Data is recorded onto the C: drive of HP3. It is then copied to the appropriate folder on the G: drive for permanent backup with all other data.
B. All sequences are copied and backed up with the data files.

XVIII. QUALITY ASSURANCE AND QUALITY CONTROL

A. All samples must be run under a passing calibration curve of 30%RSD or >0.99 linear regression. If an opening CCC does not pass, the instrument must be re-calibrated.
B. All sequences must include a blank with no targets above the MDL after the CCC, a duplicate run that falls within 30%RPD, an LCS at ≤30% recovery, and a closing CCC after all samples have run that passes less than 30%RSD. If the blank does not pass, it must be rerun. If the duplicate does not pass at ≤30%RSD, it must be rerun. If the closing CCC does not pass, the instrument needs to be recalibrated.
C. If a sample is over calibration range, it must be diluted and rerun.
D. All samples must be run within a 24-hour period after the opening CCC.

XIX. METHOD PERFORMANCE

A. Method performance is maintained through yearly MDL and P&A studies. MDL are run at a concentration 3-5 times the reporting limit or specified by the method. The calculated MDL must be greater than 10% of the standard used. If the MDL is less than 10% then the MDL study must be repeated with a low concentration. The average recovery for the MDL must be within 50-150% of the true value and must be less than the MRL. P&As must fall within 70% - 130%.

XX. POLLUTION PREVENTION AND WASTE MANAGEMENT

A. The Summa canisters are cleaned after analysis has occurred.
B. Vials are stored and disposed of in accordance with Spectrum Analyticals Chemical Hygiene Plan.

XXI. REFERENCES

A. EPA Method 3C

XXII. REVISIONS

Revision 1

A. Added reference to SOPRSK-175 to SOP title to include dissolved gases.
B. Added a section XII.C. describing the collection of aqueous samples for dissolved gases.
C. Updated sample calculation section XV.B.
D. Updated QC/QA section XVIII A & B.

Total Organic Carbon (TOC)

By Methods SW-846 9060A and/or SM5310B

Contents SOP NO. 100.0025

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		6/18/10
Quality Assurance Director		6/16/10
Operations/Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date
	QAD	7/5/11			

Revision Record

Revision Date	Revision Description	Comments	Initials
8/14/02	Added control page and renamed SOP	Was SOP WC00025B	
7/20/07	Added additional method, fixed revision number.	Equivalent	SBL
7/23/08	Lab name change		SBL
11/7/08	Added SW9060, removed E415.1	Full Rev	SBL
3/23/09 06/16/10	Dissolved TOC added, Revised to 9060A	Full 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

Total Organic Carbon

by

By Methods SW-846 9060A and/or SM5310B

SOP No. 100.0025 Rev. 8

	Signature	Date
QA Director:	Shawn B Fawler	6/16/10
Lab Director:		6/18/10
Effective Date:	6/23/10	

MITKEM LABORATORIES
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Total Organic Carbon

by

By Methods SW-846 9060A and/or SM5310B

SOP 100.0025 Rev. 8

1. Scope and Application

This Standard Operating Procedure (SOP) covers the procedure of determining the concentration of Total Organic Carbon in an aqueous sample (drinking, surface, and saline waters, domestic and industrial wastes). Total Organic Carbon includes all of the carbon in a sample, except carbon dioxide and its dissociation products.

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 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

Organic carbon in a sample is converted to CO_2 by catalytic combustion or chemical oxidation. An infrared detector then measures the CO_2 formed. An aqueous sample is introduced to a 680°C furnace in an oxygen rich environment. The water is then evaporated and removed from the system, and the total carbon (TC) content is oxidized to form carbon dioxide (CO_2). The CO_2 is then swept quantitatively to the infrared cell and is selectively detected. The total organic carbon (TOC) is determined as the difference between separate TC and TIC analyses, resulting in a true TOC value. Samples need not be filtered prior to analysis unless dissolved organic carbon (DOC) results are required. The combustion method allows quantitative carbon oxidation in the particulate so that an accurate value is reported.

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are to be preserved with acid if they can not be analyzed within 2 hours after sampling. Mitkem uses Phosphoric acid (H_3PO_4), however Hydrochloric acid (HCL) preserved samples can be used when necessary.

Method SW-846 9060A lists either hydrochloric (HCL) or Sulfuric (H_2SO_4) acids. See **section 5.5**.
Method SM5310B lists Phosphoric (H_3PO_4) or Sulfuric (H_2SO_4) acids.

4.2 Acidified samples have a holding time of 28 days from date collected.

4.3 Samples are to be refrigerated at 4°C.

4.4 Samples are stored in glass bottles.

5. Interferences and Potential Problems

5.1 The potential of leaks can be a problem. To avoid such a problem periodic leak checks should be performed on supply lines, fittings, and pneumatic plumbing.

5.2 It is important to follow good laboratory practices when making standards and preparing glassware as water, glassware, and reagents cannot be completely cleaned of carbon.

5.3 Water may interfere with the response of the detector. It is important to maintain a water free environment to the detector. This is accomplished by running the carbon dioxide stream through a drying tube before it reaches the NDIR detector. The tube should be replaced periodically, and gas flow must be maintained through the instrument.

5.4 Standards, gases, and water must be of the highest purity.

5.5 Tekmar Dohrman recommends an SO_3 mist scrubber for the Apollo 9000 when analyzing samples preserved with Sulfuric (H_2SO_4) acid. HCL or H_3PO_4 are the preferred preservatives, with H_3PO_4 being the better option.

5.6 Sample foaming can be an issue and may invalidate one or more of the quadruplicate analyses.

6. Equipment and Apparatus

6.1 Tekmar Dohrman Model Apollo 9000 Total Organic Carbon Analyzer with auto-sampler.

6.2 Volumetric flasks (25, 50, and 1000ml).

6.3 Fixed and adjustable pipettes.

6.4 DI water apparatus.

6.5 Vials.

6.6 0.45um-pore-diameter filters

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 Deionized water (should contain as little carbon as possible).

7.2 Oxygen gas (Must be free of carbon dioxide and carbon species)

7.3 Argon gas

7.4 5% (v/v) Phosphoric Acid: Used to treat unpreserved samples. (**section 8.3**)
Fisher ACS trace metals grade:
Prepared by adding 59mL of Reagent Grade (85%) H_3PO_4 to DI H_2O for a final volume of 1000mL.

7.5 5% (v/v) Hydrochloric Acid, Used to treat unpreserved samples. (**section 8.3**)
Fisher, ACS trace metals grade:
Prepared by adding 50mL conc. HCl to DI H_2O for a final volume of 1000mL.

7.5. Calibration Standard @ 1000mg/L, purchased commercially from Environmental Resource Associates as a custom prepared standard.

7.6. QC Check (CCV) Standard: LabChem Carbon Standard, 1000ppm(organic) – Catalog number LC12910-7:
Prepped by adding 2ml of standard into 100 ml of DI H_2O for a concentration of 20ppm.

7.7. Sodium Hydroxide (NaOH), Fisher, ACS certified.

8. Procedure

8.1 Instrument should remain on at all times although when not in use, it may be in standby mode. Set temperature to 680 °C

8.2 Fill humidifier vessel ¾ of the way with DI H_2O and add one NaOH pellet. The DI water should be replaced each day of TOC analysis.

8.3 Check acid supply is sufficient for sample load, *if necessary*. If not used, remove acid source.

8.4 Gas supply should be turned on at 50-60 psi by the tank regulator and 12mL/min by the regulator located inside the analyzer.

8.5 Once weekly, data files must be updated in the data system. Refer to the Users Manual for the Tekmar Dohrman Apollo 9000 for details.

8.6 Refer to the Users Manual for the Tekmar Dohrman Apollo 9000 for details on setting up the sequence in the data system.

8.7 Remove plastic cover; put the samples in the appropriately numbered cups according to the sequence in the logbook. Do not shake samples. Be sure to fill the wash cup with fresh DI water. Replace tray and plastic cover.

8.7.1 If dissolved organic carbon is to be determined, filter sample through a 0.45um-pore-diameter filter (recommended per Tekmar Dohrman) and analyze. Also analyze a filtering blank

8.8 Four replicates are analyzed per sample. Method 9060A recommends that both the average and the range are reported. Mitkem reports the average only, unless specifically requested otherwise.

8.9 Remove and empty the humidifier bottle. Reattach the bottle.

8.10. All TOC information is documented in the TOC1 Aqueous Analysis Logbook.

9. Data Reduction and Calculations

Data is recorded as TOC in mg/L. The software calculates the mean, standard deviation and RSD of the quadruplicate analyses.

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 a method blank, lab control sample, standard checks, sample duplicates and spikes, balance checks and pipette calibrations.

10.1 All samples must be verified of acid preservation ***before analysis***. Note the pH in the appropriate column of the TOC Analysis logbook.

10.2 A duplicate sample (DUP) is performed every 20 samples or every analytical run, whichever occurs more frequently.

10.3 The recovery of the standard check sample must be within 15% of the true value. The standard check sample is run at the beginning, end, and a minimum of every 10 samples.

10.4 The Method Blank (MB) should not exceed the reporting limit (PQL), although an MB may be deemed acceptable if all samples in the associated batch are at least 10 times higher than the level determined in the MB.

10.5 The LCS standard is from an independent source with recovery limits of 80-120%.

10.6 Duplicate sample results must fall within 20% or the analysis is repeated.

10.7 Spiked sample recoveries must be 75-125% or the spiked sample analysis is repeated once.

10.8 A new calibration curve is generated at least quarterly.

10.9 The Inorganic Lab Supervisor may approve deviations on a case by case basis, with technical justification.

11. Data Validation and Reporting

11.1 The standard preparation and instrument run logbooks are reviewed and signed by the Supervisor. The Supervisor reviews 100% of the data before it is reported.

11.2 The reporting group generates reports.

11.3 Concentrations are reported down to the PQL. The PQL is set as the lowest point of the calibration curve.

12. Corrective Action Procedures

12.1 If the LCS is outside the control limits, it is re-prepared and rerun. If it still fails a new calibration curve is established.

12.2 If the spike recoveries are greater or less than 25% the spiked sample is to be re-run once.

12.3 Duplicates are re-analyzed once if the RPD is greater than 20% and sufficient sample remains.

12.4 If the concentration of TOC in the Method Blank is greater than the PQL, it is re-analyzed. If there is still contamination, the source must be investigated before sample analyses.

12.5 If the check standard is outside the 15% window, it is re-prepared and re-analyzed once. If it is still out, a new curve is generated.

12.6 Samples are re-analyzed if more than 10 field samples are analyzed between check standards.

12.7 RSD values for quadruplicate analyses will be high when foaming or incomplete sampling due to low volume occurs. When foaming or incomplete sampling affects one or more of the quadruplicate analyses, the average of the remaining analyses may be used.

13. Health and Safety

Precautions to protect analysts include the nature of toxicity or carcinogenicity of analytes of reagents used in the method. Wherever possible, work under controlled conditions. Always wear a lab coat, gloves, and safety glasses at all times in the lab. Verify that all heated areas have cooled prior to handling, or wear adequate hand protection to prevent burns.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

USEPA SW-846 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Final Update IIIB, Method 9060A, Revision 1, November 2004.

Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992, Total Organic Carbon Method 5310B, Combustion Infrared Method.

Tekmar Dohrman Apollo 9000 User Manual

MITKEM LABORATORIES
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

For

Total Phosphorus and Orthophosphate Analysis for Aqueous samples by Standard Method 4500-P B (5) &E, Ascorbic Acid Method

SOP No. 100.0013

Rev. 9

	Signature	Date
QA Director:	Shawn B Lamb	10/15/09
Technical Director:		10/16/09

MITKEM LABORATORIES
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Total Phosphorus and Orthophosphate Analysis for Aqueous Samples by Standard Method 4500-P B (5) & E, Ascorbic Acid Method

SOP 100.0013 Rev. 9

1. Scope and Application

Phosphorus occurs in natural waters and waste waters almost solely as phosphates. These are classified as Orthophosphates, condensed phosphates (pyro, meta, and other phosphates), and organically bound phosphates. They occur in solution, in particles or detritus, or in the bodies of aquatic organisms. Phosphates arise from several sources; detergents, fertilizers, biological processes, body wastes, food residues, and water supply treatment. Phosphorus is essential to the growth of organisms and can be the nutrient that limits the productivity of a body of water.

Phosphates that respond to colorimetric tests without preliminary hydrolysis or oxidative digestion of the sample are termed "reactive phosphorus". While reactive phosphorus is largely a measure of orthophosphate, a small fraction of any condensed phosphate present usually hydrolyzed unavoidably in the procedure. Reactive phosphorus occurs in both dissolved and suspended forms. Total Phosphate is the sum of the hydrolyzed and non-hydrolyzed phosphate concentrations.

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 their work tasks following the procedures outlined in this SOP. Supervisors and managers are responsible for reviewing and approving new and revised SOPs. They are responsible for ensuring that SOPs are accurate, up to date, and are being implemented appropriately.

3. Summary of Procedure

Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid.

Organic phosphorus compounds may be converted to the orthophosphate form by persulfate digestion. Only orthophosphate forms a blue color in this test. The Phos Ver 3 Phosphate Reagent Powder Pillows (Ascorbic Acid included) are used for the combined reaction reagent as a modification to the original SM4500 P method. The concentration of the Molybdenum Blue is directly proportional to the concentration of orthophosphate.

4. Sample Preservation, Containers, Handling, and Storage

Samples are collected in 250mL HDPE containers. If phosphorus forms are to be differentiated, filter sample immediately after collection. Unpreserved Orthophosphate samples are to be refrigerated upon receipt and analyzed within 48 hours. If the samples are to be analyzed for total phophorus only, preserve with H_2SO_4 to pH < 2 and analyze within 28 days.

5. Interferences and Potential Problems

Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. Concentrations as low as 0.1mg As/L interfere with the phosphate determination. Hexavalent chromium and NO_2^- interfere to give results about 3% low at concentrations of 1mg/L and 10 to 15% low at 10mg/L. Sulfide (Na_2S) and silicate do not interfere at concentrations of 1.0 and 10mg/L.

6. Equipment and Apparatus

All glassware must be rinsed with 1:1 HCl and DI Water.

6.1 Spectrophotometer: Spectronic 20 Genesys, Model 4001/4, with infrared phototube for use at 880nm, providing a light path of 2.5cm or longer.

6.2 25 mL mixing cylinders.

6.3 Adapter 2.5mm tubes for SpecGenesys

6.4 Hot plate.

6.5 Glass scoop to hold the required amounts of persulfate crystals.

6.6 125 mL beakers for digestion procedure.

6.7 Watch glasses.

6.8 50 mL graduated cylinder.

6.9 Calibrated Wheaton-type pipettes.

7 Reagents

7.1 Phenolphthalein indicator: aqueous solution.

7.2 Sulfuric Acid (H_2SO_4) solution: **carefully** add 300mL conc Trace Metals H_2SO_4 to 600mL DI water and dilute to 1L with DI water.

7.3 Potassium Persulfate ($K_2S_2O_8$), solid, Fisher Scientific (P282).

7.4 Sodium Hydroxide (NaOH) solution:1N: 40g to 600mL of DI water, dilute to 1 liter of DI water.

7.5 Hach Phos Ver 3 Phosphate Reagent Powder Pillows with Ascorbic Acid included.

7.6 Phosphate-free DI water.

7.7 1:1 HCl, Trace Metals for cleaning glassware.

7.8 Stock Phosphate standard at 50mg/L.

7.9 Standard Phosphate solution (2.5mg/L): dilute 50.0 mL stock phosphate standard to 1000 mL with DI water; 1.00 mL = 2.50µg P.

7. Procedure

8.1 Persulfate Digestion for total phosphorus samples only:

8.1.1 Use 50 mL or a suitable portion of thoroughly mixed sample. Add 1 drop (0.05mL) Phenolphthalein indicator solution. If a red color develops, add Sulfuric Acid solution dropwise to just discharge the color. Then add 1.0 mL of the Sulfuric Acid solution and 0.5g $K_2S_2O_8$.

8.1.2 Boil gently on a preheated hot plate for 30 to 40 minutes or until a final volume of 10mL is reached. **Do not allow the sample to go to dryness**. Organophosphorus compounds such as AMP (Adenosine-3', 5'-cyclic-monophosphate) may require as much as 1.5 to 2 hours for complete digestion.

8.1.3 Cool, dilute to about 30 mL with DI water. To read the pH add 0.05mL (1 drop) Phenolphthalein indicator solution, and neutralize to a faint pink color with NaOH solution (pH should be 7.0 ± 0.2). Volumize to 50mL with DI water. In some samples a precipitate may form at this stage, but do not filter. For any subsequent subdividing of the sample, **shake well**. The precipitate (which is possibly a Calcium Phosphate) re-dissolves under the acid conditions of the colorimetric reactive phosphorus test.

8.1.4 Determine phosphorus by the Ascorbic Acid Method described below. All digestion information is documented in the Phosphorus Preparation Logbook **(figure 1)**. Standards require digestion too.

8.2 Ascorbic Acid Colorimetric Method:

8.2.1 Calibrate the SpecGenesys with a series of six individual standards. The standards for both Orthophosphate and Total phophorus are 0.00, 0.05, 0.10, 0.20, 0.40, and 0.80mg/L.Use a DI Water blank with the pillow mix as a reagent blank. The lowest non-zero standard must be at a concentration ≤ MRL.

8.2.2 Fill a mixing cylinder with 25mL of sample for the Orthophosphate analysis and 25mL of persulfate digestate for the total phophorus analysis. Check the pH and adjust with 1N NaOH to a pH of 7.0 ± 0.2 *if necessary*.

8.2.3 Add the contents of one PhosVer 3 pillow to the sample and mix thoroughly.

8.2.4 After at least 10 minutes but not more than 30 minutes, measure the absorbance of each sample at 880nm, using the reagent blank as the reference solution.

8.2.5 If samples are turbid, set the spectrophotometer at 0.000 absorbance with the sample blank. Then analyze the sample (with no pillow reagent). Re zero the spectrophotometer with the reagent blank before analyzing the next sample.

8.2.6 All information is documented in the Phosphate Analysis Logbook (**figure 2**). A copy of the logbook page is sent to Data Reporting for data uploading into the Omega LIMS System.

9. Data Reduction and Calculations

9.1 Full scale is determined at the beginning of the run.

9.2 Absorbance is read directly from the SpecGenesys.

9.3 The concentration of total phosphorus or orthophosphate is calculated as follows: Conc = (Abs-b)/m in which b = the y-intercept of the curve and m = the slope of the line.

9.4 The results are expressed as total phophorus in mg/L PO_4. If concentration is to be reported as mg/L P, then divide the concentration as PO_4 by 3.

