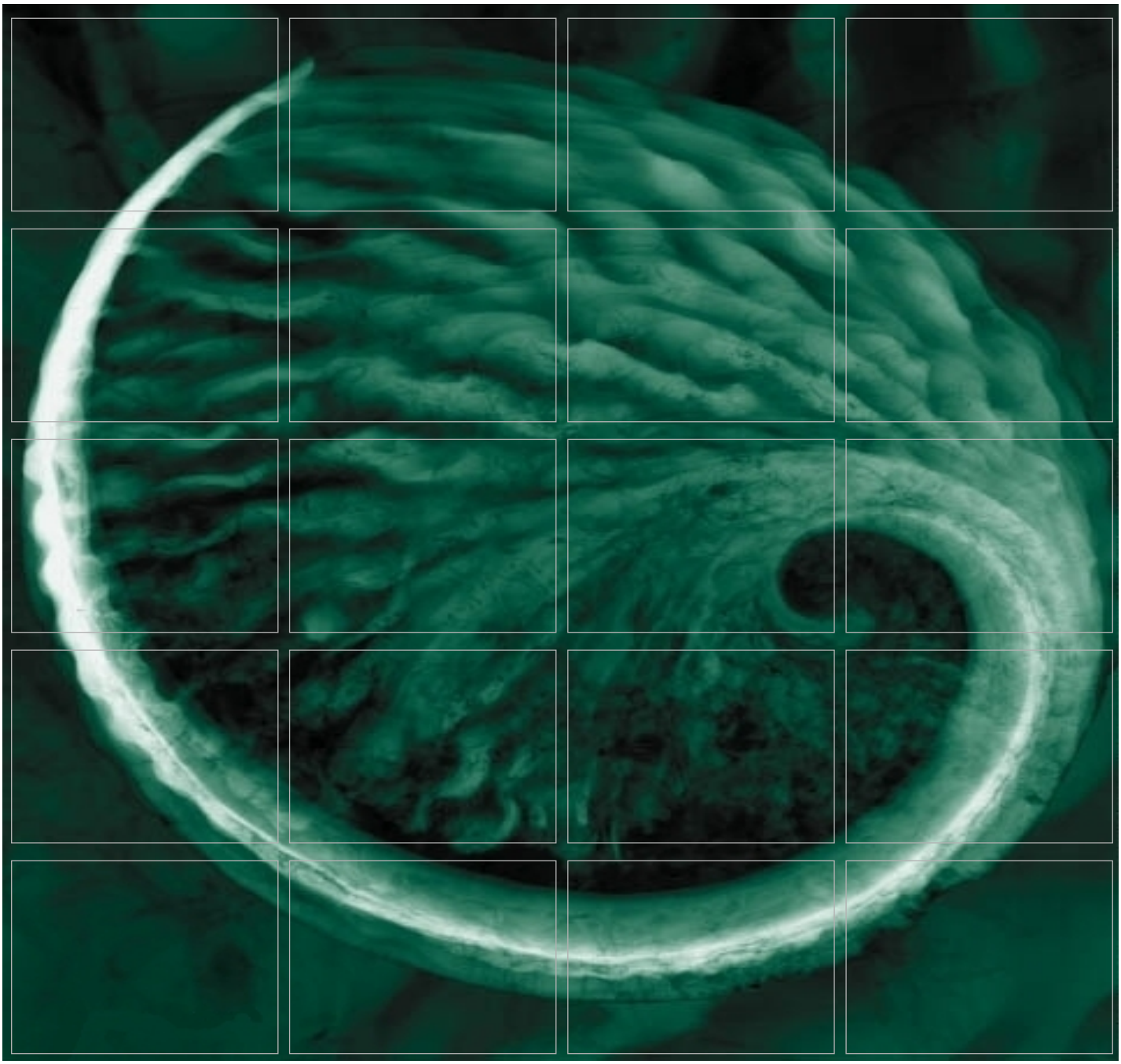

Operable Unit 1
Quality Assurance Project Plan

Fulton Avenue Superfund Site
150 Fulton Avenue
Garden City Park, Nassau County, New York

October 2011

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LIST OF ATTACHMENTS

Introduction

This Quality Assurance Project Plan (QAPP) presents the policies, organization, objectives, functional activities and specific Quality Assurance (QA) and Quality Control (QC) activities designed to achieve the data quality goals associated with the Operable Unit 1 (OU1) Remedial Design (RD) and Remedial Action (RA) to be conducted at the Fulton Avenue Superfund Site (Site) in Garden City Park, New York.

The purpose and objective of the QAPP is to ensure that the analytical results are accurate and representative of field conditions. The analytical methods and QA/QC procedures presented in this QAPP are referenced from, and shall be consistent with the guidelines established in the *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP) and Section 6 (Part B) of *Quality Systems for Environmental Data and Technology Programs - Requirements with guidance for use*, ANSI/ASQ E4 (February 2004).

This QAPP is an integral part of the OU1 Remedial Design (RD) Work Plan. This QAPP is a dynamic document that will be subject to revision as the OU1 RD/RA progresses. Revisions will likely be required to address changes in regulatory requirements or field conditions to ensure the scope of the QAPP is aligned with the needs of the OU1 RD and/or RA, and that data goals are met including the accuracy and representativeness of all analytical results.

QAPP Worksheet #1
Title and Approval Page

Site Name/Project Name: Fulton Avenue Superfund Site
Site Location: 150 Fulton Avenue, Garden City Park, New York

Document Title: Quality Assurance Project Plan, 150 Fulton Avenue Site, 150 Fulton Avenue, Garden City Park, New York

Lead Organization: Genesco Inc.

Preparer's Name and Organizational Affiliation: Chris Wenczel & Eugene Gabay Environmental Resources Management, Inc.

Preparer's Address, Telephone Number, and E-mail Address: 40 Marcus Drive, Suite 200, Melville, New York 11747, 631-756-8900, chris.wenczel@erm.com and eugene.gabay@erm.com

Preparation Date (Day/Month/Year): 24 October 2011

Investigative Organization's Project Manager (Sign and Date)
Chris Wenczel, ERM

Investigative Organization's Project QA Officer (Sign and Date)
Andrew Coenen, ERM

Lead Organization's Project Manager (Sign and Date)
Roger Sisson, Senior Vice President, Corporate Secretary and General Counsel, Genesco Inc.

Approval Authority: United States Environmental Protection Agency (USEPA) (Sign and Date)
Kevin Willis, USEPA Remedial Project Manager

Approval Authority: NYS Department of Environmental Conservation (NYSDEC) (Sign and Date)
Steven M. Scharf, P.E., NYSDEC Remedial Project Manager

QAPP Worksheet #2
QAPP Identifying Information

Site Name/Project Name: Fulton Avenue Superfund Site OU1
Site Location: 150 Fulton Avenue, Garden City Park, New York
Site Number/Code: CERCLA Site No.: NY0000110247
Registry of Inactive Hazardous Waste Disposal Sites in New York State
Site Number 130073
Operable Unit: 1 (OU1)
Contractor Name: Environmental Resources Management, Inc. (ERM)
Contractor Number: ERM Project No.: 0097881

Title: Quality Assurance Project Plan
Revision Number: 00
Revision Date: 25 October 2011

Contract Title: N/A
Work Assignment Number: N/A

1. Identify guidance used to prepare QAPP: Uniform Federal Policy for Quality Assurance Project Plans
2. Identify regulatory program: CERCLA
3. Identify approval entity: USEPA Region II
4. The QAPP is (select one): ☐Generic ☒Project Specific
5. List dates of scoping sessions that were held: See Worksheet #9
6. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date
Sampling and Analysis Plan, Quality Assurance Project Plan, and Health and Safety Plan	11/16/98

7. List organizational partners (stakeholders) and connection with lead organization:

   Roger Sisson, Senior Vice President, Corporate Secretary and General Counsel
   Genesco Inc.

8. List data users:

   USEPA, NYSDEC, New York State Department of Health (NYSDOH), Nassau County Department of Health (NCDH), Genesco Inc. and ERM.

9. 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, See QAPP Identifying Information Matrix Below.