9.5 The Minimum Reporting Limit (MRL) is 0.1mg/L PO_4 for total phophorus and 0.05mg/L PO_4 for orthophosphate.

10. Quality Assurance/Quality Control

10.1 Every digestion and/or analytical batch must include a Prep Blank (Phosphate-free DI Water) and a Laboratory Control Sample (LCS) at approximately the mid-point of the curve. The LCS is a second source standard with limits that are dictated by the current control limits for the analysis. The LCS recovery limit is 80-120%.

10.2 Duplicates and Matrix Spikes are analyzed at a frequency of 1 per 20 samples, or one set per preparatory batch. The RPD between the sample and duplicate should be less than or equal to 20%; deviations need to be reported in the project narrative. The control limits for the matrix spike is 75-125% unless sample concentration exceeds the spike added by a factor or 4 or greater. If the matrix spike fails the method criteria, a post-digestion spike is performed and reported.

10.3 The Correlation Coefficient for the standard curve must be ≥ 0.995. The calibration blank is not included in calculating the correlation coefficient of the curve.

10.4 Total phophorus in the Prep Blank for each batch must not exceed the MRL.

10.5 An Independent Check Standard is run at the beginning, end, and at least after every 10 samples in an analysis sequence. The standard is of a lot number or source independent of the calibration curve, with acceptance criteria of 90-110%. The concentration of the check standard must vary over the range of the calibration curve if the curve was not established on the day of analysis.

10.6 All standards made from a primary standard expire on or before the primary standard's expiration date.

11. Data Validation and Reporting

The analyst is responsible for checking all data, preparations, calculations, results, QC criteria, and logbook entries for accuracy and completeness. The department supervisor is responsible for reviewing all logbooks and initialing/dating weekly. The QA Director is responsible for reviewing logbooks for correctness and completeness at unscheduled intervals. A copy of the generated data will then go to Data Reporting to be entered, copied, and reported to the client.

12. Corrective Action Procedures

All out-of-control situations must immediately be reported to the department supervisor or the QA Director. Out-of-control situations must be recorded in the LIMS Corrective Action Logbook and documented the situation returned to control as described in the Corrective Action Procedures SOP No. 80.0007.

13. Health and Safety

All employees should read and be familiar with Mitkem's Safety Manual and Chemical Hygiene Plan, and comply with the safety practices described therein. The toxicity or carcinogenic properties of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achieved. Consult the MSDS sheets for specific information on all chemicals used.

14. Pollution Prevention, Waste Management, Acronyms and Definitions

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992, Method 4500-P B (5) & E, Ascorbic Acid Colorimetric Method.

Attachments:
Figure 1: Phosphorus Preparation Logbook
Figure 2: Phosphate Analysis Logbook

Figure 1:
Phosphorus Preparation Logbook

MITKEM LABORATORIES **Phosphorus Persulfate Digestion Logbook**

METHOD: SM4500-P, B(5)

Time started:

Time ended:

Date:		Sample Volume (ml)/(gram)	Initial Sample pH	H2SO4 added (ml)	K2S2O8 added (gram)	NaOH added? Y/N	Final Volume (ml)	Final Sample pH	Pillow added? Y/N	Analyst:
Beaker #	Lab ID									Comments

H2SO4 Lot No.: _____

K2S2O8 Lot No.: _____

NaOH Lot No.: _____

Logbook ID: 100.0096-05/09

Reviewed By: _____

Figure 2:
Phosphate Analysis Logbook

MITKEM LABORATORIES — Orthophosphate and Total Phosphate Analysis Logbook

Date	Sample ID		Sample Volume (ml)	Calc. ABSORB	Calc. CONC (mg/l)	Dilution Factor	Result (mg/L)	% Recovery	Comments	Analyst

Wavelength=_____ LCS:_____ m=_____

Pathlength=_____ true value=_____ b=_____

MRL=_____ r=_____

Conc of Analyte = (Abs-b)/m Curve Date: _____

Logbook ID: 100.0095-01/08 Reviewed By: _____

NY Lab #11393/11840
FL Lab #E87600/E87936

SPECTRUM ANALYTICAL INC.
Featuring
Hanibal Technology
11 Almgren Drive
Agawam, MA 01001

Standard Operating Procedure
For
Nitrate/Nitrite-N by Flow Injection Analysis

Lachat QuikChem Method 10-107-04-1-B
Nitrate via EPA 353.2
Nitrite via EPA 353.2

Prepared by — Date 2/1/11

Reviewed by — Date 2/1/11

Laboratory Director — Effective Date 2/1/11

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STANDARD OPERATING PROCEDURE
for
Nitrate/Nitrite-N by Flow Injection Analysis
Lachat QuikChem Method 10-107-04-1-B
Nitrate via EPA 353.2
Nitrite via EPA 353.2

I. SCOPE AND APPLICATION

A. This purpose of this method is to determine nitrate/nitrite in surface water, and industrial wastewater.

B. The applicable range is 0.01 to 1.0 mg N/L. The method throughput is 80 injection per hour.

C. Users of the method must demonstrate the ability to generate acceptable results, using the procedures described in this method.

II. SUMMARY OF METHOD

A. Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting water soluble dye has a magenta color which is read at 520 nm. Nitrite alone also can be determined by removing the cadmium column.

III. HEALTH AND SAFETY

To maintain the application of OSHA regulations regarding the safe handling to the chemicals specified in this method, the laboratory must follow proper safety procedures:

A. All chemicals should be transported on a cart when moved from room to room.

B. Safety glasses, gloves and protective clothing must be worn when performing any phase of the analysis.

C. The analyst must dispose of all unwanted chemicals and acids in properly marked containers inside the hood and chemical cabinets. (See Spectrum's waste disposal plan.)

D. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. The following chemicals have the potential to be highly toxic or hazardous.

1. Cadmium

2. Ammonium hydroxide

3. Sodium hydroxide
4. Phosphoric acid

5. Sulfanilamide

IV. INTERFERENCES

A. Residual chlorine can interfere by oxidizing the cadmium column.

B. Low results would be obtained for samples that contain high concentrations of iron, copper or other metals. In this method, EDTA is added to the buffer to reduce this interference.

C. Samples that contain large concentration of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with organic solvent.

D. Sample turbidity may interfere. Turbidity can by removed by filtration through a 0.45 μm pore diameter membrane filter prior to analysis.

V. REAGENTS

A. Reagent water: Distilled or deionized water, preferably with a reading of 18.2 Meg-ohm or better and containing particles no larger than 0.20 microns.

B. 15 N Sodium Hydroxide

1. Add 150g NaOH very slowly to 250 mL of water. Swirl until dissolved. Cool and
store in a plastic bottle.

C. Ammonium Chloride buffer, pH 8.5

1. In a 1L volumetric flask, dissolve 85g ammonium chloride (NH_4CL) and 1g disodium ethylenediamine tetraacetic acid dihydrate ($Na_2EDTA{\cdot}2H_20$) in about 800 mL water. Diluted to the mark and invert to mix. Adjust the pH to 8.5 with 15 N sodium hydroxide solution.

OR:

2. In a hood, to a 1L volumetric flask, add 500 mL water, 105 mL concentrated hydrochloric acid (HCL), 95 mL ammonium hydroxide (NH_4OH) and 1g disodium EDTA. Dissolve and dilute to the mark. Invert to mix. Adjust the pH to 8.5 with HCL or 15N NaOH.

D. Sulfanilamide color reagent

1. To a 1L volumetric flask, add about 600 mL water. Then add 100 mL of 85% phosphoric acid (H_3PO_4), 40g sulfanilamide, and 1g N-(1-naphthyl)ethylenediamine (NED). Shake to wet, and stir for 30 min. to dissolve. Dilute to the mark, and invert to mix. Store in dark bottle. This solution is stable for the one month.

E. Preparation of standards

Following are standards preparations for a 1 channel system determining NO_2+NO_3 or NO_2 and a 2 channel system where one channel is used for NO_2+NO_3 and the other channel is used for determining NO_2. For the 1 channel system, either NO_2 or NO_3 standards may be used. It is recommend the use of NO_3 standards when running a 1 channel method for NO_2+NO_3. For the 2 channel system, it is recommend the use of both NO_2+NO_3 standard sets.

1. Standard 1. Stock Nitrate Standard 100 mg N/L as NO_3
In a 1L volumetric flask, dissolve 0.7218g potassium nitrate (KNO_3) in about 600 mL water. Dilute to the mark and invert to mix. This solution is stable for six months.

2. Standard 2. Stock Nitrite Standard 100 mg N/L as NO_2
In a 1L volumetric flask, dissolve 0.4928g sodium nitrite ($NaNO_2$) or 0.6076g potassium nitrite (KNO_2) in approximately 800 mL water. Dilute to the mark and invert to mix. Refrigerate. This solution is stable for 3-5 days.

3. Standard 3. Working Stock Nitrate Standard 1 mg N/L as NO_3
In a 1L volumetric flask, add 10 mL of Stock Nitrate Standard 1, 100 mg N/L. Dilute to the mark and invert to mix. This solution is stable for one week.

4. Standard 4. Working Stock Nitrite Standard 1 mg N/L as NO_2
In a 1L volumetric flask, add 10 mL of Stock Nitrite Standard 2, 100 mg N/L. Dilute to the mark and invert to mix. This solution is stable for 24 hours.

NITRATE STANDARDS

WORKING STANDARDS (PREPARE DAILY)	A	B	C	D	E	F	G
CONCENTRATION MG N/L AS NO_3	1.0	0.5	0.1	0.05	0.01	0.00	0.000
VOLUME (mL) OF WORKING STANDARD 3 DILUTED TO 100Ml H_20	100	50	10	5	1	0.0	0.0

NITRITE STANDARDS

WORKING STANDARDS	A	B	C	D	E	F	G
CONCENTRATION MG N/L AS NO_2	1.0	0.5	0.1	0.05	0.01	0.00	0.000
VOLUME (mL) OF WORKING STANDARD 4 DILUTED TO 100Ml H_20	100	50	10	5	1	0	0.0

VI. APPARATUS AND MATERIALS

A. The Lachat QuikChem FIA+, Model 8000 Series, analyzer is modular in design, and consists of the following modules:

1. The dilutor performs dilutions from 1/1.6 to 1/1000.
2. The XYZ sampler with an automatic arm to sample and dilute standards and samples.
3. Regent pump is used to pump reagents onto the manifold and also aspirates sample portions from the sampler to the valve.
4. QuikChem FIA+ System Unit consists of an injection valve, a manifold, a cadmium-copper reduction column, a photometric detector, and a heating module.
5. Computer
6. Printer

B. Disposable culture tubes 13×100 mm.

C. Clear vials 40 mL.

VII. CALIBRATION

A. Prepare reagent and calibration standards as described in Section V.

B. Set up manifold and input data system parameters as shown in Section IX.

C. Pump DI water through all reagent lines and check for the leak and smooth flow. Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.

D. Place standards in the sampler. Input the information required by the data system.

E. Calibrate the instrument by injecting the standards. The data system will then associate the concentrations with the peak area for each standard to determine the calibration curve.

F. The calibration curve must be verified on each working day, and after every 10 samples. If the expected value for any analyte varies by more than +/- 10%, the test must be repeated, using fresh calibration standards. If the results are still more than +/- 10%, a new calibration curve must be prepared and run.

VIII. SAMPLE COLLECTION, PRESERVATION AND HANDLING

A. Samples should be collected in plastic or glass bottles. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis, and minimize waste disposal.

B. Samples to be analyzed for nitrate or nitrite only should be cooled to 4^0 C and analyzed within 48 hours.

C. Samples must be preserved with H_2SO_4 to a pH <2 and cooled to 4^0C at the time of collection if analyzed for nitrate/nirite combined.

D. Samples should be analyzed as soon as possible after collection. Nitrite will be oxidized by air O_2 to nitrate in a few days. If storage is required, preserved samples are maintained at 4^0C and may be held for up to 28 days.

E. If build-up of suspended matter in the reduction column restricts sample flow, the samples may be profiteered.

IX. PROCEDURE

A. Turn on the computer and log into Omnion FIA and click on Flow Injection Analysis. To open a method, click on File, Open Method and choose "lowno2.met: for nitrite or "lowno3.met" for nitrate. An Analyte Table appear on the screen where is specify the concentration units, standard concentrations. A method consists of the parameters such as valve timing, sampler timing, pump timing, which are given in the QuikChem method.

1. Data system parameters:

Pump speed:	35
Cycle period:	45

2. Analyte data:

Concentration units:	mg N/L
Peak base width:	18s
% width tolerance	100
Threshold:	17000
Inject to peak start:	10s
Chemistry:	Direct

3. Calibration data:

LEVEL	1	2	3	4	5	6	7
CONCENTRATION mg N/L	0.1	0.05	0.025	0.01	0.005	0.002	0.000

Calibration rep. Handling:	Average
Calibration fit type:	1st order polynomial
Weighting method:	None
Force through zero:	No

4. Sampler timing:

Min probe in wash period:	9s
Probe in sample period:	20s
Valve timing:	
Load time:	0s
Load period:	10s
Inject period:	35s

B. To open a tray, click on File, Open Tray and choose "nitrite.tra" for nitrite or "nitrate.tra" for nitrate. Type in the calibration standards and sample identification. Specify the number of replicates and the levels.

C. DQM (data quality management) is associated with the tray. DQM allows scheduling a quality control in the tray, or may be empty.

D. Set up manifold as shown in diagram:

E. Instrumentation must be turned on and pumped DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow the system to equilibrate until stable baseline achieved.

F. Place samples and standards in the autosampler and click Run Sample. Blank, QC standard, duplicates and spikes are required in each batch. Spike and duplicate must be made after every 10 samples.

G. If the pH of the sample is below 5 or above 9, adjust to between 5 and 9 with either conc. HCL or conc. NH_4OH.

H. The calibration curve must be verified on each working day. If the result for any analyte varies by more than +/- 10%, the test must be repeated. If the results are still more than +/- 10%, a new calibration curve must be prepared for that analyte.

I. If the response for a sample peak exceeds the working range of the system (the concentration is higher than the highest standard on the curve), dilute the sample with an appropriate volume of water or use dilutor and reanalyze.

J. Example of a run with QC

ICV
ICB
CCV
CCB
Blank
Blank Spike
SRM
Sample
DUP
MS
MSD
3 Samples
CCV
CCB
10 Samples
CCV
CCB

X. DATA CALCULATIONS AND REPORTING

A. Calibration is done by injecting standards. The data system will then prepare a calibration curve by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.

B. Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed

C. Report sample result for nitrate/nitrite in mg N/L as NO_3 or NO_2 to two significant figures for sample above the MDL. Report results below the MDL as less than the detection limit.

XI. QUALITY CONTROL

A. Lab Reagent Blank (LRB): At least one LRB must be analyzed with each batch of samples. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

B. Lab Fortified Blank (LFB): At least one LFB must be analyzed with each batch of samples. The accuracy must be calculated as percent recovery. If the recovery of any analyte falls outside the required control limits of 90-110%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

C. The matrix spike and matrix duplicate are required, every 10 samples, to demonstrate method accuracy and precision and monitoring matrix interferences.

D. Quality Control Sample is required with every analytical batch to verify the concentrations of the calibration standards. It is suggested that the quality control sample be obtained from outside source.

E. Duplicate samples are run at a frequency of > or = 5% or one per batch (if < 20 samples) analyzed to assess precision

Relative percent difference (RPD) = A -B/C X 100

Where: A = sample result
B = duplicate result
C = average of sample and duplicate results

F. The concentration of the spike in the sample shall be determined as follow:

1. If the concentration of the analyte in the sample is being check against a regulatory concentration limit, the spike level shall be at the limit or 1 to 5 times higher than the background concentration of the sample which ever is higher.

2. If the concentration of the analyte in a sample is not being checked against a limit, the spike shall be at the concentration 1 to 5 times higher than the background concentration.

XII. REVISIONS

Revision 3 01/28/11
Added section IX. J.

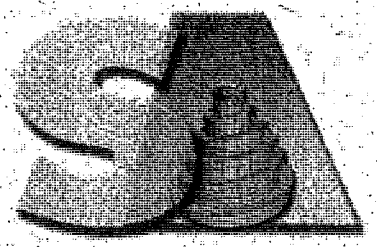
XIII. REFERENCES

A. Lachat Instruments, Determination of nitrate/nitrite in surface and wastewater by flow injection analysis, QuikChem Method 10-107-04-1-B.

B. Method 353.2; Determination of Nitrite-Nitrate Nitrogen by Automated Colorimetry; Methods for the Determination of Inorganic Substances in Environmental Samples; Rev 2.0, August, 1993.

ACCREDITED IN ACCORDANCE WITH NELAC

NY Lab #11393/11840
FL Lab # E87600/E87936



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SPECTRUM ANALYTICAL, INC.
Featuring
Hanibal Technology
11 Almgren Drive
Agawam, MA 01001

Standard Operating Procedure

for

Ammonia Nitrogen as N. (mg/L)
SM4500-NH_3-B,C, 20th Edition

Prepared by — Date: 11/14/06

Reviewed by — Date: 11/15/06

Lab Director — Date: 11/16/06

TABLE OF CONTENTS
STANDARD OPERATING PROCEDURE

Ammonia Nitrogen as N. (mg/L)

SM4500-NH_3-B.,E.

I SCOPE AND APPLICATION

This method determines the ammonia nitrogen in surface and wastewater, and also sludge and soil. The distillation and titration procedure is used for NH_3-N concentration greater than 5 mg/L.

II. SUMMARY OF METHOD

The sample is buffered at pH 9.5 with a borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds. It is distilled into a solution of boric acid. The ammonia in distillate is determined by titrimetrically method with standard H_2SO_4 and a mixed indicator.

III. INTERFERNCE

Turbidity, color, and substances precipitated by the hydroxyl ion, such as magnesium and calcium, interfere and may be removed by distillation or by precipitation with zinc sulfate and alkali. Glycine, urea, glutamic acid, cyanates, and acetamide volatile alkaline compounds also can cause interferences. Residual chlorine should also be removed before analysis.

IV. APPARATUS AND MATERIALS

A. Distillation apparatus.

B. Kjeldahl flasks – 800 mL.

C. Erlenmeyer flasks – 300 mL.

D. Various pipettes.

E. pH meter.

V. REAGENTS

A. Sodium Hydroxide (NaOH)

B. Sodium Tetraborate ($Na_2B_4O_7$)

C. Boric Acid (H_3BO_3)

D. Methyl Red Indicator

E. Methylene blue

F. Phenolphthalein

G. Ethyl or Isopropyl Alcohol

H. Anhydrous Ammonium Chloride

I. Stock Ammonia Solution: Dissolve 3.819g of anhydrous NH_4CL, dried at 100^0C, in water and dilute to 1000 mL; 1.00 mL = 1.00 mg N = 1.22 mg NH_3\

J. Standard Ammonia Solution: Dilute 10 mL of stock solution to 1000 mL with distilled water; 1.00 mL = 10.0 Ug N = 12.2 Ug NH_3

K. Borate Buffer Solution: Add 88 mL 0.1N NaOH solution to 9.5 g $Na_2B_4O_7 \times 10\ H_2O$/L and dilute to 1L.

L. Indicating Boric Acid Solution: This is the absorbent reagent. Dissolve 20.0g H_3BO_3 in distilled water. Add 10 ml mixed indicator solution (N). Dilute to one liter.

M. 0.02 N Sulfuric Acid Titrant: Prepare 1N H_2SO_4 solution by adding 28 mL of concentrated H_2SO_4 to distilled water and dilute to the 1L. Then, in a 1 liter volumetric flask, add 20 mL of the 1N H_2SO_4 and dilute to the mark. Standardize by potentiometric titration of 15mL of 0.05N Na_2CO_3.

Normality of H_2SO_4 = A×B / 53×C

Where: A – grams of Na_2CO_3 weighed in 1-liter flask.
B – mL of Na_2CO_3 solution used in titration.
C – mL of acid used

N. Mixed Indicator Solution: Dissolve 200mg of Methyl Red Indicator in 100mL of 95% Ethyl or Isopropyl Alcohol. Dissolve 100mg of Methyl Blue in 50mL of 95% Ethyl or Isopropyl Alcohol. Combine both solutions. Those should be prepared monthly.

VI. SAFETY

To maintain the application of OSHA regulations regarding the safe handling of the chemicals specified in this method, the laboratory must follow proper safety procedures:

A. All chemical solvents should be transported on a cart when moved from room to room.

B. Safety glasses, gloves and protective clothing must be worn.

VII. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

A. Sample should be collected using an appropriate sampling plan.

B. If samples are not to be analyzed within 24 hours, then preserve with H_2SO_4 to a pH< 2 and store at 4^0C for no longer than 28 days.

C. Samples with residual chlorine should be dechlorinated by using $Na_2S_2O_7$.

VIII. PROCEDURE

A. Sample Preparation: Place 200 mL of dechlorinated sample (Use 1 mL of Sodium Thiosulfate to remove 1 mg/L of Residual Chlorine in 500 mL of sample.) or portion of sample diluted to 500 mL, in to 800 mL Kjeldahl flask. Add 3 drops of alcohol phenolphthalein to each flask. Add 25 mL borate buffer and then add 6N NaOH dropwise until sample turns pink (if needed). Add 5 glass beads.

B. Distillation: Distill sample at rate 6 to 10 mL/min. collecting 150mL of distillate for water sample and 100 mL for solid and sludge. Collect distillate in a 300-mL erlenmeyer flask containing 25 mL indicating boric acid solution.

C. Titrate ammonia in distillate with standardized 0.02 N H_2SO_4. Color should change from a green to pale lavender.

IX. CALCULATION

A. For an Aqueous sample:

mg/L NH_3-N = {(A-B)×280} / mL of sample

B. For a Sludge/Solid sample:

mg/L = {(A-B)×280} / g dry weight sample

Where: A = volume of H_2SO_4 titrated for the sample, mL
B = volume of H_2SO_4 titrated for the sample, mL

X. QUALITY CONTROL

A. A reagent blank is analyzed with every set of samples to check any background concentration. A set of samples can include no more than 20 samples.

B. If an internal standard is analyzed in addition to an SRM sample, the recovery should be within 10%.

C. Duplicate and spiked samples are analyzed on at least once per batch or once every 20 samples. Matrix spike results should fall within +/- 20%.

D. An SRM sample is prepared and analyzed with every set of sample to check the accuracy of the method. Result for this sample should fall within manufacturer's limits.

E. If any set of samples has out-of-control data, the samples in question are re-analyzed. Under no circumstances will out-of-control data be used as a final result.

XI. METHOD DETECTION LIMIT

A. Spectrum is in full compliance with NELAC requirements, however, MDL studies will be performed on an annual basis in support of state and program requirements such as CAM, RCP, ASP, CLP-like deliverables and specific project quality assurance objectives.

B. To determine the MDL for each analyte, analyze a sample aliquot at 3-5X the detection limit or as specified by the method. The calculated MDL **must be greater** than 10% of the standard used. If the MDL is less than 10%, repeat the analysis using a smaller concentration. The ideal MDL will be slightly greater than 10% of the standard used.

C. The results of the MDL studies must be within 50-150% of true value.

XII. METHOD PERFORMANCE

Refer to Spectrum's Laboratory Information Management System (LIMS) for quality control charts.

XIII. POLLUTION PREVENTION

A. Never dispose of samples, reagents, chemicals, or waste waters by pouring them down the sink. Always use designated waste containers for disposal.

B. Plan accordingly to limit waste accumulation. Make only the amount of reagent that can be used before the expiration date. Do not make in excess.

C. Clients should provide a sufficient amount of the sample for the requested analysis. Excess amounts of the sample result in increased disposal fees for the laboratory.

XIV. WASTE MANAGEMENT

Spectrum Analytical is dedicated to implementing ways to efficiently utilize resources along with complying with all environmental laws and regulations in order to reduce the accumulation of waste as defined in Spectrum's Chemical Hygiene Plan. All questions and/or problems should be referred to the Health and Safety Manager.

A. Aqueous Wastes:

1. All **solvent contaminated** water must be collected in lab satellite-containers then transferred to a waste drum in the hazardous waste staging area where they are monitored and ultimately disposed of by a hazardous waste disposal facility.

2. All **non-solvent contaminated** aqueous wastes (including preserved water, digestates, instrument effluents, and corrosive aqueous wastes) are accumulated in lab satellite-containers and transferred to a drum in Hazardous Waste staging area #2 where they will be disposed by a licensed hazardous waste facility.

3. COD vials are disposed in a designated drum.

B. Solids:

1. Expired soil samples in the storage area are emptied into a drum and a sample is collected. The method of disposal will be determined by the findings of the sample profile.

2. Expired PCB containing samples (marked with yellow tape) are segregated and

collected in the waste staging area and packed for disposal by a licensed hazardous waste facility.

2. Objects containing high levels of mercury (samples, broken thermometers, etc.) are segregated and collected in the waste staging area and packed for disposal by a hazardous waste facility.

C. Sludge, Tars, Oils:

These samples are accumulated in the waste staging area and packed for disposal by a hazardous waste facility/transporter.

D. Highly contaminated objects (reagents, chemicals, vials, samples) are segregated and collected by each dept. to avoid mixing of incompatible materials. It is then collected, and packed periodically throughout the year by hazardous waste disposal facilities.

XV. REFERENCES

Standard Methods for the Examination of Water and Wastewater,20th Ed.1998, American Public Health Association, 1015 Fifteenth Street NW, Washington, DC 20005.
Method: 4500 – NH_3 B,C

XVI. GLOSSARY

mg/L	=	milligrams per liter
pH	=	potential of Hydrogen
mL	=	milliliters
mL/min.	=	milliliters per minute
°C	=	degrees Celsius

Sample Receipt, Storage, Tracking and Disposal

Contents SOP NO. 30.0003

Procedure Signatures

Title:	Signature	Date
Laboratory Director/Technical Director		7/27/11
Quality Assurance Director		7/26/11
Laboratory/Quality Designee		

Procedure Reviews

Signature	Title	Date	Signature	Title	Date

Revision Record

Revision Date	Revision Description	Comments	Initials
3/8/07	Added bullet to 8.1.9, Added R4 to 8.2.2.9	Document SDG Final sample on TR/COC, 2 refrigerators for SOM	SBL
5/10/07	Added signing of login record when storing samples	Included two additional attachments	SBL
5/10/07	Added information about VOA soil holding blanks	Per SOM audit	SBL
3/12/08	Add 24 hour waiting period after acid preservation of metals.	Per Federal Reg 3/26/07, Lab name change.	SBL
4/9/08	SOM SOW update, pH paper edit		ARN
10/16/08	Revising disposal section, added new preservative, edit DC-1	Link to SOP 30.0024	SBL
02/03/10	DoD updates	Full rev	SBL
05/20/10	Empty container storage added, separate lines in logbook if diff pres.	Minor rev	SBL
08/23/10	Commercial metals storage on M shelves now in Unit 3, ICOC	Full rev	SBL
12/23/10	Added use of R8	TOC/COD samples only, minor edit	SBL
7/25/11	Added more documents, pH excursion notification	SOP Audit by EPA, full revision	SBL
9/29/11	Update to ISM01.3, new generic DC-1 form	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

Sample Receipt, Storage, Tracking and Disposal

SOP No. 30.0003

Rev. 16

	Signature	Date
QA Director:		7/26/11
Lab Director:		7/27/11
Effective Date:	8/2/11	

SPECTRUM ANALYTICAL INC.

Featuring Hanibal Technology
Rhode Island Division

STANDARD OPERATING PROCEDURE

for

Sample Receipt, Storage, Tracking and Disposal

Rev. 16

1. Scope and Application

This Standard Operating Procedure describes the procedures that must be followed upon receipt of samples at Spectrum Analytical, Inc. RI Division. It describes the procedures to log-in and store samples. It also describes procedures for checking samples out of the storage area for analysis, and for final disposal of samples. Detailed procedures for entering work orders and samples into the Laboratory Information Management System (LIMS) are described in a separate SOP Number 20.0003.

2. Personnel Qualifications and Responsibilities

The Sample Custodian should have a two-year degree in environmental science or a related field or have six months of on-the-job experience working with a trained and qualified Sample Custodian.

3. Summary of Procedure

When sample coolers are received, cooler custody seals and temperature are checked. The chain-of-custody forms are signed and the condition of the samples is checked, including any sample custody seals. Each sample is assigned a specific individual sample number. Any discrepancies are documented and resolved. Samples are logged-in to the LIMS following procedures in SOP 20.0003. Labels are generated, and affixed to samples. The pH of aqueous samples (Inorganic only, never VOA) are checked and if need be, either acid or base is added. The addition of preservative (and its lot number) is recorded on the Condition Form. Samples are stored in secure area under documented custody conditions. Certain types of VOC soil samples require special handling, as described in **Section 8.2.2**. Samples are kept for a specific time as called for by contract with the client, and then disposed of in accordance with federal, state and local regulations. The door to the sample receiving room is locked at all times, using a keypad code lock.

4. Sample Preservation, Containers, Handling, and Storage

Samples received at Spectrum are collected by our clients. Spectrum does provide preservatives and sample containers pre-cleaned by bottle suppliers.

Tables 7-1, 7-2 and 7-3 of the lab's Quality Assurance Plan (QAP) lists the type of container needed for a particular type of analyte and the proper preservative.

5. Interferences and Potential Problems

Any problem with sample condition including custody seals, breakage, discrepancies in chain of custody or other shipping documentation, cooler temperature, missing samples, etc. must be communicated to the client promptly and resolved. The Sample Custodian or assistant documents the problem and notifies the Project Manager, who will then notify the client. For CLP the Project Manager will notify the Sample Management Office (SMO).

6. Equipment and Apparatus

6.1 NIST-calibrated temperature "gun".

6.2 Condition Forms: Non-CLP, CLP (DC-1).

6.3 Orange colored Sample Condition Notification forms.

6.4 Narrow range pH paper, capable of recording pH 0- 2.5, and 11-13.

6.5 Computer with Accessories to run the LIMS system and ICOC.

6.6 Scanner gun.

6.7 Label printer and labels.

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 Hydrochloric Acid (HCl), Trace Metal Grade.

7.2 Nitric Acid (HNO_3), Trace Metal Grade.

7.3 Sodium Hydroxide (NaOH), Certified A.C.S.

7.4 Zinc Acetate $(CH_3CO_2)_2$ Zn $2H_2O$, certified.

7.5 Sulfuric Acid (H_2SO_4), Trace Metal Grade.

7.6 Phosphoric Acid (H_3PO_4), Trace Metal Grade.

Reagents are ordered by the inorganic laboratory section, and kept in the sample receiving and bottle prep areas. Zinc Acetate is stored in the volatile laboratory as it is only used with volatile sample bottle prep.

If the reagent needed is in neat form (NaOH or Zinc Acetate) a new bottle may be obtained from the chemical storage area. If you take the next to the last or the last bottle for login dept use, you must inform the inorganic lab supervisor so more can be ordered.

8. Procedure

8.1 Log-In Procedures:

Samples received from private courier (FedEx, UPS, etc.) or Spectrum courier or delivery by the clients. All samples received must be accompanied by a chain-of-custody (COC) record (see **Attachment 7** for a Spectrum COC).

8.1.1 Prior to opening coolers the receiving hood must be turned on, and safety glasses, lab coat, and gloves worn.

8.1.2 Select the next sequential red workorder folder with a pre-assigned work order number. Work order numbers consist of one alpha character followed by a four digit number YXXXX, in which Y is the alpha character indicating the number of years that the Omega LIMS System has been operational and XXXX is a sequential number for this workorder for the current year. For example, 2006 is assigned the alpha character "E" and the 5th set of samples received during 2006 is assigned work order number E0005. Occasionally, a client wishes for a workorder number to be ongoing for several shipments of samples (i.e. EPA). The project manager will notify the sample custodian if additional samples are to be added to an existing workorder (an open Sample Delivery Group or SDG).

8.1.3 Before opening coolers, check the outside of the opening of the shipping cooler for the presence of custody seals. Remove any shipping air bills and note the work order number on them. If an EPA cooler is received without an air bill attached, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information. This information should be noted on the Sample Condition Form (See **Attachments 1 and 2**).

NOTE: There are two different types of Sample Condition Forms; one for commercial samples, and one for the US EPA contract samples. Place air bills and all other paperwork for this shipment in the appropriate red workorder folder.

8.1.4 Open the cooler underneath the hood and remove the chain-of-custody (COC) form. If a large number of coolers are received, or the cooler is extremely heavy, open it briefly in hallway or on floor and remove the COC only. This is typically located in a plastic zip-lock bag on the top of the samples or taped to the inside of the cooler lid. Write the workorder number on the COC. Sign the COC, noting the date and time of receipt of the samples. Proceed with the next steps only after the cooler is under the hood. Look for return air bills to return coolers to the client; these are typically included in EPA projects. If the COC is missing, notify the project manager using the Sample Condition Notification (printed on orange paper, See **Attachment 3**) form immediately. Do not process the samples. Store the samples in the walk-in cooler until the client is contacted and responds.

8.1.5 Take the temperature of the contents using the temperature gun or thermometer.

8.1.5.1 Use the Temperature Gun as follows:

8.1.5.1.1 Unwrap a sample in the opened cooler. Unwrap enough of the sample in order to take a temperature reading.

8.1.5.1.2 Take the temperature by holding the gun no more than two inches from the sample container. Do not aim at the label or cap of the sample. Take several readings on at least 2 samples at opposite ends of the cooler. Record the temperature on the Sample Condition Form and the lower right hand corner of the chain-of-custody or where noted on the Chain-of-Custody. The acceptable temperature range is 4° ± 2°C (2° to 6°C). If the temperature is acceptable, move on to **Section 8.1.6**. If the temperature is not within this range, enter the temperature on the Sample Condition Form, circle "yes" to the question regarding the Sample Notification form, and fill out the Sample Notification form. For CLP projects, if the temperature exceeds 10°C, the project manager will contact SMO for corrective action guidance. Give the entire folder and the Sample Notification form to the project manager. The project manager will contact the client for instructions on how to proceed and return the folder to the sample custodian indicating action to be taken. During the interim, the samples in question will not be processed, but the closed cooler will be stored in the walk-in cooler.

8.1.5.1.3 Record the Temperature Gun ID on the sample condition form.

8.1.5.2 If a temperature blank is present, use a thermometer as follows:

8.1.5.2.1 Insert the thermometer in the temperature blank.

8.1.5.2.2 Let stand for a minimum of three minutes to as long as five minutes maximum.

8.1.5.2.3 Read the temperature and record it on the Sample Condition Form and in the lower right hand corner of the COC or where noted on the Chain-of-Custody. If the temperature is not within 4° ± 2°C (2° to 6°C), proceed as in **section 8.1.4** above. For CLP projects, if the temperature exceeds 10°C, the project manager will contact SMO for corrective action.

8.1.5.3 A calibration check of the thermometer is to be done quarterly or as the thermometer is replaced by comparing it's reading to the NIST thermometer and recording the results in the QA/QC Thermometer Calibration Logbook. See **SOP No. 110.0006** for the calibration procedure and example logbook page.

8.1.5.4 The presence/absence of a temperature blank in the shipment is documented on the Form DC-1. Follow the instructions on the DC-1 Form for contacting SMO, and for documentation in the event a temperature blank is not present.

8.1.6 If the Sample Custodian is not present, the person receiving the samples should perform all steps through **Section 8.1.5.2.3** then store the cooler in the walk-in refrigerator, R1, until the Sample Custodian returns, after removing any VOC samples. Samples for VOC analysis require special handling, as described in **Section 8.2.2**. In general, if not logged-in immediately, VOA vials must be removed from the cooler and stored in refrigerator R2 until logged-in to the LIMS. EPA SOM samples needing temporary storage should be discussed with the SOM project manager. A storage blank is required if temporary storage is used. If VOC samples are received in Encore devices (typically contained in silver foil pouches), or are noted as requiring freezing to preserve, they must be removed and stored in the freezer F2. Encore samples must be extruded into pre-weighed VOC vials as soon as possible. Contact one of the following to insure this occurs: VOA Laboratory Supervisor, Sample Custodian, Project Manager, Laboratory Director or QA Director. Leave the paperwork in the red folder on the sample custodian's desk.

8.1.7 Once all of the coolers received have been inspected as above, prioritize them for logging into the LIMS with fastest turn-around times and short holding times processed first. The labs should be notified immediately about quick-turn analyses. This can be done either verbally, or by copying the COC and distributing that to the laboratories.

8.1.8 Remove and inspect samples from the coolers for the first workorder. Sample containers are removed from the cooler and lined-up on the counter in the same order as listed on the chain of custody form. Check to see that the correct number of samples is present. Check to see if any samples are broken. Check aqueous VOA sample vials for the presence of air bubbles or headspace. If bubbles or headspace are present, a Sample Notification form should be filled out and given to the Project Manager. Check to see that the quantity present is sufficient for the test requested. Make sure the client IDs on the bottles agree with the COC. Check the custody seals, if any, on the sample containers to ensure the all seals are present and intact.

8.1.9 For U.S. EPA samples, sign the COC and note the temperature on the bottom right corner of the COC. Place the project number in the top right hand corner of the COC. Record the following information on the Form DC-1.