QAPP Worksheet #2
QAPP Identifying Information (Continued)

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QAPP Worksheet #3
Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Kevin Willis	Remedial Project Manager	USEPA Region II	212-637-4252	212-637-4279	Willis.Kevin@epamail.epa.gov	rev 00-01
Steven M. Scharf, P.E.	Remedial Project Manager	NYSDEC	518-402-9620	518-402-9022	sxscharf@gw.dec.state.ny.us	rev 00-02
John Swartwout	Chief Section C, Remedial Bureau A	NYSDEC	518-402-9620	518-402-9022	jbswarto@gw.dec.state.ny.us	rev 00-03
Douglas Fischer	Assistant Regional Counsel New York/Caribbean Superfund Branch Office of Regional Counsel	USEPA	212-637-3180	212-637-3104	Fischer.Douglas@epamail.epa.gov	rev 00-04
Robert Kambic	Assistant U.S. Attorney U.S. Attorney's Office, EDNY	USDOJ	631-715-7852	631-715-7920	robert.kambic@usdoj.gov	rev 00-05
Paul Alexis, Esq.	Partner	Bradley Arant Boult Cummings LLP	615-252-2385	615-252-6385	palexis@babc.com	rev 00-06
Melissa Alexander, Esq.	Partner	Bradley Arant Boult Cummings LLP	615-252-2326	615-252-6326	malexander@babc.com	rev 00-07
James Periconi, Esq.	Principal	Periconi, LLC	212-213-5500	212-213-5030	jpericoni@periconi.com	rev 00-08
Roger Sisson, Esq.	Senior Vice President, Corporate Secretary and General Counsel	Genesco Inc.	615-367-7000	615-367-7073	RSISSON@genesco.com	rev 00-09
James Perazzo	Principal Partner	ERM	631-756-8913	631-756-8901	jim.perazzo@erm.com	rev 00-010
Chris Wenczel	Principal Consultant	ERM	631-756-8920	631-756-8901	chris.wenczel@erm.com	rev 00-011
Mr. Andrew Coenen	Project Chemist	ERM	631-756-8959	631-756-8901	andrew.coenen@erm.com	rev 00-012
Mrs. Tammy McCloskey	Laboratory Project Manager	Accutest	732-355-4562	732-329-3499	tammym@accutest.com	rev 00-013

QAPP Worksheet #4
Project Personnel Sign-Off Sheet

Organization: Genesco Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Roger Sisson	Senior Vice President, Corporate Secretary and General Counsel	615-367-7000		

Organization: ERM

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
James Perazzo	Alternate Project Coordinator/Manager	631-756-8913		
Chris Wenczel	Project Coordinator/Manager	631-756-8920		
Andrew Coenen	Laboratory QA Officer	631-756-8959		
Eugene Gabay	ERM Field Team Leader	631-756-8954		
Justin Bunton	ERM Health and Safety Officer	860 466-8506		

Organization: Accutest Laboratories

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Mrs. Tammy McCloskey	Laboratory Project Manager	732-355-4562		

QAPP Worksheet #5
Project Organizational Chart

See ERM's organizational chart presented as Attachment A.

QAPP Worksheet #6
Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Point of Contact with USEPA Remedial Project Manager and Genesco Inc.	ERM Project Manager	Chris Wenczel	631-756-8920	All documents and information about the project will be forwarded to USEPA by Mr. Wenczel. Mr. Wenczel will have responsibility for all phases of the OU1 Remediation at the site. Mr. Wenczel will delegate project tasks. All materials and information about the project will be forwarded to Genesco by Mr. Wenczel.
General Project Technical Support and QA/QC Review	ERM	James Perazzo Ernest Rossano Andrew Coenen Eugene Gabay John Mohlin, P.E.	See QAPP Worksheet #3	Project team will provide project support and correspondence through e-mail, telephone and personal communications.
Daily Site Progress	ERM Field Team Leader	Eugene Gabay	631-756-8954	Mr. Gabay will be responsible for providing daily and real-time updates from the site to Mr. Wenczel and the USEPA as requested through e-mail, telephone and personal communications.
Liaison with Analytical Laboratory	ERM	Andrew Coenen	631-756-8959	Mr. Coenen will serve as the point of contact for the analytical laboratory and will be responsible for all laboratory and analytical data QA/QC review. All correspondence with the laboratory will be conducted through e-mail or telephone communications.

QAPP Worksheet #7
Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
James Perazzo	Principal-In-Charge	ERM	• provide overall corporate project and technical management, • ensures professional services provided by ERM are cost effective and of the highest quality, • ensures all resources of ERM are available on an as-required basis, • conduct technical discussions for key technical issues with the Respondents, • managerial and technical guidance to ERM Site Manager and other staff, • final review of ERM submittals prior to issue, • primary technical support in technical discussions with state and / or federal agencies.	See professional profile in Attachment B
Chris Wenczel	Project Coordinator	ERM	• all phases of the OU1 Remedial Action at the site. Mr. Wenczel will delegate project tasks.	See professional profile in Attachment B
Andrew Coenen	QA Officer	ERM	• field and laboratory QA/QC oversight. • provides managerial and technical assistance to QA/QC personnel, • provides expertise support function during Agency negotiations, if required • procurement and contracting for analytical laboratory, • overview of laboratory activities, • decides laboratory data corrective action, • performs analytical data assessment and validation, and • assist in preparation of Design Packages.	See professional profile in Attachment B
Eugene Gabay	Field Team Leader	ERM	• coordination and management of all field activities and field QA/QC, • overall technical management of the removal activities, • overall Site Health and Safety performance, • management of project costs and schedules, • technical representation at meetings with the Respondents, • overall preparation and review of work plans, • day-to-day project coordination, facilitation and Site management, • coordination of removal activities, and • monthly progress preparation.	See professional profile in Attachment B
Justin Bunton	ERM Health and Safety Officer	ERM	• responsible for overall Site Health and Safety • overall technical assistance for Health and Safety related issues, and • provides QA/QC review for Health and Safety related documents.	See professional profile in Attachment B

QAPP Worksheet #7
Personnel Responsibilities and Qualifications Table (Continued)

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Mr. John Mohlin, P.E.	Design Team Leader	ERM	• day to day coordination and management of all design team activities • overall design QA/QC, • overall technical management of the design activities, • technical representation at meetings with the Respondents, • overall preparation and review of design documents, and • Design Package preparation.	See professional profile in Attachment B
Mrs. Tammy McCloskey	Laboratory Project Manager	Accutest Laboratories	• coordinate laboratory analyses, • supervise in-house chain-of-custody, • schedule sample analyses, • laboratory QA/QC, • oversee data review, • oversee preparation of analytical reports, • approve final analytical reports prior to submission to ERM, • oversee QA/QC documentation, and • provide technical representation of laboratory QA procedures.	NA

QAPP Worksheet #8
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
Training has been completed on an individual basis to complete the required project specific functions - See note to right and additional information below.						See Professional Profiles provided as Attachment B for specific ERM employee training and certifications. ERM training certificates available upon request

ERM staff and subcontractors who will provide field services at the site will be trained, at a minimum, per the requirements of 29 Code of Federal Regulations (CFR) 1910.120 "Hazardous Waste Operations and Emergency Response" (HAZWOPER), including both the one time 40-hour training and annual 8-hour refreshers. This training includes discussions of potential hazards, exposure limits, and a review of personal protective equipment, emergency procedures, and respirator selection and fit testing. Special service needs for this project such as drilling, MIP and/or Waterloo profiling, laboratory analytical services, underground utility clearance, etc. will be provided by specialty subcontractors for each service area. While many of the aforementioned service disciplines do not necessarily have formal specialized training resulting in some form of a certification, ERM will make diligent inquiry to confirm that only experienced and qualified subcontractor personnel will be performing the work.

QAPP Worksheet #9
Project Scoping Session Participation Sheet

Project Name: **Projected Date(s) of Sampling:** **Project Manager:**	**Site Name:** Fulton Avenue Superfund Site OU1 **Site Location:** 150 Fulton Avenue Garden City Park, New York
Date of Session: **Scoping Session Purpose:**	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role

Comments/Decisions: see below

Action Items: see below

Consensus Decisions: see below

Initial project scoping was completed by ERM in developing the OU1 Remedial Design Work Plan based on the 28 September 2007 Record of Decision (ROD), the Consent Judgment (USEPA Consent Judgment No. CV–09–3917) and attached SOW lodged with the United States District Court for the Eastern District of New York on 10 September 2009 and noticed in the Federal Register / Vol. 74, No. 179, 17 September 2009.

QAPP Worksheet #10
Problem Definition

The problem to be addressed by the project: The Fulton Avenue Property has been identified as a contributing source of Tetrachloroethene (PCE) contamination of groundwater beneath the Site creating PCE-dominant contamination in the Upper Glacial and Magothy aquifers which extend to the southwest, impacting certain public supply wells owned by the Incorporated Village of Garden City (Garden City).
The environmental questions being asked: The pre-remedial design studies are designed to produce data necessary for the design and implementation of the two key components of the OU1 Remedy: 1) In-situ chemical oxidation (ISCO) treatment of the shallower groundwater at and near the 150 Fulton Avenue Property, and 2) extraction and treatment of PCE-impacted groundwater from the deeper Magothy aquifer at locations upgradient of impacted supply wells operated by the Garden City Water District (Well Nos. 13 & 14) followed by subsequent recharge of the treated groundwater to the Upper Glacial aquifer.
Observations from any site reconnaissance reports: The defined extent of areas that require remediation are provided in Section 3 of the RI Report, Sections 1.3 and 2.0 of the FS Report, to be supplemented by the pre-design and design studies outlined in Sections 2.0 and 3.2 of the OU1 RD Work Plan..
A synopsis of secondary data or information from site reports: The secondary data and information has been compiled in the ERM 2005 Remedial Investigation Report.
The possible classes of contaminants and the affected matrices: The investigation identified ground water affected with volatile organic compounds (VOCs), predominantly PCE. The affected matrices will be remediated through ground water extraction, treatment and recharge, and focused in-situ chemical oxidation.
The rationale for inclusion of chemical and non-chemical analyses: The data from groundwater will be used to evaluate the off-site groundwater quality and to track the performance and effectiveness of the remedial components; and groundwater samples will be analyzed as set forth in outlined in Sections 2.1, 2.2, 3.2 & 3.3 of the RD Work Plan to be refined as the OU1 RD progresses. The data from the waste characterization samples will be used to help characterize the investigative/remedial derived waste.
Information concerning various environmental indicators: Remedial objectives, performance standards and specific environmental criteria are set forth in Sections 1.2, 3.2.3 and 3.3.3 of the RD Work Plan.
Project decision conditions ("If..., then..." statements): • The pre-remedial design studies are intended to develop the Site-specific data necessary to fully design and implement the two key components of the OU1 Remedy: 1) ISCO treatment of the shallower groundwater at and near the 150 Fulton Avenue Property, and 2) extraction and treatment of PCE-impacted groundwater from the deeper Magothy aquifer at locations upgradient of impacted supply wells operated by the Garden City Water District (Well Nos. 13 & 14) followed by subsequent recharge of the treated groundwater to the Upper Glacial aquifer. If data gaps still remain at the conclusion of the various planned studies, then additional work scopes to fill those data gaps will be identified and discussed with USEPA on a real-time basis so that if possible, the additional work scopes can be implemented during the same field mobilization. For example, the MIP and Waterloo profiling pre-remedial design investigations are intended to obtain the data necessary to design the ISCO pilot testing program (i.e., locations, depths, etc.). If the real-time results of the MIP and/or Waterloo profiling activities suggest that deeper or additional boring locations are necessary, then additional locations or new target depths would be identified and the rationale discussed with USEPA as soon as possible so that the work could be completed during the same mobilization.

QAPP Worksheet #11
Project Quality Objectives/Systematic Planning Process Statements

Who will use the data? USEPA, and ERM
What will the data be used for? • The data will be used to evaluate the off-site hydrogeologic and groundwater quality conditions at specific locations within the PCE-dominant portion of the plume, design the ISCO and pump and treat remedial components to address the PCE-dominant portion of the plume, and to track the short and long-term performance and effectiveness of the remedial components; and • The data from the waste characterization samples will be used to profile and dispose of investigative/remedial derived waste at appropriately permitted facilities.
What type of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques) Groundwater samples will be analyzed as set forth in outlined in Sections 2.1, 2.2, 3.2 & 3.3 of the RD Work Plan to be refined as the OU1 RD progresses. Details regarding specific analytical protocols and collection methods are presented in latter sections of this QAPP. Waste characterization samples may be analyzed for the presence of hazardous material by the Toxic Characteristic Leaching Potential (TCLP) test according to USEPA Methods for PCB's, organics, inorganics and Toxicity Characteristic (TC) criteria (i.e., corrosivity (pH), ignitability (flashpoint), and reactivity). The required criteria will depend upon the media evaluated and the requirements of the specific off-Site temporary storage and disposal facility (TSDF).
How "good" do the data need to be in order to support the environmental decision? The data needs to meet the QA/QC criteria and the target detection limits for compounds listed in Worksheet #12 and #15 of this QAPP.
How much data are needed? (number of samples for each analytical group, matrix, and concentration) The anticipated criteria for the groundwater sampling are outlined in Sections 2.1, 2.2, 3.2 & 3.3 of the RD Work Plan to be refined as the OU1 RD progresses. The number of waster characterization samples will depend on the volume of waste generated and the requirement of the TSDF.
Where, when, and how should the data be collected/generated? The criteria for the groundwater sampling program are outlined in Sections 2.1, 2.2, 3.2 & 3.3 of the RD Work Plan. The anticipated criteria and frequency for the waste characterization samples is also mentioned in these sections but may change based on the specific criteria requested by the TSDF.
Who will collect and generate the data? It is anticipated that samples will be collected by ERM's field personnel.
How will the data be reported? The results of the data will be provided in the final RA report.
How will the data be archived? Data will be archived in ERM's central file. Electronic copies of all data will be provided to USEPA for archival purposes.

QAPP Worksheet #12
Measurement Performance Criteria Table

Matrix	All
Analytical Group	All
Concentration Level	All

Sampling Procedure1	Analytical Method/SOP2	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)
All	All	Precision – Lab	RPD 40%	Lab Duplicate	A
		Precision – Field	RPD 50% (aqueous) RPD 100% (soil/air)	Blind Field Duplicate	S & A
		Accuracy/Bias Contamination	No target compounds above QL	Method blank, preparation blank, field blank, trip blank	A
		Sensitivity	In house QC criteria	Lab Check Sample	A
		Completeness	90 % acceptable (non-rejected) [3] data	Data Completeness Check	S & A

1. See Worksheet #21 for detailed information.
2. See Worksheet #23 for detailed information.
3. Only data undergoing validation may be rejected.

QAPP Worksheet #13
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
Public supply well sampling and pumpage data Regional hydrogeologic information	Garden City Water Department United States Geological Survey	ERM	Support the OU1 RD and monitoring the performance and effectiveness of the OU1 RA	N/A

QAPP Worksheet #14
Summary of Project Tasks

Sampling Tasks: ▪ Collection of waste characterization samples and ▪ Collection of groundwater monitoring samples.
Analysis Tasks: Accutest Laboratories will perform all laboratory analysis. The specific criteria for each project sampling task are detailed in Worksheet #18.
Quality Control Tasks: QA/QC sampling requirements are outlined in Worksheet #26. All project personnel are expected to review and comply with the QA/QC protocol and guidance presented in this document.
Secondary Data: See Worksheet #13.
Data Management Tasks: After appropriate QA/QC review data will be compiled in an electronic database and presented in the RA Report.
Documentation and Records: All documents will be managed and retained by the ERM project manager in the central project file.
Assessment/Audit Tasks: QA/QC audits will be performed by Project Manager, ERM Principal In Charge and ERM QA Officer.
Data Review Tasks: QA/QC review and validation of data will be managed by ERM QA officer.