- Sign DC-1. Note that the person signing should be the person who signed for receipt on the COC. There will be one DC-1 form for each cooler received for a particular case. Only the samples in that particular cooler will be listed on that DC-1 form.
- EPA Case number from the COC.
- List sample tag control numbers and compare these with the COC records. Then place sample tags in clear Ziploc bag.
- Document whether or not these numbers agree.
- If Sample tag numbers are not listed on the COC form, record this fact.
- Record the custody seal numbers if present.
- Write the work order number on the air bill and place it in the red project folder. Record the name of the courier and the air bill number on Form DC-1.
- Date of receipt.
- Time of receipt.
- EPA sample numbers.
- SDG-Final Sample must be documented on EPA TR/COC.
- SDG (Sample Delivery Group) number. The SDG is the lowest alphanumeric sample on the first shipment and remains the same until the work order is closed.
- EPA sample numbers.
- Assigned Lab ID numbers. Note that with EPA projects, shipments may be received over several days to be added to the same work order, up to twenty field samples, not including PE samples. The SDG number stays the same, but the sample ID numbers run consecutively.
- Whether samples were delivered by hand or not.
- Any problems and discrepancies in "remarks" box.
- The cooler temperature is entered in item #10 of the Form DC-1.
- Inorganic sample pH.
- Note presence of ice in "remarks" box.

Note from the COC whether the work order is closed, or if more samples are expected to be added (work order/SDG is still open). EPA projects are closed automatically after one week from the date of receipt, for cases with a turnaround time of 14 or 21 days and three days for samples with a turnaround time of 7 days, of the first shipment or sooner if the client checks the box that the project is closed.

Discrepancies in EPA shipments should be reported to the Project Manager directly. Any irregularities in the above information should be documented and the client contacted by either Project Manager or the Sample Custodian. Until an answer is received from the client, samples should be stored in the walk-in cooler. Volatile samples must be stored in R/F2.

If there are any discrepancies, note them on the Sample Condition Notification Form and give the form and the workorder folder to the project manager. Samples with issues will not be processed further, but stored in the walk-in cooler until a resolution has been reached with the client. For CLP (EPA) samples, the Sample Management Office (SMO) will be contacted for corrective action. Communication of the corrective action will be placed in the red project folder.

8.1.10 Log the workorder and samples into the Laboratory Information Management System (LIMS) following procedures described in SOP 20.0003. Labels are generated and attached to each sample container. In the case of pre-preserved samples in VOA vials (sodium bisulfate, DI water and/or methanol), be sure the lab sample ID label does not cover the tare weight of the vials.

8.1.11 Have the project manager or a second person check the Lab IDs on the labels vs. the client IDs on the COC to confirm the bottles have been properly labeled. Ensure the reviewer initials the proper line of the Sample Condition Form or DC-1 form.

8.1.12 Scan the individual containers into the ICOC system per SOP No. 30.0030.

8.1.13 Inorganic preserved samples must now be tested for pH as follows:

For metals, wet chemistry, etc., pour a small amount of sample over a strip of pH paper that is suspended over the mouth of a waste container. The pH of the acidified samples is taken by using the narrow range pH strips which measures pH 0 to 2.5. The sample pH should be less than two, as indicated by the color on the pH indicator package. The pH of the basic samples is taken by using the narrow range pH strips which measures pH 11.0 – 13.0. The basic samples should have a pH 12 or greater, as indicated by the color of the pH indicator package. (See **Attachment 4** for instructions on the use of the pH paper.) Acidified samples must have a pH of less than 2. Any pH adjustments that need to be made are recorded on the Sample Condition Form for the workorder. Record the lot number and ID of the reagent used and the approximate volume used to adjust the pH. For metals samples requiring acidification, alert the

inorganic laboratory to allow 24 hours before metals preparation to allow enough time for the acids to dissolve any metals that adsorb to container walls. Record the pH on the DC-1 form or Sample Condition form (non-EPA). For ISM samples, if the samples require any pH adjustment, this must be noted in the SDG narrative. If the pH of a cyanide sample is <12, SMO must be contacted for further instruction. Do not adjust the pH or proceed with any preparation/analysis.

8.1.14 Some samples (i.e. fecal coliform bacteria) which require subcontracting must be done immediately as the analysis has an extremely short hold time. A list of analyses typically subcontracted can be printed from the LIMS system. An Outside Services Purchase Order is also generated from the LIMS system when the batch of samples to subcontract is created. See SOP No. 110.0023 Project Management for more details and examples.

8.1.14.1 Receiving personnel make one copy of the sub contract COC. This COC accompanies the samples and is signed by the laboratory that will perform the analysis. A copy of the COC is placed into the red workorder folder and one copy is sent to Accounting.

8.2 Storage and Location of Samples:

8.2.1 Semivolatile Organic, Pesticides/PCBs, and Wet Chemistry Samples:

Samples are stored in the main walk-in refrigerator (R1) in the sample receipt room at 4° ± 2°C. Wet Chemistry samples for TOC (SM5310B/SW9060) and/or COD (SM5220D) may be stored in refrigerator (R8) in Unit 3 as well.

8.2.2 Volatile Organic Samples:

Samples for VOA are stored separately from all other samples, in refrigerators or freezers dedicated for only this purpose. These refrigerators and freezers are located in the VOA Laboratory. A soil sample for VOA may contain one or more of the sample types listed below. Care must be taken to insure each sample vial type receives the proper handling. Soil samples for volatiles analysis must have special handling performed by the VOA lab staff.

Storage blanks must accompany all VOA samples requiring EPA/CLP or New York State ASP analyses when placed in the VOA refrigerators. The storage blanks are prepared in the volatiles department using the VOA Type I water. The 40ml vial is filled so there is **no** headspace, capped immediately and tightly, and labeled with the date prepared and any preservative added. The storage blanks for unpreserved low/medium soil samples are prepared by placing approximately 5 g of inert sand in dry closed-system purge-and-trap vials. These inert sand storage blanks will be stored in the same freezer with unpreserved low/medium soil samples.

8.2.2.1 Samples suspected of containing high concentrations of volatile organics must be stored in a separate VOA refrigerator designated for such samples. This isolates these high concentration samples from other samples, and helps to control cross-contamination.

8.2.2.2 Soil samples in pre-weighed, dry closed-system vials (no liquid inside the vial) should be stored in a VOA freezer (containing no standards or sample extracts). These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.3 Soil samples that contain up to 5 mL of water. These vials are also to be stored in a VOA freezer. These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.4 Soil samples that contain 5mL of water containing a sodium bisulfate preservative. These vials are also to be held in a VOA refrigerator (specifically for EPA CLP samples). Samples for Method 8260 analysis may be held in a VOA refrigerator. These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.5 Soil samples received in the Encores (typically contained in silver foil pouches), the samples must be extruded into tared, dry closed-system purge-and-trap vials by the VOA lab staff, and then re-weighed to obtain the final sample weight. This should happen immediately (for EPA CLP samples), but no longer than 48hours of sample collection. The vials are then to be placed into a VOA freezer.

8.2.2.6 Soil samples may also arrive as methanol preserved samples. These samples do not require freezing, but they shall be held in a VOA refrigerator to be weighed by VOA lab staff prior to analysis. If possible, check that the methanol covers the soil samples within the vial. If the methanol is insufficient, note this on the Sample Condition Form and relay the information to the VOA lab.

8.2.2.7 Unpreserved VOA soil samples should be stored in a VOA freezer (EPA CLP samples) or a VOA refrigerator (for Method 8260 samples) until the time of analysis.

8.2.2.8 Water samples are stored in VOA refrigerators. Trace Volatile samples for the EPA/CLP SOM01.2 contract must be stored in a separate refrigerator from all other samples. At present, this has been designated as **R13 and R4**.

8.2.2.9 Log the samples into the VOA lab. Enter the workorder and other pertinent information in the Volatile Receiving Logbook (See

Attachment 6) and sign off that the samples have been relinquished. Different sample preservation types that result in different storage designations require separate line entries in the logbook (ex: SOM methanol extracts into refrigerator on one line, associated unpreserved soils into freezer on another line) If the samples are ever removed from VOA they must be signed out of the VOA lab as well.

8.2.3 Metals Samples:

Aqueous preserved samples for metals analyses that do not require refrigeration are stored on the "M" rack in Unit 3.

8.2.3.1 ISM water and soil samples require refrigeration at $4^{o} \pm 2^{o}C$ and must be stored in the walk-in. Cyanide aqueous samples must also be protected from light. Only aqueous cyanide samples are required to remain stored in refrigeration after distillation. All other matrices and ISM test methods may be stored at room temperature after preparation, until disposal.

Standards must be stored in separate freezers or refrigerators isolated from samples and sample extracts.

8.3 Storage and Location of Extracts:

Extracts for semivolatile and Pest/PCB analyses must be stored, shielded from light, at $4 \pm 2^{o}C$ in refrigerators in the laboratory. Extracts must be stored separately from standards.

Extracts for semivolatile and Pest/PCB must be stored until the proper time to dispose of them per client approval. The lab normally retains sample extracts for a period of three months from the date of delivery of the final report to the client

USEPA CLP and New York State ASP extracts must be stored for 365 days after delivery of a complete data package to the client.

8.4 Temperature Monitoring:

The temperature of the storage areas must be monitored on a daily basis, including weekends and holidays. This temperature is recorded electronically using temperature probes that are affixed inside all refrigerator and freezer units. The temperatures are recorded electronically multiple times daily. Specific employees are notified of temperatures outside of the acceptance ranges by email so that corrective action can be initiated. If any refrigerator/freezer fails, first investigate the problem. Was the door ajar? Is something preventing the door from shutting fully? If the situation can be rectified, do so and check the temperature again after a sufficient period of time. Adjust the temperature control knob in the unit if other attempts have not fixed the problem and continue to monitor the temperature. All adjustments and resolutions, as well as

potential impact of the temperature issues, must be entered into the Temperature Probe Program "Event" log. When the temperature meets criteria, document that the refrigerator/freezer has been *"returned to control"*. If there is no obvious issue with the refrigerator/freezer, or the refrigerator does not maintain the correct temperature, the QA Director or Lab Director must be notified immediately to correct the problem. Acceptable temperature ranges are $2^{\circ} - 6^{\circ}C$ for refrigerators and $-10^{\circ}C$ to $-20^{\circ}C$ for freezers. See SOP 80.0020, Temperature Monitoring Systems for more information.

8.5 Tracking of Samples and Extracts:

8.5.1 Sample Log-out:

When samples are taken from their storage areas, they must be signed out using the ICOC system. During 2010, we transitioned from a paper documentation system to the use of bar-coded scanners to document chain of custody of samples from the storage area into the laboratories for preparation and/or analysis. Extracts and digestates are not bar-coded. See SOP No. 30.0030 for full details on the use of the ICOC system and examples of ICOC reports.

The analyst retrieves the samples from the walk-in cooler using the shelf location information stored in LIMS, and checks the sample numbers. The analyst logs into the ICOC system which records his/her identity. The sample containers are then scanned out using the barcode system. The lab sample numbers as well as the bottle # are recorded (i.e. J0140-02B#2). The time and date are also recorded. When the sample is logged-out by an analyst, it is considered to be in the custody of that analyst until he/she returns the sample to its storage area.

Upon the sample's return to the storage area the samples are scanned back into the receiving area using the same procedure. Samples that were originally scanned out as "consumed" will not be returned to storage. EPA samples empty containers are required to be stored per **Section 8.5.4**.

A list of Authorized Laboratory Personnel is posted on the Receiving area bulletin board. (See **Attachment 5**) Authorized laboratory personnel are employees who are knowledgeable in regard to the receipt and log-in of samples.

8.5.2 Sample Extraction:

If a sample is signed out for extraction, the sample is logged into the lab by entering the Lab ID into the appropriate extraction logbook. After extraction, any remaining sample volume is returned to the storage area and scanned back into the Receiving area for storage as noted in **Section 8.5.1**.

When working with samples in the preparation laboratories, the following procedures must be adhered to:

° All activities performed on the sample must be recorded.
° Entries in the logbooks must be signed and dated (mm/dd/yyyy).
° All entries must be made in ink.
° Corrections and additions must be made by drawing a single line through the errors, entering the correct information, and initializing and dating the new information.
° Unused portions of the logbooks must be lined-out or "Z-ed" out.
° Logbook entries must be made in chronological order.
° For US EPA/CLP samples, entries are recorded for only one SDG on a page, except in the event where SDGs share quality control samples.
° Information inserted in the logbooks must be permanently affixed, signed, and dated across the insert.
° Copies of all pertinent logbook pages must be placed in the workorder file.
° Proper subsampling techniques can be located in the Subsampling SOP, 110.0039.

8.5.3 Extracts/Digestates:

The organic sample extracts, once completed by the preparation laboratory, are transferred to the appropriate instrumentation lab refrigerator. Inorganic metals and mercury digestates are transferred to the instrumentation labs. Transfer of extracts and digestates is documented in the preparation batch logbook by the prep technician. Organic extracts are transferred directly to a specific refrigerator. Inorganic digestates/distillates are transferred directly to either the mercury or metals lab, or Unit 3 for Cyanide (See example prep log, **Attachment 8**).

When the time period for organic extract storage has expired, extracts may be disposed of in the same manner as would used/expired standards. CLP extracts must be stored for one year.

When the time period for inorganic digestate storage has expired, digestates are disposed in the same manner as would used/expired inorganic standards.

See SOP No. 30.0024 Sample and Waste Disposal for details on the actual disposal of above extracts/digestates.

8.5.4 Sample Disposal:

The unused portion of sample is returned to the Receiving area for long term storage. Such portions must be stored for a certain length of time as required by client contract and then properly disposed.

All unused portions of EPA SOM and ISM samples must be protected from light and refrigerated at 4°C ± 2°C(where appropriate for sample type) from the time of receipt until 60 days after the delivery of a complete, reconciled data package. Empty containers must also be retained for a period of 60 days after data submission, but do not require refrigeration. Presently empty containers are held in the storage hallway on specified shelves, in Unit 3 and/or the locked storage shed. A sample list is maintained. After 60 days, the samples and containers may be disposed of in a manner that complies with all applicable regulations.

Unused sample is disposed of with the use of a licensed hazardous waste contractor. Routinely, one of the sample custodians runs the LIMS Omega Disposal program to produce a list of samples ready for disposal. See SOP No. 30.0024 Sample and Waste Disposal for details on the different waste streams and disposal options.

All soil samples received from outside of the 48 contiguous United States must be disposed of following special procedures outlined by the USDA, as detailed in SOP No. 30.0024 Sample and Waste Disposal section 8.1.4 Lab Solids, Broken Glass, Methylene Chloride, Acetone.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

10.1 The Sample Custodian will calibrate the temperature gun using a NIST calibrated thermometer on a quarterly basis. The correction factor will be noted on the temperature gun and recorded in the NIST Thermometer Calibration Logbook. The serial number of the NIST thermometer will also be recorded in the logbook.

10.2 On a daily basis, the Project Managers will review that the Sample Condition Forms are filled out correctly.

10.3 Multiple times daily, the temperatures of the refrigerators and freezers will be checked and the temperatures recorded electronically. Deviations will be noted in the electronic logbook and appropriate personnel notified for corrective action. If after 36 hours the temperature problem cannot be corrected with manual adjustments, the refrigerator or freezer will be replaced.

10.4 The Hazardous Waste Inspection Logbook will be completed weekly, after an inspection of the Hazardous Waste shed by a Sample Custodian. Major problems will be reported to the Hazardous Waste Coordinator.

11. Data Validation and Reporting

A copy of the chain of custody and Sample Condition Notification form accompanies the data reports to the client. Also any communication records are to be included in the data package (telephone conversations, e-mail communication, etc.). Print outs of the ICOC reports may be included in data packages as required.

12. Data Management and Records Management

All final data packages and associated department .mdb files (zipped files) are stored in the associated electronic workorder file. NRD data associated with the project may or may not include additional documents related to sample receipt, storage, tracking and disposal. This data is managed per SOP No. 110.0029 Electronic Data Management.

13. Corrective Action Procedures

Corrective actions to be implemented in the event QC results are outside of the acceptance range. Notification reports (orange sheets) are generated in the event of an out-of-control situation occurs at sample log-in that cannot be corrected. Project Managers contact the client in order to ensure what type, if any, corrective action needs to be taken. Other corrective actions concerning the Receiving Area are recorded using the LIMS Corrective Action Report.

14. Health and Safety

The sample custodian and analysts must wear a lab coat, safety glasses, and gloves when handling samples and preservative chemicals. Samples coolers should be opened under a hood. All samples should be handled as though they are potentially hazardous. The outside of sample containers should also be handled as though they are contaminated. Any broken sample containers must be handled very carefully to prevent exposure to the sample.

15. Pollution Prevention, Waste Management, and Abbreviations

See sections 19.0 (Waste Management) and 20.0 (Definitions, Acronyms, and Abbreviations) of the current Quality Assurance Plan.

16. References

US EPA CLP SOW SOM01.2
US EPA CLP SOW ISM01.3
US ACOE Shell for Chemical Analytical Requirements, Appendix H
Quality Assurance Plan

Attachments:

1. **Attachment 1**: Sample Condition Form.
2. **Attachment 2**: EPA Sample Condition Form (DC-1).

3. **Attachment 3**: Sample Condition Notification Form.
4. **Attachment 4**: Instructions for the use of pH paper.
5. **Attachment 5**: Example Authorized Personnel list.
6. **Attachment 6**: VOA Receiving Logbook.
7. **Attachment 7**: Spectrum Chain of Custody
8. **Attachment 8**: Example Prep log.

Attachment 1
Sample Condition Form

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

Received By: [redacted]	Page 01 of 01
Reviewed By: [redacted]	Log-in Date [redacted]
Work Order: [redacted]	Client Name: [redacted]
Project Name/Event: [redacted]	

Remarks: (1/2) Please see associated sample/extract transfer logbook pages submitted with this data package.

1. Custody Seal(s)	Present / Absent
	Intact / Broken
2. Custody Seal Nos.	N/A
3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists	Present / Absent
4. Airbill	AirBill / Sticker
	Present / Absent
5. Airbill No.	FedEx 7975 5003 4627
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	3 °C
10. Does information on TR/COCs and sample tags agree?	Yes / No
11. Date Received at Laboratory	09/24/2011
12. Time Received	08:30

Sample Transfer	
Fraction (1) TVOA/VOA	Fraction (2) SVOA/PEST/ARO
Area #	Area #
By	By
On	On

Lab Sample ID	Preservation (pH) HNO3	H2SO4	HCl	NaOH	H3PO4	VOA Matrix	Soil HeadSpace or Air Bubble > or equal to 1/4"
[redacted]	<2		<2			H	

IR Temp Gun ID:

CoolantCondition: ICE

Preservative Name/Lot No:

VOA Matrix Key:

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

See Sample Condition Notification/Corrective Action Form Yes / No

Rad OK Yes / No

WO: K1839 / CID:000469 / CW10:000672

Sample Condition Form

Attachment 2

EPA Sample Condition Form (DC-1)

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.