QAPP Worksheet #15

Analytical Parameters, Project Action Levels and Laboratory Reporting Limits

Sampling Location: Groundwater Monitoring Samples

Matrix: Aqueous **Concentration Level: Low** **Analytical Group: VOCs**

Target Compound List (TCL) [1]	CAS Number [2]	Project Action Limit (µg/l) [3]	Achievable Laboratory Limits [4]	
			QLs [5] (µg/l)	MDLs [6] (µg/l)
Dichlorodifluoromethane	75-71-8	5	1	0.39
Chloromethane	74-87-3	5	1	0.2
Vinyl chloride	75-01-4	2	1	0.77
Bromomethane	74-83-9	5	1	0.39
Chloroethane	75-00-3	5	1	0.65
Trichlorofluoromethane	75-69-4	5	1	0.43
1,1-Dichloroethene	75-35-4	5	1	0.49
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5	2	0.92
Acetone	67-64-1	50	5	4.6
Carbon disulfide	75-15-0	60	1	0.38
Methyl acetate	79-20-9	5	5	0.54
Methylene chloride	75-09-2	5	2	0.53
trans-1,2-Dichloroethene	156-60-5	5	1	0.18
Methyl tert-butyl ether	1634-04-4	10	1	0.29
1,1-Dichloroethane	75-34-3	5	1	0.089
cis-1,2-Dichloroethene	156-59-2	5	1	0.18
2-Butanone	78-93-3	50	5	1.4
Bromochloromethane	74-97-5	5	1	0.5
Chloroform	67-66-3	7	1	0.18
1,1,1-Trichloroethane	71-55-6	5	1	0.094
Cyclohexane	110-82-7	5	5	0.13
Carbon tetrachloride	56-23-5	5	1	0.53
Benzene	71-43-2	1	1	0.37
1,2-Dichloroethane	107-06-2	0.6	1	0.57
1,4-Dioxane	123-91-1	5	130	18
Trichloroethene	79-01-6	5	1	0.16
Methylcyclohexane	108-87-2	5	1	0.35
1,2-Dichloropropane	78-87-5	1	1	0.5
Bromodichloromethane	75-27-4	50	1	0.14
cis-1,3-Dichloropropene	10061-01-5	0.4	1	0.56
4-Methyl-2-pentanone	108-10-1	5	5	0.5
Toluene	108-88-3	5	1	0.41
trans-1,3-Dichloropropene	10061-02-6	0.4	1	0.15
1,1,2-Trichloroethane	79-00-5	1	1	0.15
Tetrachloroethene	127-18-4	5	1	0.39
2-Hexanone	591-78-6	50	5	0.35
Dibromochloromethane	124-48-1	50	1	0.17
1,2-Dibromoethane	106-93-4	0.0006	1	0.39
Chlorobenzene	108-90-7	5	1	0.74
Ethylbenzene	100-41-4	5	1	0.44
o-Xylene	95-47-6	5	1	0.34
m,p-Xylene	179601-23-1	5	1	0.76

QAPP Worksheet #15

Analytical Parameters, Project Action Levels and Laboratory Reporting Limits (Continued)

Sampling Location: Groundwater Monitoring Samples

Matrix: Aqueous **Concentration Level: Low** **Analytical Group: VOCs**

Target Compound List (TCL) [1]	CAS Number [2]	Project Action Limit (µg/l) [3]	Achievable Laboratory Limits [4]	
Styrene	100-42-5	5	1	0.069
Bromoform	75-25-2	50	1	0.52
Isopropylbenzene	98-82-8	5	1	0.64
1,1,2,2-Tetrachloroethane	79-34-5	5	1	0.11
1,3-Dichlorobenzene	541-73-1	3	1	0.32
1,4-Dichlorobenzene	106-46-7	3	1	0.45
1,2-Dichlorobenzene	95-50-1	3	1	0.34
1,2-Dibromo-3-chloropropane	96-12-8	0.04	5	0.62
1,2,4-Trichlorobenzene	120-82-1	5	2	0.13
1,2,3-Trichlorobenzene	87-61-6	5	2	0.14

1. Target Compound List (TCL) from Multi-Media, Multi-Concentration Organics Analysis, SOM01.2, Exhibit C, 1.0.
2. Chemical Abstracts Service (CAS) Registry Number.
3. New York State Ambient Ground Water Quality Standards and Guidance Values (AWGS) as listed in TOGS 1.1.1 (June 1998) and in 6 NYCRR 703.5.
4. As per Accutest Laboratories, 2235 Route 130, Dayton, New Jersey 08810.
5. QL – Quantitation Limit
6. MDL – Method Detection Limit.

Sampling Location: Investigative Derived Waste (IDW)

Matrix: Soil **Concentration Level: Low** **Analytical Group: TCLP**

Compound List [1]	CAS Number [2]	Project Action Limit (mg/l) [3]	Achievable Laboratory Limits [4]	
			QLs [5] (mg/l)	MDLs [6] (mg/l)
Benzene	71-43-2	0.5 (D018)	0.005	0.0012
2-Butanone (MEK)	78-93-3	200 (D035)	0.1	0.0081
Carbon tetrachloride	56-23-5	0.5 (D019)	0.005	0.0013
Chlorobenzene	108-90-7	100 (D021)	0.005	0.0019
Chloroform	67-66-3	6 (D022)	0.005	0.0012
1,4-Dichlorobenzene	106-46-7	7.5 (D027)	0.005	0.0014
1,2-Dichloroethane	107-06-2	0.5 (D028)	0.005	0.0017
1,1-Dichloroethene	75-35-4	0.7 (D029)	0.005	0.002
Tetrachloroethene	127-18-4	0.7 (D039)	0.005	0.0013
Trichloroethene	79-01-6	0.5 (D040)	0.005	0.0012
Vinyl chloride	75-01-4	0.2 (D043)	0.025	0.0022

1. From EPA's Toxicity Characteristic Leaching Procedure (TCLP).
2. Chemical Abstracts Service (CAS) Registry Number.
3. Maximum Concentration of Contaminants for Toxicity Characteristic (Table 1, D List). EPA Hazardous Waste code in parenthesis.
4. As per Accutest Laboratories, 2235 Route 130, Dayton, New Jersey 08810.
5. QL – Quantitation Limit
6. MDL – Method Detection Limit.

QAPP Worksheet #16
Project Schedule

See the OU1 RD and OU1 RA project schedules presented in the OU1 RD Work Plan as Figures 10 & 11.

QAPP Worksheet #17
Sampling Design and Rationale

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

Pre-design Comprehensive Groundwater Sampling Event: A total of 40 groundwater samples will be collected from wells located within the footprint of the PCE-dominant portion of the groundwater plume to get an updated snapshot of groundwater levels and quality conditions from the Fulton Property to the multi-level wells on the Garden City Country Club Golf Course. The water level and sample analytical data will be used to evaluate current groundwater flow and quality conditions within the footprint of the PCE-dominant portion of the groundwater plume for design of the ISCO and the groundwater pump and treatment components of the OU1 RA. If necessary, adjustments to the ISCO pre-design investigation will be affected based on the current distribution of VOCs in groundwater in close proximity to the Fulton Avenue Property. The 40 groundwater samples (plus appropriate QA/QC samples) will be collected from multi-level wells MWs 26 & 27 (MWs 26A-H & 27A-H), and the following conventional wells: MWs 15A, 15B, 21A, 21B, 21C, 23A, 23B, 23C, 23D, and GCP 01, 01D, 04, 08, 09, 15S, 17S, 17D, 18S, 18D and 19S plus remaining wells in the vicinity of the Soil IRM Area (wells VOWs 1D, 3D, 4D & VEW-1). All groundwater samples will be analyzed for TCL VOCs using USEPA Method 8260B.

Pre-remedial ISCO Characterization Studies: Effective implementation of in situ remediation technologies (e.g., chemical oxidation) requires a detailed understanding of the three-dimensional (3D) distribution (i.e., architecture) of contaminant mass in the subsurface. In situ remedial technologies require direct contact of a stoichiometrically appropriate amount of the remedial additive with the contaminants. Typically, the distribution of chlorinated solvents in the subsurface is complex and concentration gradients are steep. Therefore, the success and efficiency of a remediation are enhanced when the distribution of the most contaminated zones (i.e., source zones and plume cores) is accurately defined. Therefore, it is necessary to effectively define the source area and/or plume architecture to an appropriate scale to enable successful remediation. Accordingly, a pre-remedial investigation will use high resolution techniques to identify and characterize subsurface groundwater intervals at, and near the Fulton Property where higher concentrations of PCE reside. This information will be used to design a targeted ISCO treatment program that makes the most of reducing PCE concentrations in shallow groundwater before it migrates vertically to and into the Magothy Aquifer. The high resolution characterization will entail: advancement of 16 Membrane Interface Probe (MIP) borings on and immediately downgradient of the Fulton Property to a depth of approximately 130 feet (i.e., approximate depth of the Upper Glacial-Magothy boundary). The MIP boring locations were placed conservatively on or immediately downgradient of the Fulton Property to ensure the highest potential for identifying zones wherein higher concentrations of PCE reside. Data generated from the MIP logs will be used to create real-time plan view and cross-sectional diagrams of the VOC distribution in the subsurface to aid in data interpretation, and support the dynamic decision-making process which will subsequently included installation of eight soil borings and a WaterlooAPS groundwater profiling tool will be deployed to collect up to 10 discrete-interval groundwater samples per boring for laboratory analysis of VOCs by EPA Method 8260B.

Continued Groundwater Monitoring: The SOW requires a continuation of the groundwater monitoring program currently being performed under the Order at a yet to be determined frequency to monitor groundwater quality immediately upgradient and downgradient of Village of Garden City Public Supply Well Nos. 13 & 14. This Continued sampling activity will occur at a frequency yet to be determined but will cover the interim period of time between November 2011 and implementation of the OU1 RA components at which time this monitoring would be replaced by short- term performance monitoring followed by long-term effectiveness monitoring. The results thereof, in conjunction with the existing data set will be used to select and propose the frequency of the Continued interim sampling to USEPA. Each Continued sampling event will involve the collection 19 groundwater samples (plus appropriate QA/QC samples) will be collected from multi-level wells MWs 26 & 27 (MWs 26A-H & 27A-H), and the following conventional wells: MWs 21A, 21B & 21C. All groundwater samples will be analyzed for TCL VOCs using USEPA Method 8260B.

Short-Term Performance and Long-Term Effectiveness Groundwater Monitoring: Short-term performance and long-term remedial effectiveness monitoring, groundwater monitoring would be conducted for the duration of each OU1 RA component. Those proposed programs have not yet been designed but will be as part of the OU1 RD as part of the Operations, Maintenance & Monitoring Plan (OM&M) Plan. Examples of those programs are presented in Section 3.5.1 of the OU1 RD Work Plan.

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 to map or Worksheet #18 for details]: These details are provided in other sections of this document. Please refer to Worksheet #2 for the QAPP Identifying Information and in Worksheet #18.

QAPP Worksheet #18
Sampling Locations and Methods/SOP Requirements Table

Sampling Location	Matrix	Depth (feet)	Analytical Group	Analytical Method	Number of Samples 1	Sampling SOP Reference 2	Rationale for Sampling Location
Groundwater Monitoring Samples	Aqueous	Multiple [3]	VOCs	8260B	Number and locations of samples to be determined based on the specific groundwater sampling activity	SOPs 1, 2, 3, 4, 5, 6, 9, 10 & 11	See Worksheet #17
Investigative Derived Waste Samples	Soil	N/A	TCLP VOCs	1311/8260B	1 sample (estimated; may change based on volume of soil generated and specific requirements of TSDF. Collection of QA/QC samples for waste characterization purposes is not anticipated.	SOP-12	See Worksheet #17

1. QA/QC samples collected at the frequency specified on Worksheet #20.
2. See Worksheet #21 for additional information.
3. Each well has a specified depth for sample collection.

QAPP Worksheet #19
Analytical SOP Requirements Table

Sample Location	Matrix	Analytical Group	Preparation and Analytical Method/ SOP Reference [1]	Containers (number, size, and type)	Preservation Requirements	Maximum Holding Time (preparation/ analysis) [2]
Groundwater Monitoring Samples	Aqueous	VOCs	EPO5030-03 EMS8260-19	3 – 40 ml glass VOA vials	Cool 4°C, pH<2 (HCl)	NA / 10 days
Investigative Derived Waste Samples	Soil	TCLP VOCs	EPO5030-03 EMS8260-19	1 – 8 oz. glass jar	Cool, 4°C	7 / 10 days

1. See Worksheet #23 for additional information.
2. New York State Analytical Services Protocol (NYS ASP) holding times and are from date of sample receipt.

QAPP Worksheet #20
Field Quality Control Sample Summary Table

Sample Location	Matrix	Analytical Group	Analytical and Preparation SOP Reference1	No. of Sampling Locations	No. of Blind Field Duplicate Samples	No. of MS/MSD Pairs	No. of Field Blanks	No. of Trip. Blanks	No. of PT Samples	Total No. of Samples to Lab
Groundwater Monitoring Samples	Aqueous	VOCs	EPO5030-03 EMS8260-19	See Worksheet #18	1 minimum frequency of 1 out of every 20 samples.	1 minimum frequency of 1 out of every 20 samples.	To be determined. Minimum frequency of 1 per each sample collection event	To be determined. Minimum frequency of 1 per each sample collection event	None	>250
Investigative Derived Waste Samples	Soil	TCLP VOCs	EPO5030-03 EMS8260-19	N/A	Collection of a field duplicate is not anticipated.	Collection of a MS/MSD pair is not anticipated	Collection of a field blank is not anticipated	None	None	To be determined.

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

BLIND FIELD DUPLICATES

Blind field duplicate samples are two (or more) field samples taken at the same time in the same location. They are intended to represent the same population and are taken through all steps of the analytical procedure in an identical manner. These samples are used to assess precision of the entire data collection activity, including sampling, analysis, and site heterogeneity. One of the samples is given identification such that the laboratory does not know the true location of the sample. Blind field duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and are treated in an identical manner during storage, transportation, and analysis. The Field Sampling Manager shall assign to the sample containers a unique identification number in the field. Specific locations should be designated for collection of Blind field duplicate samples prior to the beginning of sample collection. A minimum of one Blind field duplicate sample shall be included for every 20 field samples per matrix and evaluated as detailed on Worksheet #28.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE

The matrix spike (MS) and matrix spike duplicate (MSD) is an aliquot of sample spiked with known concentrations of all target analytes. The spiking occurs prior to sample preparation and analysis. Each analyte in the MS and MSD shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. The MS/MSDs are used to document potential matrix effects. A minimum of one MS and one MSD shall be analyzed for every 20 samples. The performance of the MS and MSD is evaluated as detailed on Worksheet #28.

QAPP Worksheet #20
Field Quality Control Sample Summary Table (Continued)

FIELD BLANK
The field blank consists of American Society for Testing and Materials (ASTM) Type II reagent grade or organic-free water poured into appropriate sample containers at the sampling site (in the same vicinity as the associated environmental samples). It is handled like an environmental sample and transported to the laboratory for analysis for all laboratory analytes requested for the environmental samples. Field blanks are used to assess the potential introduction of contaminants from surrounding sources of various COCs to the samples during sample collection. A field blank shall be collected for each sampling event where the potential for introduction of contaminants from surrounding sources exist. The decision whether to collect a field blank will be made by the Field Sampling Manager with the written concurrence of the Quality Assurance Manager. Field blank samples shall be collected downwind of possible sources of COCs. Results associated with a contaminated blank shall be flagged accordingly. Field blanks will be evaluated as detailed on Worksheet #28.

EQUIPMENT BLANK
An equipment blank is a sample of ASTM Type II reagent grade water or organic-free water poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis. These may also be called rinse blanks or rinsate blanks. In instances where dedicated sampling equipment is used for sample collection, equipment blanks will not be collected. In these instances, field blanks will be used to assess field QC procedures. Equipment blanks are used to assess the effectiveness of equipment decontamination procedures. Equipment blanks shall be collected immediately after the equipment has been decontaminated after each sampling event, as appropriate, or at a minimum frequency of two per week. The equipment blank samples shall be analyzed for all laboratory analytes requested for the environmental samples collected at the site. Results associated with a contaminated blank shall be qualified accordingly.