Attachment 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: _____

_____

_____

_____

_____

Attachment 4

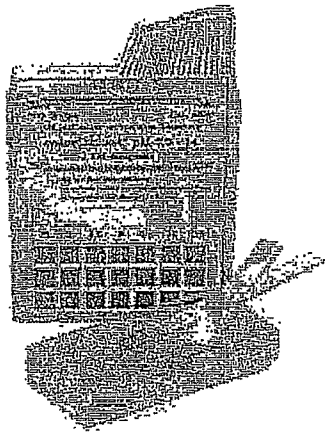
Instructions for use of pH paper

General Procedure for the Use of colorpHast® pH Test Strips

1. Remove a strip from the box.
2. Immerse the reaction zone of the strip into the solution for approximately 30 seconds to 1 minute, allowing enough time for the reaction to take place.
3. Remove the strip from the solution, wiping the strip along the edge of the vessel to remove excess liquid from the strip.
4. Compare the strip's reaction zones to the color chart on the box while holding the strip to the top of the box and the reaction zones to the bottom of the box.
5. Record the results of the closest matching color to the strip.

For additional application or product information, ordering information or other technical questions, please call our Technical Service Department at:

1-800-222-0342 *or* **1-856-423-6300**



EMD Chemicals Inc.
480 South Democrat Road
Gibbstown, NJ 08027

Phone: 856-423-6300
Fax: 856-423-4389
www.emdchemicals.com

Attachment 5

Example Authorized Personnel list

Sample log-in/out: **Authorized Personnel**		
Name	**Dept**	**Initials**
Anderson, Courtney	Inorganics	*CJA*
Appolonia, Gary	Inorganics	*GMD*
Cardoso, Antonio	Oprep	*AC*
Cartwright, Jillian	Oprep	*JC*
Datta, Avijit	Inorganics	*AED*
Ding, Yihai	Mitkem	*YD*
Dlhopolsky, Brian	Oprep	*BD*
Dorsey, James Kyle	Oprep	*JKD*
Gaudreau, Veronica	Receiving	*VEG*
Huntley, Agnes	Project Management	*ARH*
Kaczorowski, Przemyslaw	SVOA	*PK*
Lawler, Edward	Project Management	*EL*
Lawler, Sharyn	QA/QC	*SBL*
Luo, Wei	VOA	*WL*
Lucas, Derek	PEST/PCB	*DL*
Maczewska, Beata	SVOA	*BM*
Marquis, Ashley	VOA	*ALM*
McDaniel, Timothy	Oprep	*TM*
Mosher, Catherine	SVOA	*CLM*
Montmarquet, Timothy	Mitkem	*TM*
Muratori, Katherine	VOA	*KMM*
Nadeau, Cory	Receiving	*CAN*
Ng, Shirley	Project Management	*SN*
Rosadzinski, Tomasz	Inorganics	*TR*
Scarpaci, Matthew	SVOA	*MMS*
Sawyer, Tom	Inorganics	*TS*
Slavin, Dennis	Oprep	*DS*
Smart, Dawne	Mitkem	*DES*
Thomas, Michael	Oprep	*MT*
Thomson, Cassandra	PEST/PCB	*CT*
Zharkova, Sofya	VOA	*SZ*
Zhao-Anderson, Huiyan	Inorganics	*HZA*

Most common personnel using log-in/out

Attachment 6

Volatiles Receiving Logbook

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 90.0191-07/11

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

Attachment 7:
Spectrum Chain of Custody

MITKEM LABORATORIES

A Division of SPECTRUM ANALYTICAL, INC. Featuring HANIBAL TECHNOLOGY

CHAIN OF CUSTODY RECORD

Page _____ of _____

Special Handling:

- ☐ Standard TAT - 7 to 10 business days
- ☐ Rush TAT - 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: _____

Invoice To: _____

Project No.: _____

Site Name: _____

Location: _____ State: _____

Project Mgr.: _____

P.O. No.: _____ RQN: _____

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=_____ 10=_____

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	Preservative	Containers: # of VOA Vials	# of Amber Glass	# of Clear Glass	# of Plastic		Analyses:								QA Reporting Notes: (check if needed)
																				☐ Provide MA DEP MCP CAM Report ☐ Provide CT DEP RCP Report **QA/QC Reporting Level** ☐ Standard ☐ No QC ☐ Other_____ State specific reporting standards:

☐ Fax results when available to (____) _____ ☐ E-mail to _____ EDD Format _____	Relinquished by:	Received by:	Date:	Time:
Condition upon receipt: ☐ Iced ☐ Ambient ☐ °C _____				

175 Metro Center Boulevard • Warwick, RI 02886-1755 • 401-732-3400 • Fax 401-732-3499 • www.mitkem.com

Attachment 8:
Example Prep Log

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

Prep Start Date: **4/12/2011 8:50:00 A**
Prep End Date: **4/13/2011 3:44:00 P** Prep Code: **SOM01.0_SVOA_LOW_S_PR** Prep Type: **SONC/SW3550B** Prep Factor Units: mL / g
Prep Batch ID: **58533** Technician: **Antonio AP Cardoso**

QC Matrix: NASO4	Solvent (1): ACE	Solvent (3): N/A	Solvent (5): N/A	Clean Up (1): N/A	Clean Up (3): N/A
QC Matrix Lot: 103963	Solvent (1) Lot: 107200	Solvent (3) Lot: N/A	Solvent (5) Lot: N/A	Clean Up (1) Lot: N/A	Clean Up (1) Lot: N/A
Filter?: FILTER	Solvent (2): MECL2	Solvent (4): N/A	Solvent (6): N/A	Clean Up (2): N/A	Clean Up (4): N/A
Filter Lot: FC002266	Solvent (2) Lot: DD795	Solvent (4) Lot: N/A	Solvent (6) Lot: N/A	Clean Up (2) Lot: N/A	Clean Up (4) Lot: N/A

Start Time: N/A End Time: N/A | Cycles/Hour 0 | Sonicator Tuned?Yes | Bath Temp1 (C): 87 | Therm ID1: 88

Mitkem Sample ID	Client Samp ID	M	Initial (mL/g)	Final (mL)	Surrogate Spike ID	Surr (mL)	LCS/D MS/D Spike ID	Spike (mL)	A* Init	W* Init	Due Date	Bottle Number	Trans Date	Trans By	Storage	pH	pH >11	pH <2	CNCNTR Unit
MB-58533	BatchQC		30	10	OSW110328A	1			APC	TM			04/13/11	JLC	R7		☐	☐	KD 1
K0570-01A	B9248	S	30.3	10	OSW110328A	1			APC	TM	04/15/11	01	04/13/11	JLC	R7		☐	☐	KD 1
	CLEAN UP (K0570-01A): GPC3_110412A/tmcdaniel																		
K0570-02A	B9249	S	30.4	10	OSW110328A	1			APC	TM	04/15/11	01	04/13/11	JLC	R7		☐	☐	KD 1
	CLEAN UP (K0570-02A): GPC3_110412A/tmcdaniel																		
K0570-03A	B9250	S	30	10	OSW110328A	1			APC	TM	04/15/11	01	04/13/11	JLC	R7		☐	☐	KD 1
	CLEAN UP (K0570-03A): GPC3_110412A/tmcdaniel																		
K0570-04A	B9251	S	30.6	10	OSW110328A	1			APC	TM	04/15/11	01	04/13/11	JLC	R7		☐	☐	KD 1
	CLEAN UP (K0570-04A): GPC3_110412A/tmcdaniel																		
K0570-05A	B9252	S	30.5	10	OSW110328A	1			APC	TM	04/15/11	01	04/13/11	JLC	R7		☐	☐	KD 1
	CLEAN UP (K0570-05A): GPC3_110412A/tmcdaniel																		
K0570-06A	B9253	S	30.5	10	OSW110328A	1			APC	TM	04/15/11	01	04/13/11	JLC	R7		☐	☐	KD 1
	CLEAN UP (K0570-06A): GPC3_110412A/tmcdaniel																		
K0570-07A	B9254	S	30.3	10	OSW110328A	1			APC	TM	04/15/11	01	04/13/11	JLC	R7		☐	☐	KD 1
	CLEAN UP (K0570-07A): GPC3_110412A/tmcdaniel																		
K0570-08A	B9255	S	30.1	10	OSW110328A	1			APC	TM	04/15/11	01	04/13/11	JLC	R7		☐	☐	KD 1
	CLEAN UP (K0570-08A): GPC3_110412A/tmcdaniel																		
K0570-09A	B9256	S	30.1	10	OSW110328A	1			APC	TM	04/15/11	01	04/13/11	JLC	R7		☐	☐	KD 1
	CLEAN UP (K0570-09A): GPC3_110412A/tmcdaniel																		
K0570-10A	B9257	S	30	10	OSW110328A	1			APC	TM	04/15/11	01	04/13/11	JLC	R7		☐	☐	KD 1
	CLEAN UP (K0570-10A): GPC3_110412A/tmcdaniel																		

Jill L Cartwright 04/13/2011
Analyst Reviewed Date

Timothy McDaniel 04/13/2011
Manager Reviewed Date

Comments:

*A = Analyst (Spiked) *W = Witnessed (Spike) *T = Transferred

ATTACHMENT G

FIELD SAMPLING STANDARD OPERATING PROCEDURES

SOP Title: Decontamination of Field Equipment

1.0 Purpose and Scope

This document defines the standard operating procedures (SOP) for decontaminating field equipment commonly used during groundwater sampling activities at the Hooker/Ruco Site.

2.0 Equipment List

- Bladder pump
- Multi-parameter water quality meter
- Water level meter
- Plastic bristle brush
- Three deep water basins or 4-inch PVC pipes
- Deionized or distilled water
- Potable water
- Non-phosphate soap such as Alconox®
- Paper towels

3.0 Procedures

This section provides the step-by-step procedures for decontaminating the field equipment.

Bladder Pump:

1. All material on the exterior of the pump shall be removed with a brush.
2. The bladder pump shall be operated for a period of 5 minutes in a deep basin or PVC pipe filled with potable water.
3. The bladder pump will then be operated for a period of 5 minutes in a deep basin or PVC pipe filled with non-phosphate soap solution.
4. The bladder pump will then be operated for a period of 5 minutes in a deep basin or PVC pipe filled with deionized or distilled water.
5. The bladder pump will then be allowed to air dry. Once dry, the pump will be wrapped in aluminum foil until its next use.

Multi Parameter Water Quality Meter (Horiba U-22 or equivalent):

1. The flow-through cell will be removed from the upper portion of the meter.

2. Using potable water and a brush, remove all sediment and debris from the sensors and the flow through cell. Non-phosphate soap may be used, if needed, to clean the meter.
3. Rinse the sensors and flow-through cell using potable water.
4. Re-assemble the meter.

Water Level Meter:

1. Using a paper towel, remove all debris from the water level tape and end probe.
2. Rinse the water level tape and sensor probe using potable water. If debris remains on the meter, use a brush and/or non-phosphate soap to clean.
3. Allow the meter to air dry.

Waste Handling

1. Wash waters will be collected in appropriate portable containers (e.g., 5-gallon carboys) and transported to the control building.
2. The waters will be discharged to the sanitary sewer system via the connection within the control building.
3. Solid wastes (e.g., paper towels) will be placed in appropriate containers (e.g., plastic garbage bags) and disposed with the household refuse.

SOP Title: Hach Kit Ferrous Iron Test

1.0 Purpose and Scope

This document defines the standard operating procedures (SOP) for conducting ferrous iron tests using the Hach Kit Model DR/820. This SOP describes the equipment and field procedures necessary to determine the ferrous iron content in groundwater samples.

2.0 Equipment List

- Hach DR/820 Colorimeter
- Cleaned 25 ml sample cell vials
- Ferrous iron reagent powder pillow
- Paper towels
- Field notebook

3.0 Test Procedures

This section provides the step-by-step procedures for performing the ferrous iron test using the Hach Kit. Observations made during testing will be recorded in the field notebook.

Steps:

1. Enter the stored program number for ferrous iron (Fe^{2+}) powder pillows.
   Press **PRGM**. The display will show **PRGM ?**
   Note: Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not determined**.**
2. Press **33 ENTER**. The display will show **mg/L, Fe** and the **ZERO** icon.
3. Fill a sample cell with 25 ml of sample (the blank). Remove any water on exterior of sample cell with a paper towel.
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.
5. Press **ZERO**. The cursor will move to the right, then the display will show: **0.00 mg/L Fe**.
6. Fill another sample cell with 25 ml of sample.
7. Add the contents of one ferrous iron reagent powder pillow to the sample cell (the prepared sample). Cap and invert to mix. Note: Undissolved powder does not affect accuracy.
8. Press: **TIMER ENTER**. A three minute reaction period will begin. Note: An orange color will form if ferrous iron is present.

9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.
10. Press **READ.** The cursor will move to the right, then the result in mg/l of ferrous iron will be present. Note: Standard Adjust may be performed using a prepared standard. Record the value in the field notebook.
11. Remove the water from the sample cell and rinse with potable water quickly to prevent staining of the sample cell. If the staining on the sample cell cannot be removed with soap and water, discard the sample cell.

Waste Handling

1. Wash waters will be collected in appropriate portable containers (e.g., 5-gallon carboys) and transported to the control building.
2. The water will be discharged to the sanitary sewer system via the connection in the control building.
3. Solid wastes (e.g., paper towels) will be placed in appropriate containers (e.g., plastic garbage bags) and disposed with the household refuse.

SOP Title: Soil Vapor Sampling

1.0 Purpose and Scope

This document defines the standard operating procedures (SOP) for soil vapor sampling at the Hooker/Ruco Site. Soil vapor probes are sampled in accordance with the NYSDOH's Guidance for Evaluating Soil Vapor Intrusion, dated October 2006. The soil vapor samples will be chemically analyzed using the procedures and protocols described in the Sampling, Sample Preparation, and Analysis Requirements of EPA Compendium Method TO-15.

2.0 Equipment List

- Photoionization detector (PID)
- Helium tracer gas
- Summa canister
- Silicone and teflon tubing
- Personal sampling pump (for purging)
- Helium detector

3.0 Procedures

This section provides the step-by-step procedures for soil vapor sampling.

1. Soil gas samples for assessing the vapor intrusion pathway will be collected using certified clean Summa™ canisters. Only canisters certified clean at the 100 percent level can be used for soil gas sampling activities (i.e., pre-cleaned at the laboratory in accordance with U.S. EPA's TO-15 method and documentation of the cleaning activities will be provided by the laboratory.
2. The Summa™ canisters will be fitted with a laboratory calibrated critical orifice flow regulation device sized to restrict the maximum soil gas sample collection flow rate to approximately 100 milliliters per minute (mL/min).
3. A vacuum gauge will be supplied by the laboratory and used during sample collection to measure the initial canister vacuum, canister vacuum during sample collection, and residual canister vacuum at the end of sample collection. The vacuum gauge will be returned to the laboratory and used by the laboratory to measure the residual canister vacuum upon receipt of the canisters by the laboratory. Using the same vacuum gauge throughout the entire sampling process will eliminate discrepancies between vacuum measurements that can arise from using different gauges with a potentially different sensitivity and/or calibration.
4. The canister will be connected to the soil gas probe valve at the surface casing. The sampling assembly is connected using short lengths (e.g., 1-foot) 1/4-inch or 3/8-inch diameter silicone or teflon tubing and airtight stainless

steel or brass tee-connectors and tee-valves. Fresh tubing will be used for each sample.

5. Prior to collecting a soil gas sample, the stagnant air in the sampling assembly tubes and soil gas probe casing/sand pack must be removed. The soil gas probes will be purged prior to sampling using the personal sampling pump at a flow rate of less than 200 mL/min. This ensures that the collected soil gas sample is representative of actual soil gas concentrations within the formation. Prior to sample collection, two to three purge volumes should be drawn from the probe/sample assembly, unless otherwise required by the applicable regulatory guidance. The purge data (calculated purge volume, purging rate, and duration of purging) should be recorded in the field logbook.

6. Prior to purging, leak testing will be conducted. A tracer compound (e.g., helium) is released at ground surface inside a 5-gallon bucket immediately around the soil gas probe surface casing. The tracer test is used to test for ambient air leakage down the annulus of the soil gas probe and into the soil gas sample. The tracer compound is monitored for in the field using a meter connected in-line to the sampling assembly (e.g., helium).

7. Following the leak-testing, the soil gas probe purging will commence by opening the valve to the soil gas probe. At the start and the end of the purging period, the total concentration of volatile organic vapors of the personnel sampling pump exhaust gas will be monitored using a portable PID. The PID meter will be connected in series after the personal sampling pump. PID readings will be recorded and entered in the field logbook and chain of custody form.

8. Following purging, the valve to the soil gas probe and Summa™ canister will be opened to draw the soil gas sample into the canister. The vacuum gauge reading will be recorded during sample collection. Should the vacuum gauge reading remain elevated above 10 inches mercury (Hg) for more than 30 minutes, this will be taken to indicate that the initial vacuum in the canister has not sufficiently dissipated, and that the soil screened by the soil gas probe does not produce sufficient soil gas to permit sample collection.

9. To ensure some residual vacuum in each canister following sample collection, the canister vacuum will be recorded at approximately 80 percent through the expected sample collection duration. A maximum residual vacuum of 10-inches Hg is allowed. A canister residual vacuum above this value will require continued sampling until vacuum reading is below this threshold, unless the vacuum remains above 10-inches Hg for more than 30 minutes, as described above. A minimum 0.5 to 1-inch Hg residual vacuum will be required for the sample to be considered valid, or the sampling will be repeated using a fresh Summa™ canister. Once the vacuum is measured, the safety cap will be securely tightened on the inlet of the Summa™ canister prior to shipment to the laboratory under chain-of-custody procedures.

10. The vacuum gauge provided by the laboratory will be returned with the canister samples to check residual vacuum in the laboratory prior to sample analysis and recorded on the analytical data report. This check will ensure sample integrity prior to laboratory analysis, and that the canister has not become compromised during shipment to the laboratory.
11. The canisters will be labeled noting the unique sample designation number, date, time, and sampler's initials. A bound field logbook will be maintained to record all soil gas sampling data.

Waste Handling

1. Used tubing will be placed in an appropriate container (e.g., plastic garbage bag) and disposed with the household refuse.

1.0 PURPOSE AND SCOPE

This document defines the standard operating procedure (SOP) for conducting passive groundwater samples at the Site. This SOP describes the equipment and field procedures necessary to collect groundwater samples using the passive sampling method.