TRIP BLANK
The trip blank consists of a VOC sample vial filled in the laboratory by the laboratory with ASTM Type II reagent grade or organic-free water, transported to the sampling site, handled like an environmental sample and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are analyzed for VOCs only. Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. Each cooler of samples sent to the laboratory for analysis containing VOC samples shall contain a trip blank. Trip blanks will be evaluated as detailed on Worksheet #28.

PROFICIENCY TESTING (PT) SAMPLES
PT samples will not be analyzed for this project.

QAPP Worksheet #21
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
SOP-1	Water Level Measurement Procedures	ERM	N/A	☐	
SOP-2	Groundwater Sampling Procedures	ERM	N/A	☐	
SOP-3	Field Blanks	ERM	N/A	☐	
SOP-4	Trip Blanks	ERM	N/A	☐	
SOP-5	Membrane Interface Probe (MIP) Procedures	ERM	N/A	☐	
SOP-6	Waterloo Vertical Profile Boring with Groundwater Sampling Procedures	ERM	N/A	☐	
SOP--7	Pump Test Procedures	ERM	N/A	☐	
SOP-8	Geologic Boring Drilling Procedures	ERM	N/A	☐	
SOP-9	Temporary Well Installation and Sampling Procedure	ERM	N/A	☐	
SOP-10	Potable Water Blanks	ERM	N/A	☐	
SOP-11	Decontamination Procedures	ERM	N/A	☐	
SOP-12	Investigative Derived Waste	ERM	N/A	☐	

QAPP Worksheet #22
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]
Photo Ionization Detector (PID) MinRAe 2000 or equivalent	2 point calibration with isobutylene and zero gas	Cleaning as required and replacement of consumable filters. All maintenance to be performed by equipment rental facility	Test operation of unit comparable to a known calibration standard gas	Condition and operation of unit will be inspected before each use	Daily, before each use	+/- 5 NTU (assumes low range calibration w/ 100 NTU or less standards)	Contact equipment rental firm	Field Team Leader	N/A, reference manufacturer's instructions.
Turbidity Meter Lamotte 2020 or equivalent	2 point calibration or as specified by the manufacturers instructions	All maintenance to be performed by equipment rental facility	Test operation of unit comparable to a known calibration standard	Condition and operation of unit will be inspected before each use	Daily, before each use	+/- 5 NTU (assumes low range calibration w/ 100 NTU or less standards)	Contact equipment rental firm	Field Team Leader	N/A, reference manufacturer's instructions.
Water Quality Instrument dissolved oxygen, temperature, conductivity, pH and oxidation-reduction potential (ORP) YSI Model 600 or equivalent	Calibrate with rental facility supplied standard(s)	All maintenance to be performed by equipment rental facility	Test operation of unit comparable to a known calibration standard	Condition and operation of unit will be inspected before each use	Daily, before each use	+/- 0.03 mg/l for DO, +/- 0.1 pH unit, +/- 0.03% for conductivity, +/- 0.15 C for temp, +/- 1 mv for ORP	Contact equipment rental firm	Field Team Leader	N/A, reference manufacturer's instructions.
Membrane Interface Probe	Response tests conducted before and after each hole.	As needed	Field test in accordance with Geoprobe and Subcontractor's SOP.	Various diagnostics (including flow, pressure, continuity and resistivity) evaluated throughout MIP investigation.	Ongoing	Response Tests: ECD minimum response = 100 mV, PID minimum response = 2 V. Other variable tolerances listed in SOP	Cleaning, repairing and redoing response tests	MIP Chemist	Stone SOP10.12.1
Waterloo Profiler Hach DO Meter	40ml VOA vial with water saturated air.	Stored dry. Chip replacement annually.	Field test in accordance with the manual	Visually inspect probes for cleanliness and wear.	Once a week.	DO within 5%	Cleaning and recalibration	Field staff	See equipment manual

QAPP Worksheet #22
Field Equipment Calibration, Maintenance, Testing, and Inspection Table (Continued)

Waterloo Profiler Hach DO Meter	40ml VOA vial with water saturated air.	Stored dry. Chip replacement annually.	Field test in accordance with the manual	Visually inspect probes for cleanliness and wear.	Once a week.	DO within 5%	Cleaning and recalibration	Field staff	See equipment manual
Waterloo Profiler KPRO Data Acquisition	Pressure Transducer is calibrated with a graduated cylinder of with 1' of water and a 5' measuring stick. The calibration slope is calculated by the data acquisition software.	Replace transducer as needed. Backup units are always included with the equipment and can be changed out within minutes.	Field test in accordance with Subcontractor's SOP.	None.	Once a week.	Pressure slope should be between 22 and 28.	Change transducer and recalibrate.	Field staff	Stone SOP 10.5.3
Waterloo Profiler KPRO Data Acquisition	Flow Meter is calibrated using a graduated cylinder. The calibration slope is calculated by the data acquisition software.	Replace flow meter as needed. Can be sent to the manufacturer for repair. Backup units are always included with the equipment and can be changed out within minutes	Field test in accordance with Subcontractor's SOP.	Check that internal wheel is turning freely.	Once a week.	Flow slope should be between 32 and 38.	Change flow meter and recalibrate.	Field staff	Stone SOP 10.5.3
Waterloo Profiler KPRO Data Acquisition	Depth measurement - String Potentiometer is calibrated with a 5' measuring stick. The calibration slope is calculated by the data acquisition software.	Replace cables and / or string potentiometer as needed. Can be sent to the manufacturer for repair. Backup units are always included with the equipment and can be changed within minutes.	Field test in accordance with Subcontractor's SOP.	Visually inspect data cables and plugs.	Once a week.	The data acquisition software calculates a slope based on the calibration. String pot slope should be between 1.6 and 1.8.	Change string pot and recalibrate.	Field staff	Stone SOP 10.5.3

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

QAPP Worksheet #22
Field Equipment Calibration, Maintenance, Testing, and Inspection Table (Continued)

FIELD INSTRUMENT PREVENTATIVE MAINTENANCE

Preventative maintenance of field instruments will include cleaning after each use and replacement of consumable components such as used filters. Field instruments will also be examined prior to each mobilization for field activities to identify maintenance issues. If maintenance issues exist, maintenance will be performed by the equipment rental facility. The equipment rental facility will be responsible for providing a timely replacement for any malfunctioning equipment.

CALIBRATION PROCEDURES AND FREQUENCY

Before a field instrument is used, the calibration will be verified using standard reference materials. The calibration verification may range from a single point to multiple points. The concentration of the standard, reference identification number, instrument response, instrument identification number, date, and time will be recorded on the daily instrument calibration log and referenced in the site field book. The calibration verification will be performed at least daily, or more frequently as warranted by field conditions. Instruments which do not meet minimum requirements for calibration will not be used and will be replaced by a properly calibrated instrument. It is anticipated that all field instruments which will require calibration will be provided by an equipment rental vendor. The specific model of the instrument provided may vary and the manufacturer's calibration and maintenance instructions should be referenced.