2.0 EQUIPMENT LIST

Passive Sampling

- Photoionization Detector.
- Oil/water interface probe with 0.01-foot increments.
- Water quality multi-parameter meter.
- Hach® Test Kit for Fe^{+2} readings
- Calibration fluids for multi-parameter water quality meter.
- Plastic buckets for storing purged water.
- Tool kit (knife, screwdriver, etc.).
- Paper towels.
- Vendor supplied polyethylene rope tether with pre-assembled sample rings for attaching the passive samplers.
- Vendor supplied pre-filled passive diffusion bag (PDB) samplers (48"x0.75").
- Vendor supplied pre-filled deionized (DI) water blank bag and certificate of analysis for batch of DI used in the pre-filled bags.
- Vendor supplied Hydrasleeve samplers (48"x1.75" and 12"x1.75").
- DI water for field blank and certificate of analysis for batch of DI water used for field blanks.
- Ladder or some elevated stand to suspend the sampler above the well casing.
- Laboratory supplied sample jars (with preservatives added) and labels.
- Laboratory supplied cooler (ice procured separately).
- Well completion information sheet, Chain-of-Custody (COC) forms, and field notebook.
- Field calibration sheets (Table 1)
- Waterproof and permanent marker/pen.

3.0 GROUNDWATER SAMPLING PROCEDURES

This section provides step-by-step procedures for collecting passive groundwater samples. Observations [e.g., site conditions, odors, hindrances to collection, etc.) made during sample collection should be recorded in the field notebook and/or field data sheet. Prior to collecting the groundwater samples, presence of any immiscible layers will be assessed using the oil/water interface probe. If light non-aqueous phase liquid (LNAPL) and/or dense non-aqueous phase liquid (DNAPL) are present, the field personnel will contact the project team. Unless otherwise specified by the project team, no groundwater samples will be collected from wells that contain measurable LNAPL or DNAPL.

3.1 Sampler Deployment

The following procedure will be followed prior to and during deployment of the passive

samplers at each pre-specified monitoring well:

- Note the condition of the outer well casing, concrete well pad, protective posts (if present), and any unusual conditions of the area around the well in the field notebook.
- Open the pressure relief valve at the well cap to release excess pressure build-up due to the remediation system.
- Measure vapors within the outer and inner well casing with a PID.
- Measure depth to water (to nearest 0.01-foot) from the marked measuring point on the top of the inner well casing.
- Connect the passive samplers to the tether at the appropriate sample intervals (pre-installed on the tether by the Vendor) as follows:
  - Attach the 12 inch Hydrasleeve to the top sampling interval.
  - Place the 48-inch PDB bag into the protective 1-inch diameter PVC screen and connect the screen to the tether with the provided clips. Note that the protective screen is in place to prevent the PDB bag from potential damage/puncture during deployment and recovery.
  - Attach the 48-inch Hydrasleeve to the third sample interval. In the case where there is only one sample interval on a tether the 48-inch Hydrasleeve should be connected with a top weight to allow for compression of the bag within the well.
- Slowly lower sample tether with the attached samplers into the monitoring well until the tether has been completely unwound from the spool.
- Secure the sampling tether to the well cap and close the well.
- Collect the field blank and trip blank samples.
- Place blank samples into an iced cooler and deliver/ship to the laboratory.
- Retrieve the passive samplers no less than 14 days and no more than 3 months following deployment.

3.2 Sampler Collection

Groundwater samples will be collected for chemical analysis no sooner than 14 days and no later than 3 months after deploying the passive samplers. The following procedure will be followed to retrieve the passive samplers and collect the groundwater samples:

- Open the pressure relief valve at the well cap to release excess pressure build-up due to the remediation system.
- Complete identification labels for sample bottles and affix to laboratory supplied sample bottles.
- If preservatives are required for analysis, confirm that preservatives have been added to sample bottles.
- Open the well and grasp the sampler tether with two hands.
- Lift the tether in a slow, steady continuous motion to allow the Hydrasleeve samplers to expand and fill.
- Continue to remove the tether and samplers at a slow and steady pace.

- Detach the samplers from the tether as they are accessible and hang them from the back of the sampling vehicle or the elevated stand until all bags have been retrieved.
- Cut a 1-inch slice into the 12-inch Hydrasleeve bag immediately below the reed valve and, using the multi-parameter water quality meter, collect the water quality readings by inserting each probe into the sliced opening one at a time. With left over water in the Hydrasleeve use the Hach kit to collect the Fe^{+2} reading. Note that the water from this bag is to be used solely for the collection of water quality readings and will not be utilized to collect samples for laboratory analysis.
- After the water quality parameters have been measured, begin collecting the water sample(s) from the separate 48-inch Hydrasleeve bag and the 48-inch passive diffusion bag.
- Fill out and attach sample labels to the laboratory supplied sample jars prior to removing the samplers from the well being sampled at that time.
- Pierce the PDB sampler with the Vendor supplied sampling straw and fill the sample jars supplied by the laboratory for VOC analysis.
- Pierce the Hydrasleeve sampler with the Vendor supplied sampling straw and collect the remaining sample volume required for laboratory analysis into the appropriate bottleware.
- Place all sample bottleware into an iced cooler and deliver/ship the cooler to the lab or relinquish the cooler to the lab's courier.
- Once all samples have been collected, measure depth-to-water (to the nearest 0.01 foot) from the marked measuring point on the top of the well casing. Record measurements in the field notebook and/or field sampling sheet.
- Record the time of sampling in field notebook and on the COC.
- Re-insert the sampling tether, secure it to the well cap, and seal the well cap.
- Place used samplers in a garbage bag or PPE drum for proper disposal.
- Complete field documentation as referenced in section 5.0.

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4.0 CALIBRATION

Calibration of the multi-parameter water quality meters and PIDs, will be conducted on a daily basis prior to field activities. The calibrations for the water quality meters will be conducted for each parameter in accordance with the manufacturer's recommendations. Calibrations for the PIDs will be conducted in accordance with the manufacturer's instructions. All calibration readings will be recorded on the Field Instrument Calibration Sheet (Table 1).

5.0 DOCUMENTATION

Field activities will be documented in a project dedicated, bound field notebook. Water quality parameters measured during sampling will be recorded in the field notebook or on sampling sheets if preferred.

Field notebook entries for groundwater sampling should include the following at a minimum:

- Names of personnel collecting samples.
- Weather condition.
- Location Identification (Well ID).
- Sampling method used.
- Date and time of sampling.
- Condition of the well.
- Sample identification number.
- Equipment descriptions.
- Record of any QC samples from the Site.

Table 1
Field Instrument Calibration Sheet

Site:_____

Field Personnel:_____

Date:_____ Start Time:_____ Stop Time:_____

Temperature (°C):_____ Barometric Pressure (inHg):_____

Weather:_____

Fill In Calibration Information for the Appropriate Meter Below

Horiba

Model/Unit ID/Owner:_____

Parameter	Standard	Lot/Exp Date	Initial Reading	Cal Reading
DO (mg/L)	100% Saturated Air	N/A	N/A	
Temp (°C)	N/A	N/A	N/A	
Turbidity (NTU)	0.0			
	100.0			
pH (std untis)	4.0			
	7.0			
	10.0			
Conductivity (µs/cm)	4,490			
	1000			

YSI

Model/Unit ID/Owner:_____

Parameter	Standard	Lot/Exp Date	Initial Reading	Cal Reading
DO (mg/L)	100% Saturated Air	N/A	N/A	
Temp (°C)	N/A	N/A	N/A	
Turbidity (NTU)	0.0			
	100.0			
pH (std untis)	4.0			
	7.0			
	10.0			
Conductivity (µs/cm)	4,490			
	1000			

PID/FID

Model/Unit ID/Owner:_____

Initial Reading (ambient air):_____

Zeroed Reading (ambient air):_____

Cal Gas Type/Concentration:_____

Calibrated Reading:_____

ATTACHMENT H

WELL INSTALLATION DETAILS

TABLE 3.3

WELL INSTALLATION DETAILS
OPERABLE UNIT-3 BIOSPACE SYSTEM
HOOKER/RUCO SITE, HICKSVILLE, NEW YORK

Well Designation	Date Completed	Ground Surface (ft amsl)	Measuring Point Elevation [1] (ft amsl)	Top of Sandpack (ft bgs)	Top of Sandpack (ft amsl)	Top of Screen (ft bgs)	Top of Screen (ft amsl)	Bottom of Screen (ft bgs)	Bottom of Screen (ft amsl)	Bottom of Sandpack (ft bgs)	Bottom of Sandpack (ft amsl)	Well Diameter (in)	Well Screen Slot Size	Well Material
IW-1D1A	04/28/11		NA	185.0		190.0		195.0		200.0		1.25	10	BI/SS
IW-1D1L	04/28/11		NA	215.0		220.0		230.0		235.0		1.25	10	Sch. 80 PVC
IW-1D2A	04/28/11		NA	255.0		260.0		265.0		270.0		1.25	10	BI/SS
IW-2D1A	04/8/11		NA	195.0		200.0		205.0		210.0		1.25	10	BI/SS
IW-2D1L	04/8/11		NA	220.0		225.0		235.0		240.0		1.25	10	Sch. BO PVC
IW-2D2A	04/8/11		NA	255.0		260.0		265.0		270.0		1.25	10	BI/SS
IW-3D1A	03/25/11		NA	195.0		200.0		205.0		210.0		1.25	10	BI/SS
IW-3D1L	03/25/11		NA	215.0		225.0		235.0		240.0		1.25	10	Sch. 80 PVC
IW-3D2A	03/25/11		NA	255.0		260.0		265.0		270.0		1.25	10	BI/SS
IW-4D1A	01/27/11		NA	220.0		225.0		230.0		235.0		1.25	10	BI/SS
IW-4D1L	01/27/11		NA	240.0		245.0		255.0		260.0		1.25	10	Sch. 80 PVC
IW-4D2A	01/27/11		NA	270.0		275.0		280.0		285.0		1.25	10	BI/SS
IW-5D1A	04/12/11		NA	210.0		215.0		220.0		225.0		1.25	10	BI/SS
IW-5D1L	04/12/11		NA	255.0		260.0		270.0		275.0		1.25	10	Sch. 80 PVC
IW-5D2A	04/12/11		NA	295.0		300.0		305.0		310.0		1.25	10	BI/SS
IW-6D1A	01/17/11		NA	250.0		255.0		260.0		265.0		1.25	10	BI/SS
IW-6D1L	01/17/11		NA	270.0		275.0		285.0		290.0		1.25	10	Sch. 80 PVC
IW-6D2A	01/17/11		NA	295.0		300.0		305.0		310.0		1.25	10	BI/SS
IW-7D1A	03/29/11		NA	260.0		265.0		270.0		290.0		1.25	10	BI/SS
IW-7D1L	03/29/11		NA	260.0		275.0		285.0		290.0		1.25	10	Sch. 80 PVC
IW-7D2A	03/29/11		NA	295.0		300.0		305.0		310.0		1.25	10	BI/SS
IW-15D1A	10/05/10		NA	348.0		355.0		360.0		361.0		1.25	10	BI/SS
IW-15DlL	10/05/10		NA	366.0		368.0		378.0		379.0		1.25	10	BI/SS
IW-15D2A	10/05/10		NA	401.0		406.0		411.0		416.0		1.25	10	BI/SS
IW-16D1A	11/01/05	121.6	NA	380.0	-258.4	395.0	-273.4	400.0	-278.4	401.0	-279.4	1.0	10	BI/SS
IW-16D1L	11/01/05	121.6	NA	380.0	-258.4	385.0	-263.4	400.0	-278.4	401.0	-279.4	1.0	10	Sch. 80 PVC
IW-16D2A	11/01/05	121.6	NA	420.0	-298.4	425.0	-303.4	430.0	-308.4	435.0	-313.4	1.0	10	BI/SS
IW-17D1A	12/01/05	121.8	NA	330.0	-208.2	345.0	-223.2	350.0	-228.2	355.0	-233.2	1.0	10	BI/SS
IW-17D1L	12/01/05	121.8	NA	330.0	-208.2	335.0	-213.2	350.0	-228.2	355.0	-233.2	1.0	10	Sch. 80 PVC
IW-17D2A	12/01/05	121.8	NA	415.0	-293.2	420.0	-298.2	425.0	-303.2	429.5	-307.7	1.0	10	BI/SS
IW-18D1A	01/09/06	121.5	NA	344.75	-223.3	359.83	-238.3	364.83	-243.3	367.0	-245.5	1.0	10	BI/SS
IW-18D1L	01/09/06	121.5	NA	344.75	-223.3	349.83	-228.3	364.83	-243.3	367.0	-245.5	1.0	10	Sch. 80 PVC
IW-18D2A	01/09/06	121.5	NA	412.0	-290.5	420.0	-298.5	425.0	-303.5	430.0	-308.5	1.0	10	BI/SS
IW-19D1A	01/13/06		NA	337.0		355.0		360.0		360.5		1.0	10	BI/SS

TABLE 3.3

WELL INSTALLATION DETAILS
OPERABLE UNIT-3 BIOSPACE SYSTEM
HOOKER/RUCO SITE, HICKSVILLE, NEW YORK

Well Designation	Date Completed	Ground Surface (ft amsl)	Measuring Point Elevation [1] (ft amsl)	Top of Sandpack (ft bgs)	Top of Sandpack (ft amsl)	Top of Screen (ft bgs)	Top of Screen (ft amsl)	Bottom of Screen (ft bgs)	Bottom of Screen (ft amsl)	Bottom of Sandpack (ft bgs)	Bottom of Sandpack (ft amsl)	Well Diameter (in)	Well Screen Slot Size	Well Material
IW-19D1L	01/13/06		NA	337.0		345.0		360.0		360.5		1.0	10	Sch. 80 PVC
IW-19D2A	01/13/06		NA	415.0		420.0		425.0		430.0		1.0	10	BI/SS
IW-20D1A	10/13/10		NA	362.0		367.0		372.0		377.0		1.25	10	BI/SS
IW-20D1L	10/13/10		NA	385.0		390.0		400.0		402.0		1.25	10	Sch. 80 PVC
IW-20D2A	10/13/10		NA	425.0		430.0		435.0		440.0		1.25	10	BI/SS
IW-21D1A	10/23/10		NA	370.0		375.0		380.0		385.0		1.25	10	BI/SS
IW-21D1L	10/23/10		NA	395.0		400.0		410.0		411.0		1.25	10	Sch. 80 PVC
IW-21D2A	10/23/10		NA	430.0		435.0		440.0		445.0		1.25	10	BI/SS
IW-22D1A	11/03/10		NA	350.0		355.0		360.0		365.0		1.25	10	BI/SS
IW-22D1L	11/03/10		NA	380.0		385.0		395.0		396.0		1.25	10	Sch. 80 PVC
IW-22D2A	11/03/10		NA	415.0		420.0		425.0		430.0		1.25	10	BI/SS
MW-50D1[2]	02/23/95	130.6	132.63	279	-148.4	285	-154.4	305	-174.4	305	-174.4	2	10	Sch. 80 PVC
MW-50D2[2]	02/13/95	130.0	132.03	405	-275.0	415	-285.0	435	-305.0	435	-305.0	2	10	Sch. 80 PVC
MW-51D1	10/24/95	129.2	131.85	224	-94.8	235	-105.8	255	-125.8	260	-130.8	2	10	Sch. 80 PVC
MW-51D2	10/02/95	128.8	130.38	342	-213.2	350	-221.2	365	-236.2	370	-241.2	2	10	Sch. 80 PVC
MW-52S[2]	01/17/96	125.8	125.48	119.4	6.4	125	0.8	140	-14.2	142	-16.2	2	10	Sch. 80 PVC
MW-52I[2]	12/14/95	125.6	125.30	213.7	-88.1	220	-94.4	235	-109.4	237	-111.4	2	10	Sch. 80 PVC
MW-52D[2]	12/12/95	126.1	125.88	366.2	-240.1	371	-244.9	386	-259.9	387	-260.9	2	10	Sch. 80 PVC
MW-53I	06/08/95	120.7	120.73	145	-24.3	150	-29.3	170	-49.3	173	-52.3	2	10	Sch. 80 PVC
MW-53D1	06/19/95	120.8	120.80	294	-173.2	300	-179.2	330	-209.2	335	-214.2	2	10	Sch. 80 PVC
MW-53D2	06/05/95	120.7	120.66	415	-294.3	430	-309.3	460	-339.3	460	-339.3	2	10	Sch. 80 PVC
MW-56S[2]	01/26/96	133.9	133.60	98.5	35.4	105	28.9	120	13.9	123	10.9	2	10	Sch. 80 PVC
MW-56I[2]	01/25/96	133.9	133.47	253.5	-119.6	260	-126.1	275	-141.1	280	-146.1	2	10	Sch. 80 PVC
MW-57S	01/23/96	127.9	127.68	131.7	-3.8	137	-9.1	152	-24.1	155.5	-27.6	2	10	Sch. 80 PVC
MW-57I	01/25/96	128.0	127.48	184.5	-56.5	191	-63.0	206	-78.0	208	-80.0	2	10	Sch. 80 PVC
MW-58D	03/26/02	116.22	115.99	395	-278.8	400	-283.8	410	-293.8	415	-298.8	2	10	BI/SS
MW-58D1	03/26/02	116.22	115.99	460	-343.8	465	-348.8	475	-358.8	480	-363.8	2	10	BI/SS
MW-58D2	03/26/02	116.22	115.99	495	-378.8	500	-383.8	510	-393.8	515	-398.8	2	10	BI/SS
MW-59D	04/06/02	117.37	117.13	395	-277.6	400	-282.6	410	-292.6	415	-297.6	2	10	BI/SS
MW-59D1	04/06/02	117.37	117.13	460	-342.6	465	-347.6	475	-357.6	480	-362.6	2	10	BI/SS
MW-59D2	04/06/02	117.37	117.13	495	-377.6	500	-382.6	510	-392.6	515	-397.6	2	10	BI/SS
MW-60D1	03/05/02	119.02	118.70	325	-206.0	330	-211.0	340	-221.0	345	-226.0	2	10	BI/SS
MW-60S	03/08/02	118.96	118.93	175	-56.0	180	-61.0	190	-71.0	195	-76.0	2	10	BI/SS
MW-60I	03/08/02	118.96	118.93	225	-106.0	230	-111.0	240	-121.0	245	-126.0	2	10	BI/SS

TABLE 3.3

WELL INSTALLATION DETAILS
OPERABLE UNIT-3 BIOSPACE SYSTEM
HOOKER/RUCO SITE, HICKSVILLE, NEW YORK

Well Designation	Date Completed	Ground Surface (ft amsl)	Measuring Point Elevation [1] (ft amsl)	Top of Sandpack (ft bgs)	Top of Sandpack (ft amsl)	Top of Screen (ft bgs)	Top of Screen (ft amsl)	Bottom of Screen (ft bgs)	Bottom of Screen (ft amsl)	Bottom of Sandpack (ft bgs)	Bottom of Sandpack (ft amsl)	Well Diameter (in)	Well Screen Slot Size	Well Material
MW-60D	03/08/02	118.96	118.93	275	-156.0	280	-161.0	290	-171.0	295	-176.0	2	10	BI/SS
MW-61S	02/22/02	121.19	120.91	165	-43.8	170	-48.8	180	-58.8	185	-63.8	2	10	BI/SS
MW-61I	02/22/02	121.19	120.91	200	-78.8	205	-83.8	215	-93.8	220	-98.8	2	10	BI/SS
MW-61D1	02/22/02	121.19	120.91	265	-143.8	270	-148.8	280	-158.8	285	-163.8	2	10	BI/SS
MW-61D2	03/12/02	121.15	121.05	360	-238.9	365	-243.9	375	-253.9	380	-258.9	2	10	BI/SS
MW-62I	05/14/02	128.27	128.15	255	-126.7	260	-131.7	270	-141.7	275	-146.7	2	10	Sch. 80 PVC
MW-62D	04/20/02	128.03	127.82	325	-197.0	330	-202.0	340	-212.0	345	-217.0	2	10	BI/SS
MW-63S	02/18/02	118.67	118.45	175	-56.3	180	-61.3	190	-71.3	195	-76.3	2	10	Sch. 80 PVC
MW-63I	02/18/02	118.67	118.45	210	-91.3	215	-96.3	225	-106.3	230	-111.3	2	10	Sch. 80 PVC
MW-63D1	02/18/02	118.67	118.45	245	-126.3	250	-131.3	260	-141.3	265	-146.3	2	10	Sch. 80 PVC
MW-63D2	02/18/02	118.67	118.45	280	-161.3	285	-166.3	295	-176.3	300	-181.3	2	10	Sch. 80 PVC
MW-64S	02/09/02	125.66	125.59	175	-49.3	180	-54.3	190	-64.3	200	-74.3	2	10	Sch. 80 PVC
MW-64I	02/09/02	125.66	125.59	245	-119.3	250	-124.3	260	-134.3	265	-139.3	2	10	Sch. 80 PVC
MW-64D	02/09/02	125.66	125.59	285	-159.3	290	-164.3	300	-174.3	305	-179.3	2	10	Sch. 80 PVC
MW-66D2[2]	06/08/02	118.60	118.15	450	-331.4	455	-336.4	465	-346.4	475	-356.4	2	10	BI/SS
MW-66I[2]	06/19/02	118.27	118.20	290	-171.7	295	-176.7	305	-186.7	310	-191.7	2	10	BI/SS
MW-66D1[2]	06/19/02	118.27	118.20	350	-231.7	355	-236.7	365	-246.7	320	-201.7	2	10	BI/SS
MW-67S	01/11/03	118.68	118.37	440.0	-321.3	445.0	-326.3	455.0	-336.3	460.0	-341.3	2	10	BI/SS
MW-67D	01/11/03	118.68	118.33	490.0	-371.3	495.0	-376.3	505.0	-386.3	510.0	-391.3	2	10	BI/SS
MW-68S	02/09/03	119.20	118.97	455.0	-335.8	457.0	-337.8	467.0	-347.8	470.0	-350.8	2	10	BI/SS
MW-68D	02/09/03	119.20	119.00	485.0	-365.8	490.0	-370.8	500.0	-380.8	505.0	-385.8	2	10	BI/SS
MW-70D1	02/02/11			191.0		196.0		206.0		211.0		2	10	BI/SS
MW-70D2	02/02/11			241.0		246.0		256.0		257.0		2	10	BI/SS
MW-72D1	03/16/11			195.0		200.0		210.0		215.0		2	10	BI/SS
MW-72D2	03/16/11			255.0		260.0		270.0		271.0		2	10	BI/SS
MW-73D1	02/11/11			215.0		220.0		230.0		235.0		2	10	BI/SS
MW-73D2	02/11/11			255.0		260.0		270.0		271.0		2	10	BI/SS
MW-75D1	05/02/11			155.0		160.0		170.0		175.0		2	10	BI/SS
MW-75D2	05/02/11			220.0		225.0		235.0		236.0		2	10	BI/SS
MW-76S	03/03/11			75.0		80.0		90.0		95.0		2	10	BI/SS
MW-76I	03/03/11			120.0		125.0		135.0		136.0		2	10	BI/SS
MW-76D1	02/15/11			190.0		195.0		205.0		210.0		2	10	BI/SS
MW-76D2	02/15/11			260.0		265.0		275.0		276.0		2	10	BI/SS
MW-77D1	02/26/11			240.0		245.0		255.0		260.0		2	10	BI/SS

TABLE 3.3

WELL INSTALLATION DETAILS
OPERABLE UNIT-3 BIOSPACE SYSTEM
HOOKER/RUCO SITE, HICKSVILLE, NEW YORK

Well Designation	Date Completed	Ground Surface (ft amsl)	Measuring Point Elevation [1] (ft amsl)	Top of Sandpack (ft bgs)	Top of Sandpack (ft amsl)	Top of Screen (ft bgs)	Top of Screen (ft amsl)	Bottom of Screen (ft bgs)	Bottom of Screen (ft amsl)	Bottom of Sandpack (ft bgs)	Bottom of Sandpack (ft amsl)	Well Diameter (in)	Well Screen Slot Size	Well Material
MW-77D2	02/26/11			290.0		295.0		305.0		306.0		2	10	Sch. 80 PVC
MW-81D1	11/01/05	121.60	121.07	300.0	-178.40	305.0	-183.4	315.0	-193.4	319.0	-197.4	2	10	Sch. 80 PVC
MW-81D2	11/01/05	121.60	121.05	397.0	-275.40	405.0	-283.4	415.0	-293.4	416.0	-294.4	2	10	Sch. 80 PVC
MW-82D1	02/15/06	120.50	120.14	337.0	-216.50	345.0	-224.5	355.0	-234.5	355.5	-235.0	2	10	Sch. 80 PVC
MW-82D2	02/15/06	120.50	120.15	404.8	-284.30	409.4	-288.9	419.4	-298.9	420.5	-300.0	2	10	BI/SS
MW-83D1	11/06/05	121.58	120.99	300.0	-178.42	305.0	-183.4	315.0	-193.4	321.0	-199.4	2	10	Sch. 80 PVC
MW-83D2	11/06/05	121.58	121.02	385.0	-263.42	390.0	-268.4	400.0	-278.4	401.0	-279.4	2	10	Sch. 80 PVC
MW-84D1	04/12/06	121.34	120.90	335.6	-214.26	345.0	-223.7	355.0	-233.7	358.0	-236.7	2	10	Sch. 80 PVC
MW-84D2	04/12/06	121.34	120.94	382.6	-261.26	390.6	-269.3	400.6	-279.3	405.0	-283.7	2	10	BI/SS
MW-85S	12/04/10			213.0		218.0		228.0		233.0		2	10	BI/SS
MW-85I	12/04/10			273.0		277.0		287.0		287.0		2	10	BI/SS
MW-85D1	12/02/10			335.0		340.0		350.0		355.0		2	10	BI/SS
MW-85D2	12/02/10			390.0		395.0		405.0		407.0		2	10	BI/SS
MW-86D1	11/11/10			195.0		200.0		210.0		215.0		2	10	BI/SS
MW-86D2	11/11/10			345.0		350.0		360.0		365.0		2	10	BI/SS
MW-87D1	10/04/05	121.05	120.55	299.0	-177.95	307.0	-186.0	317.0	-196.0	319.0	-198.0	2	10	Sch. 80 PVC
MW-87D2	10/04/05	121.05	120.55	400.0	-278.95	405.0	-284.0	415.0	-294.0	416.0	-295.0	2	10	Sch. 80 PVC
MW-88D1	03/21/06	120.89	120.17	297.7	-176.81	305.0	-184.1	315.0	-194.1	320.4	-199.5	2	10	Sch. 80 PVC
MW-88D2	03/21/06	120.89	120.05	398.5	-277.61	405.6	-284.7	415.6	-294.7	416.0	-295.1	2	10	BI/SS
MW-89D1	12/19/10			340.0		345.0		355.0		360.0		2	10	BI/SS
MW-89D2	12/19/10			375.0		380.0		390.0		391.0		2	10	BI/SS
MW-90D1	03/28/06	123.31	122.93	222.0	-98.69	238.0	-114.7	243.0	-119.7	245.0	-121.7	1.5	10	BI/SS
MW-90D2	03/28/06	123.29	122.85	262.0	-138.71	267.0	-143.7	272.0	-148.7	280.0	-156.7	1.5	10	BI/SS
MW-92D1	03/11/11			205.0		210.0		220.0		225.0		2	10	BI/SS
MW-92D2	03/11/11			250.0		255.0		265.0		266.0		2	10	BI/SS
MW-93D1	03/03/11			200.0		205.0		215.0		220.0		2	10	BI/SS
MW-93D2	03/03/11			255.0		260.0		270.0		271.0		2	10	BI/SS
VZ-1S	03/15/11			4.0		6.0		8.0		9.0		1.0	10	Sch. 40 PVC
VZ-1D	03/15/11			39.0		41.0		46.0		47.0		1.0	10	Sch. 40 PVC
VZ-2S	02/12/11			4.0		6.0		8.0		8.5		1.0	10	Sch. 40 PVC
VZ-2D	02/12/11			42.0		44.0		49.0		57.0		1.0	10	Sch. 40 PVC
VZ-4S	04/30/11			4.0		6.0		8.0		9.0		1.0	10	Sch. 40 PVC
VZ-4D	04/30/11			41.0		43.0		48.0		50.0		1.0	10	Sch. 40 PVC
VZ-5S	03/11/11			4.0		6.0		8.0		9.0		1.0	10	Sch. 40 PVC

TABLE 3.3

WELL INSTALLATION DETAILS
OPERABLE UNIT-3 BIOSPACE SYSTEM
HOOKER/RUCO SITE, HICKSVILLE, NEW YORK

Well Designation	Date Completed	Ground Surface (ft amsl)	Measuring Point Elevation [1] (ft amsl)	Top of Sandpack (ft bgs)	Top of Sandpack (ft amsl)	Top of Screen (ft bgs)	Top of Screen (ft amsl)	Bottom of Screen (ft bgs)	Bottom of Screen (ft amsl)	Bottom of Sandpack (ft bgs)	Bottom of Sandpack (ft amsl)	Well Diameter (in)	Well Screen Slot Size	Well Material
VZ-5D	03/11/11			39.0		41.0		46.0		47.0		1.0	10	Sch. 40 PVC
VZ-6S	02/26/11			4.0		6.0		8.0		9.0		1.0	10	Sch. 40 PVC
VZ-6D	02/26/11			42.0		44.0		49.0		57.0		1.0	10	Sch. 40 PVC
VZ-10S	01/19/06	121.90	121.81	4.0	117.90	6.0	115.9	8.0	113.9	8.5	113.4	1.0	10	Sch. 40 PVC
VZ-10D	01/19/06	121.90	121.81	49.0	72.90	51.0	70.9	56.0	65.9	60.0	61.9	1.0	10	Sch. 40 PVC
VZ-11S	02/28/06	121.35	120.64	4.0	117.35	6.0	115.4	8.0	113.4	8.5	112.9	1.0	10	Sch. 40 PVC
VZ-11D	02/28/06	121.35	120.60	42.0	79.35	44.0	77.4	49.0	72.4	63.0	58.4	1.0	10	Sch. 40 PVC
VZ-12S	12/05/10			4.0		6.0		8.0		8.5		1.0	10	Sch. 40 PVC
VZ-12D	12/05/10			41.5		43.5		48.5		49.0		1.0	10	Sch. 40 PVC
VZ-14S	10/07/05	121.32	120.97	4.0	117.32	6.0	115.3	8.0	113.3	8.5	112.8	1.0	10	Sch. 40 PVC
VZ-14D	10/07/05	121.32	121.01	47.0	74.32	49.0	72.3	54.0	67.3	69.0	52.3	1.0	10	Sch. 40 PVC
VZ-15S	11/04/05	121.46	121.31	4.0	117.46	6.0	115.5	8.0	113.5	12.5	109.0	1.0	10	Sch. 40 PVC
VZ-15D	11/04/05	121.46	121.32	49.0	72.46	51.0	70.5	56.0	65.5	63.0	58.5	1.0	10	Sch. 40 PVC
VZ-16S	01/23/06	120.42	120.13	4.0	116.42	6.0	114.4	8.0	112.4	8.5	111.9	1.0	10	Sch. 40 PVC
VZ-16D	01/23/06	120.42	120.27	49.0	71.42	51.0	69.4	56.0	64.4	60.0	60.4	1.0	10	Sch. 40 PVC
VZ-17S	12/20/10			4.0		6.0		8.0		8.5		1.0	10	Sch. 40 PVC
VZ-17D	12/20/10			31.0		33.0		38.0		50.0		1.0	10	Sch. 40 PVC

Notes:

(1) Measuring Point is generally top of well riser pipe. Measuring point is marked.
(2) Abandoned
amsl above mean sea level
bgs below ground surface
BI Black Steel Riser
SS Stainless Steel Well Screen
PVC Polyvinyl Chloride

ATTACHMENT I

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE
SPECTRUM ANALYTICAL, INC.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2012
Issued April 01, 2011
Revised June 10, 2011

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Issued in accordance with and pursuant to section 502 Public Health Law of New York State

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SPECTRUM ANALYTICAL INC. FEATURING HANIBAL TECH.
RI DIVISION
175 METRO CENTER BLVD
WARWICK, RI 02886

NY Lab Id No: 11522
EPA Lab Code: RI00907

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 8260C
Acrylonitrile	EPA 8260C
Amines	
2-Nitroaniline	EPA 8270D
3-Nitroaniline	EPA 8270D
4-Chloroaniline	EPA 8270D
4-Nitroaniline	EPA 8270D
Carbazole	EPA 8270D
Diphenylamine	EPA 8270D
Pyridine	EPA 625
	EPA 8270D
Benzidines	
3,3'-Dichlorobenzidine	EPA 625
	EPA 8270D
Benzidine	EPA 625
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 608
	EPA 8081B
4,4'-DDE	EPA 608
	EPA 8081B
4,4'-DDT	EPA 608
	EPA 8081B
Aldrin	EPA 608
	EPA 8081B

Chlorinated Hydrocarbon Pesticides	
alpha-BHC	EPA 608
	EPA 8081B
alpha-Chlordane	EPA 8081B
beta-BHC	EPA 608
	EPA 8081B
Chlordane Total	EPA 608
	EPA 8081B
delta-BHC	EPA 608
	EPA 8081B
Dieldrin	EPA 608
	EPA 8081B
Endosulfan I	EPA 608
	EPA 8081B
Endosulfan II	EPA 608
	EPA 8081B
Endosulfan sulfate	EPA 608
	EPA 8081B
Endrin	EPA 608
	EPA 8081B
Endrin aldehyde	EPA 608
	EPA 8081B
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 608
	EPA 8081B

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Mineral		**Nutrient**	
Chloride	SM 18-21 4500-Cl- E (97)	Nitrate (as N)	EPA 353.2 Rev. 2.0
Fluoride, Total	EPA 300.0 Rev. 2.1	Nitrite (as N)	EPA 300.0 Rev. 2.1
Hardness, Total	EPA 200.7 Rev. 4.4	Orthophosphate (as P)	EPA 300.0 Rev. 2.1
Sulfate (as SO4)	EPA 300.0 Rev. 2.1	Phosphorus, Total	SM 18-21 4500-P E
	SM 15 426 C		
Nitroaromatics and Isophorone		**Phthalate Esters**	
2,4-Dinitrotoluene	EPA 625	Benzyl butyl phthalate	EPA 625
	EPA 8270D		EPA 8270D
2,6-Dinitrotoluene	EPA 625	Bis(2-ethylhexyl) phthalate	EPA 625
	EPA 8270D		EPA 8270D
Isophorone	EPA 625	Diethyl phthalate	EPA 625
	EPA 8270D		EPA 8270D
Nitrobenzene	EPA 625	Dimethyl phthalate	EPA 625
	EPA 8270D		EPA 8270D
Nitrosoamines		Di-n-butyl phthalate	EPA 625
N-Nitrosodimethylamine	EPA 625		EPA 8270D
N-Nitrosodi-n-propylamine	EPA 625	Di-n-octyl phthalate	EPA 625
	EPA 8270D		EPA 8270D
N-Nitrosodiphenylamine	EPA 625	**Polychlorinated Biphenyls**	
	EPA 8270D	PCB-1016	EPA 608
Nutrient			EPA 8082A
Ammonia (as N)	SM 18 4500-NH3 C	PCB-1221	EPA 608
Kjeldahl Nitrogen, Total	SM 18 4500-NH3 C		EPA 8082A
Nitrate (as N)	EPA 300.0 Rev. 2.1	PCB-1232	EPA 608
			EPA 8082A
		PCB-1242	EPA 608

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Polychlorinated Biphenyls	
PCB-1242	EPA 8082A
PCB-1248	EPA 608
	EPA 8082A
PCB-1254	EPA 608
	EPA 8082A
PCB-1260	EPA 608
	EPA 8082A
PCB-1262	EPA 8082A
PCB-1268	EPA 8082A
Polynuclear Aromatics	
Acenaphthene	EPA 625
	EPA 8270D
Acenaphthylene	EPA 625
	EPA 8270D
Anthracene	EPA 625
	EPA 8270D
Benzo(a)anthracene	EPA 625
	EPA 8270D
Benzo(a)pyrene	EPA 625
	EPA 8270D
Benzo(b)fluoranthene	EPA 625
	EPA 8270D
Benzo(ghi)perylene	EPA 625
	EPA 8270D
Benzo(k)fluoranthene	EPA 625

Polynuclear Aromatics	
Benzo(k)fluoranthene	EPA 8270D
Chrysene	EPA 625
	EPA 8270D
Dibenzo(a,h)anthracene	EPA 625
	EPA 8270D
Fluoranthene	EPA 625
	EPA 8270D
Fluorene	EPA 625
	EPA 8270D
Indeno(1,2,3-cd)pyrene	EPA 625
	EPA 8270D
Naphthalene	EPA 625
	EPA 8270D
Phenanthrene	EPA 625
	EPA 8270D
Pyrene	EPA 625
	EPA 8270D
Priority Pollutant Phenols	
2,4,5-Trichlorophenol	EPA 8270D
2,4,6-Trichlorophenol	EPA 625
	EPA 8270D
2,4-Dichlorophenol	EPA 625
	EPA 8270D
2,4-Dimethylphenol	EPA 625
	EPA 8270D