QAPP Worksheet #23
Analytical SOP References Table

Analytical Group	Matrix	Analytical SOP Title	Analytical SOP Document Number	Analytical SOP Revision Number	Analytical SOP Revision Date	Organization Performing Analysis	Definitive or Screening Data	Modified for Project Work?
VOCs	Aqueous	Method 5030A: Purge and Trap	EPO5030-03	03	08/06/03	Accutest	Definitive	No
		Analytical Methods for the Analysis of GC/MS Volatile Samples – 8260B	EMS8260-19	19	09/11/09	Accutest	Definitive	No
TCLP VOCs	Soil	Method 5030A: Purge and Trap	EPO5030-03	03	08/06/03	Accutest	Definitive	No
		Analytical Methods for the Analysis of GC/MS Volatile Samples – 8260B	EMS8260-19	19	09/11/09	Accutest	Definitive	No

QAPP Worksheet #24
Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA [1]	SOP Reference [2]
GC/MS	Tune	Every 12 Hrs.	Method Specifications	Re-tune	Analyst	EMS8260-19
	Initial Calibration	When CCV Fails	± 30% RSD; R > 0.99 or Grand Mean > 30%	Re-Calibrate	Analyst	
	CCV	12 Hours	± 25% RSD; R >0.99 or Grand Mean > 30%	Re-Calibrate	Analyst	

1. Each instrument has a different analyst.
2. See Worksheet #23 for additional information.

QAPP Worksheet #25

Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person [1]	SOP Reference[2]
HP5973, HP5975 & HP5970	Change Injection Liners		Daily	N/A	Replace or Clean	Analyst	EQA036-02
	Column Ferrules		As Required	N/A	Replace	Analyst	EQA036-02
	Injection Port Disc		As Needed	N/A	Clean as Needed	Analyst	EQA036-02
	Injection Port Wellman Assembly		As Needed	N/A	Replace as Needed	Analyst	EQA036-02
	Columns		As Needed	meets calibration criteria	Trim column or replace	Analyst	EQA036-02
	FID Jet		As Needed	N/A	Clean	Analyst	EQA036-02
	MS Source Parts		As Needed	N/A	Clean	Analyst	EQA036-02
	Purge Tubes		Daily & as Needed	N/A	Rinse	Analyst	EQA036-02
	Traps		Daily & as Needed	N/A	Clean / Replace	Analyst	EQA036-02
	Transfer Lines		As Needed	N/A	Rinse	Analyst	EQA036-02
	Syringe		Daily & as Needed	N/A	Clean	Analyst	EQA036-02
HP 5970 only	Jet Separator		As Needed	N/A	Clean as Needed	Analyst	EQA036-02

1. Each instrument has a different analyst.
2. See Worksheet #23 for additional information.

QAPP Worksheet #26
Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Eugene Gabay / ERM
Sample Packaging (Personnel/Organization): Eugene Gabay / ERM
Coordination of Shipment (Personnel/Organization): Eugene Gabay / ERM
Type of Shipment/Carrier: Priority Overnight / Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodian / Accutest Laboratories (Dayton, New Jersey)
Sample Custody and Storage (Personnel/Organization): Sample Custodian / Accutest Laboratories (Dayton, New Jersey)
Sample Preparation (Personnel/Organization): Individual Department Heads / Accutest Laboratories (Dayton, New Jersey)
Sample Determinative Analysis (Personnel/Organization): Project Manager – Accutest Laboratories (Dayton, New Jersey)
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Samples collected in the field will be preserved as specified in Worksheet #19 and placed in a chilled cooler for priority overnight shipment to the analytical laboratory. It is the responsibility of the sample collection personnel to maintain appropriate custody of the cooler, ensure samples are packed appropriately to prevent breakage and ensure that the samples are preserved appropriately (e.g., chilled on ice). If special circumstances arise and the samples cannot be shipped the same day of sample collection, it is the sampler's responsibility to maintain appropriate custody and the temperature of the cooler until the samples are shipped the next day. Sample holding times and preservation methods are presented in Table #19.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): See Worksheet #19
Biological Sample Storage (No. of days from sample collection): N/A
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodian / Accutest Laboratories (Dayton, New Jersey)
Number of Days from Analysis: 1 month from submission of the hard copy report to ERM unless otherwise requested.

QAPP Worksheet #27
Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):

The following documentation procedures will be used during sampling and analysis to provide custody control during transfer of samples from collection through storage. A sample is defined as being under a person's custody if any of the following conditions exist: 1) it is in their possession, 2) it is in their view, after being in their possession, 3) it was in their possession and they locked it up, or 4) it is in a designated secure area. Recordkeeping documentation will include the use of the following:

- a field logbook (bound, with numbered pages) to document sampling activities in the field,
- labels to identify individual samples,
- and- chain-of-custody forms to document the analyses to be performed

In the field the sampler will record in the field logbook the following information for each sample collected:

- sample identification,
- sample matrix,
- name of the sampler,
- sample location,
- sample time and date,
- additional pertinent data,
- analysis to be conducted,
- sampling method,
- sample appearance (e.g., color, turbidity),
- preservative (if required),
- number of sample bottles an types, and- weather conditions

Samples will be packaged in a manner to prevent breakage of sample containers in a pre-chilled cooler. Custody of the samples and cooler will be the responsibility of the sampling personnel. Samples will be picked up by an Accutest courier or shipped via Federal Express Priority Overnight service to the analytical laboratory the same day samples are collected.

Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal): Each sample or group of samples shipped to the laboratory for analysis will be given a unique identification number. The laboratory sample custodian will record the client name, number of samples and date of receipt of the samples. The remaining sample aliquots not used by the laboratory for analysis will be archived for a period of 30 days. After the archive period has passed the sample will be disposed of by the laboratory unless a request to hold the sample is made by ERM.

QAPP Worksheet #27
Sample Custody Requirements (Continued)

Sample Identification Procedures: Each sample collected will be designated by an alpha-numeric code that will identify the type of sampling location and a specific sample designation (identifier). Location types will be identified by a two-letter code. Groundwater samples collected from various existing and future groundwater monitoring wells. Samples collected for waste characterization will begin with "WC". Samples collected from the treatment system Influent will be labeled "IN". For example sample nomenclature for monitoring well samples, waste characterization samples and treatment system samples will be assigned as indicated in the following examples:

MW-1A = Monitoring Well Sample-Well ID,
WC-S-01 = Waste Characterization Sample-Soil-Sequential Sample Number (date sample was collected) and
WC-A-01 = Waste Characterization Sample-Aqueous-Sequential Sample Number (date sample was collected).

In the case of QC samples such as field blanks, trip blanks and blind field duplicate samples, six digits will follow FB, TB and DUP respectively to represent the date (e.g., FB (050107) would represent a field blank collected on 01 April 2007). For matrix spike/matrix spike duplicate samples, MS/MSD will be added following the applicable sample identification.

Chain-of-custody Procedures: The sampling crew shall maintain chain-of-custody records for all field and field QC samples.

The following information concerning the sample shall be documented on the chain of custody form:

- Unique sample identification for each container,
- Date and time of sample collection,
- Source of sample (including name, location, and sample type),
- Designation of MS/MSD;
- Preservative used;
- Analyses required;
- Name of collector(s);
- Serial numbers of custody seals and transportation cases (if used);
- Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories; and
- Bill of lading or transporter tracking number (if applicable).

QAPP Worksheet #28
QC Samples Table – Aqueous VOCs

Matrix **Analytical Group**	Aqueous VOCs		**Sampler's Name**	To Be Determined	
Concentration Level	Low		**Field Sampling Organization**	ERM	
Sampling SOP	SOPs 1, 2, 3, 4, 5, 6, 9, 10 & 11		**Analytical Organization**	Accutest Laboratories	
Analytical Method/ SOP Reference	8260B / EMS8260-19		**No. of Sample Locations**	To Be Determined By Specific Sampling Activity	
QC Sample:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**
Method Blank	1 / day (12 Hr BFB)	No targets above QL	If no gross detections, qualify with a B. For gross detections reanalyze samples.	Tammy McCloskey (Accutest)	Accuracy/Bias-Contamination
Surrogates	2 per sample	Recovery must fall within in-house QC criteria [1]	Reanalyze sample in order to determine matrix effect.	Tammy McCloskey (Accutest)	Accuracy/Bias
Lab Check Sample	1 / 20 samples	Recovery must fall within in-house QC criteria [1]	Reanalyze if gross exceedances.	Tammy McCloskey (Accutest)	Laboratory Accuracy
Blind field duplicate	1 / 20 samples	Relative percent difference (RPD) 50%	Qualify data during validation process.	Andrew Coenen (ERM)	Precision / Reproducibility
Matrix Spike / Matrix Spike Duplicate Pair	1 / 20 samples	Recovery must fall within in-house QC criteria [1]	Qualify data during validation process.	Andrew Coenen (ERM)	Accuracy/Bias
Field Blank Trip Blank	1 / day 1 / shipment of VOCs	Monitor for targets	Qualify data during validation process.	Andrew Coenen (ERM)	Contamination