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Priority Pollutant Phenols	
2,4-Dinitrophenol	EPA 625
	EPA 8270D
2-Chlorophenol	EPA 625
	EPA 8270D
2-Methyl-4,6-dinitrophenol	EPA 625
	EPA 8270D
2-Methylphenol	EPA 8270D
2-Nitrophenol	EPA 625
	EPA 8270D
4-Chloro-3-methylphenol	EPA 625
	EPA 8270D
4-Methylphenol	EPA 8270D
4-Nitrophenol	EPA 625
	EPA 8270D
Pentachlorophenol	EPA 625
	EPA 8270D
Phenol	EPA 625
	EPA 8270D
Residue	
Settleable Solids	SM 18-21 2540 F (97)
Solids, Total	SM 18-21 2540B (97)
Solids, Total Dissolved	SM 18-21 2540C (97)
Semi-Volatile Organics	
1,2-Dichlorobenzene, Semi-volatile	EPA 8270D
1,3-Dichlorobenzene, Semi-volatile	EPA 8270D
1,4-Dichlorobenzene, Semi-volatile	EPA 8270D
2-Methylnaphthalene	EPA 8270D
Benzaldehyde	EPA 8270D
Benzoic Acid	EPA 8270D
Benzyl alcohol	EPA 8270D
Caprolactam	EPA 8270D
Dibenzofuran	EPA 8270D
Volatile Aromatics	
1,2,4-Trichlorobenzene, Volatile	EPA 8260C
1,2,4-Trimethylbenzene	EPA 8260C
1,2-Dichlorobenzene	EPA 624
	EPA 8260C
1,3,5-Trimethylbenzene	EPA 8260C
1,3-Dichlorobenzene	EPA 624
	EPA 8260C
1,4-Dichlorobenzene	EPA 624
	EPA 8260C
2-Chlorotoluene	EPA 8260C
4-Chlorotoluene	EPA 8260C
Benzene	EPA 624
	EPA 8260C
Chlorobenzene	EPA 624
	EPA 8260C
Ethyl benzene	EPA 624
	EPA 8260C

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Volatile Aromatics	
Isopropylbenzene	EPA 8260C
Naphthalene, Volatile	EPA 8260C
n-Butylbenzene	EPA 8260C
n-Propylbenzene	EPA 8260C
p-Isopropyltoluene (P-Cymene)	EPA 8260C
sec-Butylbenzene	EPA 8260C
Styrene	EPA 8260C
tert-Butylbenzene	EPA 8260C
Toluene	EPA 624
	EPA 8260C
Total Xylenes	EPA 624
	EPA 8260C
Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 8260C
1,1,1-Trichloroethane	EPA 624
	EPA 8260C
1,1,2,2-Tetrachloroethane	EPA 624
	EPA 8260C
1,1,2-Trichloroethane	EPA 624
	EPA 8260C
1,1-Dichloroethane	EPA 624
	EPA 8260C
1,1-Dichloroethene	EPA 624
	EPA 8260C
1,1-Dichloropropene	EPA 8260C

Volatile Halocarbons	
1,2,3-Trichloropropane	EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 8260B
	EPA 8260C
	EPA 8270D
1,2-Dibromoethane	EPA 8260B
	EPA 8260C
1,2-Dichloroethane	EPA 624
	EPA 8260C
1,2-Dichloropropane	EPA 624
	EPA 8260C
1,3-Dichloropropane	EPA 8260C
2,2-Dichloropropane	EPA 8260C
2-Chloroethylvinyl ether	EPA 624
	EPA 8260C
Bromochloromethane	EPA 8260C
Bromodichloromethane	EPA 624
	EPA 8260C
Bromoform	EPA 624
	EPA 8260C
Bromomethane	EPA 624
	EPA 8260C
Carbon tetrachloride	EPA 624
	EPA 8260C
Chloroethane	EPA 624
	EPA 8260C

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Volatile Halocarbons	
Chloroform	EPA 624
	EPA 8260C
Chloromethane	EPA 624
	EPA 8260C
cis-1,2-Dichloroethene	EPA 8260C
cis-1,3-Dichloropropene	EPA 624
	EPA 8260C
Dibromochloromethane	EPA 624
	EPA 8260C
Dibromomethane	EPA 8260C
Dichlorodifluoromethane	EPA 8260C
Hexachlorobutadiene, Volatile	EPA 8260C
Methylene chloride	EPA 624
	EPA 8260C
Tetrachloroethene	EPA 624
	EPA 8260C
trans-1,2-Dichloroethene	EPA 624
	EPA 8260C
trans-1,3-Dichloropropene	EPA 624
	EPA 8260C
trans-1,4-Dichloro-2-butene	EPA 8260C
Trichloroethene	EPA 624
	EPA 8260C
Trichlorofluoromethane	EPA 624
	EPA 8260C
Volatile Halocarbons	
Vinyl chloride	EPA 624
	EPA 8260C
Volatiles Organics	
1,4-Dioxane	EPA 8260C
2-Butanone (Methylethyl ketone)	EPA 8260C
2-Hexanone	EPA 8260C
4-Methyl-2-Pentanone	EPA 8260C
Acetone	EPA 8260C
Carbon Disulfide	EPA 8260C
Vinyl acetate	EPA 8260C
Wastewater Metals I	
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
Calcium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A

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Wastewater Metals I		**Wastewater Metals II**	
Iron, Total	EPA 200.7 Rev. 4.4	Antimony, Total	EPA 6010C
	EPA 6010C		EPA 6020A
Lead, Total	EPA 200.7 Rev. 4.4	Arsenic, Total	EPA 200.7 Rev. 4.4
	EPA 6010C		EPA 6010C
	EPA 6020A		EPA 6020A
Magnesium, Total	EPA 200.7 Rev. 4.4	Beryllium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C		EPA 6010C
Manganese, Total	EPA 200.7 Rev. 4.4		EPA 6020A
	EPA 6010C	Chromium VI	EPA 7196A
	EPA 6020A		SM 18-19 3500-Cr D
Nickel, Total	EPA 200.7 Rev. 4.4	Mercury, Total	EPA 245.1 Rev. 3.0
	EPA 6010C		EPA 7470A
	EPA 6020A	Selenium, Total	EPA 200.7 Rev. 4.4
Potassium, Total	EPA 200.7 Rev. 4.4		EPA 6010C
	EPA 6010C		EPA 6020A
Silver, Total	EPA 200.7 Rev. 4.4	Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C		EPA 6010C
	EPA 6020A		EPA 6020A
Sodium, Total	EPA 200.7 Rev. 4.4	Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 6010C		EPA 6010C
Wastewater Metals II			EPA 6020A
Aluminum, Total	EPA 200.7 Rev. 4.4	**Wastewater Metals III**	
	EPA 6010C	Cobalt, Total	EPA 200.7 Rev. 4.4
	EPA 6020A		EPA 6010C
Antimony, Total	EPA 200.7 Rev. 4.4		EPA 6020A

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Wastewater Metals III		**Sample Preparation Methods**
Molybdenum, Total	EPA 200.7 Rev. 4.4	EPA 3010A
	EPA 6010C	EPA 3510C
	EPA 6020A	EPA 3520C
Thallium, Total	EPA 200.7 Rev. 4.4	EPA 5030B
	EPA 6010C	SM 18-20 4500-P b.5
	EPA 6020A	SM 18-21 4500-N Org B or C (97)
Tin, Total	EPA 200.7 Rev. 4.4	SM 18-21 4500-NH3 B (97)
	EPA 6010C	
Wastewater Miscellaneous		
Boron, Total	EPA 200.7 Rev. 4.4	
	EPA 6010C	
Bromide	EPA 300.0 Rev. 2.1	
Color	SM 18-21 2120B (01)	
Cyanide, Total	EPA 335.4 Rev. 1.0	
	EPA 9012A	
Hydrogen Ion (pH)	EPA 9040B	
	SM 18-21 4500-H B (00)	
Oil & Grease Total Recoverable (HEM)	EPA 1664A	
Organic Carbon, Total	SM 18-21 5310B (00)	
Phenols	EPA 420.1 Rev. 1978	
Specific Conductance	EPA 120.1 Rev. 1982	
Sulfide (as S)	SM 18-21 4500-S D (00)	
Temperature	SM 18-21 2550B (00)	
Sample Preparation Methods		
	EPA 3005A	

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Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. YIHAI DING
SPECTRUM ANALYTICAL INC. FEATURING HANIBAL TECH.
RI DIVISION
175 METRO CENTER BLVD
WARWICK, RI 02886

NY Lab Id No: 11522
EPA Lab Code: RI00907

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 8260C
Acrylonitrile	EPA 8260C
Amines	
2-Nitroaniline	EPA 8270D
3-Nitroaniline	EPA 8270D
4-Chloroaniline	EPA 8270D
4-Nitroaniline	EPA 8270D
Carbazole	EPA 8270D
Diphenylamine	EPA 8270D
Benzidines	
3,3'-Dichlorobenzidine	EPA 8270D
Characteristic Testing	
Corrosivity	EPA 9040B
Ignitability	EPA 1010
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 8081A
4,4'-DDE	EPA 8081A
4,4'-DDT	EPA 8081A
Aldrin	EPA 8081B
alpha-BHC	EPA 8081B
alpha-Chlordane	EPA 8081B
beta-BHC	EPA 8081B
Chlordane Total	EPA 8081B
delta-BHC	EPA 8081B

Chlorinated Hydrocarbon Pesticides	
Dieldrin	EPA 8081A
Endosulfan I	EPA 8081A
Endosulfan II	EPA 8081A
Endosulfan sulfate	EPA 8081A
Endrin	EPA 8081A
Endrin aldehyde	EPA 8081A
Endrin Ketone	EPA 8081A
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081A
Heptachlor epoxide	EPA 8081A
Lindane	EPA 8081B
Methoxychlor	EPA 8081A
Toxaphene	EPA 8081A
Chlorinated Hydrocarbons	
1,2,3-Trichlorobenzene	EPA 8260C
1,2,4-Trichlorobenzene	EPA 8270D
2-Chloronaphthalene	EPA 8270D
Hexachlorobenzene	EPA 8270D
Hexachlorobutadiene	EPA 8270D
Hexachlorocyclopentadiene	EPA 8270D
Hexachloroethane	EPA 8270D
Haloethers	
4-Bromophenylphenyl ether	EPA 8270D
4-Chlorophenylphenyl ether	EPA 8270D
Bis (2-chloroisopropyl) ether	EPA 8270D

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Haloethers	
Bis(2-chloroethoxy)methane	EPA 8270D
Bis(2-chloroethyl)ether	EPA 8270D
Metals I	
Barium, Total	EPA 6010C
	EPA 6020A
Cadmium, Total	EPA 6010C
	EPA 6020A
Calcium, Total	EPA 6010C
Chromium, Total	EPA 6010C
	EPA 6020A
Copper, Total	EPA 6010C
	EPA 6020A
Iron, Total	EPA 6010C
Lead, Total	EPA 6010C
	EPA 6020A
Magnesium, Total	EPA 6010C
Manganese, Total	EPA 6010C
	EPA 6020A
Nickel, Total	EPA 6010C
	EPA 6020A
Potassium, Total	EPA 6010C
Silver, Total	EPA 6010C
	EPA 6020A
Sodium, Total	EPA 6010C

Metals II	
Aluminum, Total	EPA 6010C
	EPA 6020A
Antimony, Total	EPA 6010C
	EPA 6020A
Arsenic, Total	EPA 6010C
	EPA 6020A
Beryllium, Total	EPA 6010C
	EPA 6020A
Chromium VI	EPA 7196A
Mercury, Total	EPA 7471B
Selenium, Total	EPA 6010C
	EPA 6020A
Vanadium, Total	EPA 6010C
	EPA 6020A
Zinc, Total	EPA 6010C
	EPA 6020A
Metals III	
Cobalt, Total	EPA 6010C
	EPA 6020A
Molybdenum, Total	EPA 6010C
	EPA 6020A
Thallium, Total	EPA 6010C
	EPA 6020A
Miscellaneous	
Cyanide, Total	EPA 9012A

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Miscellaneous	
Hydrogen Ion (pH)	EPA 9040B
	EPA 9045C
Nitroaromatics and Isophorone	
2,4-Dinitrotoluene	EPA 8270D
2,6-Dinitrotoluene	EPA 8270D
Isophorone	EPA 8270D
Nitrobenzene	EPA 8270D
Pyridine	EPA 8270D
Nitrosoamines	
N-Nitrosodi-n-propylamine	EPA 8270D
N-Nitrosodiphenylamine	EPA 8270D
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015 B
Gasoline Range Organics	EPA 8015 B
Phthalate Esters	
Benzyl butyl phthalate	EPA 8270D
Bis(2-ethylhexyl) phthalate	EPA 8270D
Diethyl phthalate	EPA 8270D
Dimethyl phthalate	EPA 8270D
Di-n-butyl phthalate	EPA 8270D
Di-n-octyl phthalate	EPA 8270D
Polychlorinated Biphenyls	
PCB-1016	EPA 8082A

Polychlorinated Biphenyls	
PCB-1221	EPA 8082A
PCB-1232	EPA 8082A
PCB-1242	EPA 8082A
PCB-1248	EPA 8082A
PCB-1254	EPA 8082A
PCB-1260	EPA 8082A
PCB-1262	EPA 8082A
PCB-1268	EPA 8082A
Polynuclear Aromatic Hydrocarbons	
Acenaphthene	EPA 8270D
Acenaphthylene	EPA 8270D
Anthracene	EPA 8270D
Benzo(a)anthracene	EPA 8270D
Benzo(a)pyrene	EPA 8270D
Benzo(b)fluoranthene	EPA 8270D
Benzo(ghi)perylene	EPA 8270D
Benzo(k)fluoranthene	EPA 8270D
Chrysene	EPA 8270D
Dibenzo(a,h)anthracene	EPA 8270D
Fluoranthene	EPA 8270D
Fluorene	EPA 8270D
Indeno(1,2,3-cd)pyrene	EPA 8270D
Naphthalene	EPA 8270D
Phenanthrene	EPA 8270D
Pyrene	EPA 8270D

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Priority Pollutant Phenols		**Volatile Aromatics**	
2,4,5-Trichlorophenol	EPA 8270D	1,2,4-Trichlorobenzene, Volatile	EPA 8260C
2,4,6-Trichlorophenol	EPA 8270D	1,2,4-Trimethylbenzene	EPA 8260C
2,4-Dichlorophenol	EPA 8270D	1,2-Dichlorobenzene	EPA 8260C
2,4-Dimethylphenol	EPA 8270D	1,3,5-Trimethylbenzene	EPA 8260C
2,4-Dinitrophenol	EPA 8270D	1,3-Dichlorobenzene	EPA 8260C
2-Chlorophenol	EPA 8270D	1,4-Dichlorobenzene	EPA 8260C
2-Methyl-4,6-dinitrophenol	EPA 8270D	2-Chlorotoluene	EPA 8260C
2-Methylphenol	EPA 8270D	4-Chlorotoluene	EPA 8260C
2-Nitrophenol	EPA 8270D	Benzene	EPA 8260C
4-Chloro-3-methylphenol	EPA 8270D	Bromobenzene	EPA 8260C
4-Methylphenol	EPA 8270D	Chlorobenzene	EPA 8260C
4-Nitrophenol	EPA 8270D	Ethyl benzene	EPA 8260C
Pentachlorophenol	EPA 8270D	Isopropylbenzene	EPA 8260C
Phenol	EPA 8270D	n-Butylbenzene	EPA 8260C
Semi-Volatile Organics		n-Propylbenzene	EPA 8260C
1,1'-Biphenyl	EPA 8270D	p-Isopropyltoluene (P-Cymene)	EPA 8260C
1,2-Dichlorobenzene, Semi-volatile	EPA 8270D	sec-Butylbenzene	EPA 8260C
1,3-Dichlorobenzene, Semi-volatile	EPA 8270D	Styrene	EPA 8260C
1,4-Dichlorobenzene, Semi-volatile	EPA 8270D	tert-Butylbenzene	EPA 8260C
2-Methylnaphthalene	EPA 8270D	Toluene	EPA 8260C
Benzaldehyde	EPA 8270D	Total Xylenes	EPA 8260C
Benzyl alcohol	EPA 8270D	**Volatile Halocarbons**	
Caprolactam	EPA 8270D	1,1,1,2-Tetrachloroethane	EPA 8260C
Dibenzofuran	EPA 8270D	1,1,1-Trichloroethane	EPA 8260C
		1,1,2,2-Tetrachloroethane	EPA 8260C

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Volatile Halocarbons	
1,1,2-Trichloroethane	EPA 8260C
1,1-Dichloroethane	EPA 8260C
1,1-Dichloroethene	EPA 8260C
1,1-Dichloropropene	EPA 8260C
1,2,3-Trichloropropane	EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 8260C
1,2-Dibromoethane	EPA 8260C
1,2-Dichloroethane	EPA 8260C
1,2-Dichloropropane	EPA 8260C
1,3-Dichloropropane	EPA 8260C
2,2-Dichloropropane	EPA 8260C
Bromochloromethane	EPA 8260C
Bromodichloromethane	EPA 8260C
Bromoform	EPA 8260C
Bromomethane	EPA 8260C
Carbon tetrachloride	EPA 8260C
Chloroethane	EPA 8260C
Chloroform	EPA 8260C
Chloromethane	EPA 8260C
cis-1,2-Dichloroethene	EPA 8260C
cis-1,3-Dichloropropene	EPA 8260C
Dibromochloromethane	EPA 8260C
Dibromomethane	EPA 8260C
Dichlorodifluoromethane	EPA 8260C
Hexachlorobutadiene, Volatile	EPA 8260C
Methylene chloride	EPA 8260C
Tetrachloroethene	EPA 8260C
trans-1,2-Dichloroethene	EPA 8260C
trans-1,3-Dichloropropene	EPA 8260C
Trichloroethene	EPA 8260C
Trichlorofluoromethane	EPA 8260C
Vinyl chloride	EPA 8260C
Volatile Organics	
1,4-Dioxane	EPA 8260C
2-Butanone (Methylethyl ketone)	EPA 8260B
	EPA 8260C
2-Hexanone	EPA 8260C
4-Methyl-2-Pentanone	EPA 8260C
Acetone	EPA 8260C
Carbon Disulfide	EPA 8260C
Methyl tert-butyl ether	EPA 8260C
Vinyl acetate	EPA 8260C
Sample Preparation Methods	
	EPA 1311
	EPA 3050B
	EPA 3060A
	EPA 3540C
	EPA 3541
	EPA 3545
	EPA 3550B

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Sample Preparation Methods

EPA 5035

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