1. In house QC criteria subject to change through out the project. Will be monitored during the validation process.

QAPP Worksheet #28
QC Samples Table - Soil TCLP VOCs

Matrix **Analytical Group**	Soil TCLP VOCs	**Sampler's Name**	To Be Determined
Concentration Level	Low	**Field Sampling Organization**	ERM
Sampling SOP	SOPs 3, 4, 11 & 12	**Analytical Organization**	Accutest Laboratories
Analytical Method/ **SOP Reference**	88260B / EMS8260-19	**No. of Sample Locations**	To Be Determined By Specific Sampling Activity

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Method Blank	1 / day (12 Hr BFB)	No targets above QL	If no gross detections, qualify with a B. For gross detections reanalyze samples.	Tammy McCloskey (Accutest)	Accuracy/Bias-Contamination
Surrogates	2 per sample	Recovery must fall within in-house QC criteria [1]	Reanalyze sample in order to determine matrix effect.	Tammy McCloskey (Accutest)	Accuracy/Bias
Lab Check Sample	1 / 20 samples	Recovery must fall within in-house QC criteria [1]	Reanalyze if gross exceedances.	Tammy McCloskey (Accutest)	Laboratory Accuracy
Blind field duplicate	None	N/A	N/A	N/A	N/A
Matrix Spike / Matrix Spike Duplicate Pair	None	N/A	N/A	N/A	N/A
Field Blank Trip Blank	None	N/A	N/A	N/A	N/A

1. In house QC criteria subject to change through out the project. Will be monitored during the validation process.

QAPP Worksheet #29
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 Notebook, - Monitoring Well Construction Logs, - Well Development Log sheets, - Sampling Equipment Checklists, - Groundwater Sampling Log Sheets, - Chain-of-Custody Forms, - Air Bills, and - Telephone Logs.	- Daily Instrument Calibration Logs, and - Telephone Logs.	- Sample Receipt, Custody and Tracking Records - Telephone Logs, - Laboratory Analytical Reports, and - Raw Data (archived electronically.	- Data Validation Reports, - Field Audit Checklists, and - Data Usability Summary Report.	All documents generated during the project will be recompiled and retained in the central project file. At the conclusion of the project an RA Report will be presented which will include as appendices many of the related project documents and records. Any documents not provided in the report will be presented to USEPA upon request.

QAPP Worksheet #30
Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP (s)	Data Package Turnaround [1]	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number)
All	All	All	All	All	21 days	Accutest Laboratories 2235 Route 130 Dayton, New Jersey 08810 Tammy McCloskey Laboratory Project Manager 732-355-4562	It is not anticipated that a backup laboratory will be required, however Accutest has an extensive laboratory network, and the Accutest New England facility follows all QA/QC protocol as the Accutest New Jersey facility. 495 Technology Center West Building One Marlborough, Massachusetts 01752 508-481-6200

1. Final laboratory deliverable will be a NYSDEC Category B deliverable.
2. Expedited turnaround for preliminary results may be required and requested.

QAPP Worksheet #31
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)
Field Sampling Protocol	Once at a minimum during sampling activities	Internal	ERM	ERM QA Officer ERM Field Team Leader	ERM Principal In Charge ERM QA Officer	ERM Project Manager	ERM Project Manager
Handling and Custody of Samples	Once at a minimum during sampling activities	Internal	ERM	ERM QA Officer ERM Field Team Leader	ERM Principal In Charge ERM Laboratory QA Officer	ERM Project Manager	ERM Project Manager
Analytical Laboratory Performance	The data validation process will satisfy the requirements of this audit	External	ERM	ERM Laboratory QA Officer	ERM Principal In Charge ERM Laboratory QA Officer	ERM Project Manager	ERM Project Manager

QAPP Worksheet #32
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
Field Sampling Protocol	Electronic mail which documents the results of the audit will be submitted to the project manager.	Chris Wenczel ERM Project Manager	24 hours after audit	Electronic mail	All ERM project personnel listed on Worksheet #4-2	24 hours after notification
Handling and Custody of Samples	Electronic mail which documents the results of the audit will be submitted to the project manager.	Chris Wenczel ERM Project Manager	24 hours after audit	Electronic mail	All ERM project personnel listed on Worksheet #4-2	24 hours after notification
Analytical Laboratory Performance	Electronic mail which documents the results of the audit will be submitted to the project manager.	Chris Wenczel ERM Project Manager	24 hours after audit	Electronic mail	All ERM project personnel listed on Worksheet #4-2	24 hours after notification

QAPP Worksheet #33
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 Reports See Worksheets # 35 & #36	Applicable only to Groundwater Monitoring samples	Three weeks after receipt of the laboratory data deliverable.	Mr. Andrew Coenen Laboratory QA Officer ERM	Mr. Chris Wenczel ERM Project Manager
Data Usability Assessment See Worksheet #37	Once after validated data is reviewed.	End of the Project prior to completion of final project report.	Mr. James Perazzo Mr. Chris Wenczel Mr. Eugene Gabay Mr. Ernie Rossano Mr. Andrew Coenen All ERM Personnel	Mr. Chris Wenczel ERM Project Manager
Final RA Report	Once at the end of the Project.	End of the Project.	Mr. Chris Wenczel ERM Project Manager	Distribution List presented on Worksheet # 3 less Mrs. Tammy McCloskey, Accutest Laboratories

QAPP Worksheet #34
Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain of Custody Forms	Chain of Custody (COC) Forms and FedEx shipping papers will be reviewed after the forms have been completed by the ERM sampler but prior to shipping any laboratory samples off-Site. All elements of the COC (requested analysis, bottle qty., project information, etc) will be compared to the analytical criteria specified in the QAPP and to confirm that the labels and qty. of bottles in the cooler match the information specified on the COC. The FedEx shipping form will be reviewed to certify that the address information is correct, all requested information is provided and that the appropriate shipping method (e.g., priority overnight, Saturday delivery) has been marked so that the samples arrive at the lab according to holding time and temperature preservation requirements specified in the QAPP.	Internal	Eugene Gabay ERM Field Team Leader
Audit Reports	The results of the audit reports and project assessments presented in Worksheets #31 through #33 will be retained in the project file. As specified, the results and findings will be reviewed with the appropriate members of the project teem and confirmation that all corrective measures have been completed will be the responsibility of the project manager. Reference Worksheets #31 through #33 for further details.	Internal	Mr. Chris Wenczel ERM Project Manager
Field Notes	It is imperative that detailed field notes are recorded real-time in the field to document project field activities. The field notes will be referenced during preparation of the OU1 RD Package and the Final RA Report and will be retained in the project file. A copy of the field notes will be provided as an Appendix to the final RA Report.	Internal	Eugene Gabay ERM Field Team Leader Mr. Chris Wenczel ERM Project Manager
Laboratory Data	All laboratory data will be reviewed internally by the analytical laboratory prior to reporting analytical results to ERM. All analytical laboratory data packages will comply with the 2005 NYSDEC ASP Category B reporting and deliverable requirements presented in Attachment E. Data generated from the Groundwater Monitoring samples will be validated according to the procedures specified in Worksheets # 35 and #36. A Data Usability Assessment will be prepared at the end of the project according to the protocol specified in Worksheet #37.	External Internal	Mrs. Tammy McCloskey Accutest Laboratories Project Manager Mr. Andrew Coenen ERM Laboratory QA Officer

QAPP Worksheet #35
Validation (Steps IIa and IIb) Process Table

Step IIa / IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	Review of Chain of Custodies (COCs)	The validator will review each COC as it is received by the laboratory from the field for accuracy of sample nomenclature and requested analysis. Issues will be brought to the attention of the laboratory contact and corrected immediately.	Mr. Andrew Coenen, ERM Laboratory QA Officer
IIb	Field documentation	The Project manager will review all field forms for completeness and adherence to the QAPP.	Mr. Chris Wenczel, ERM Project Manager
IIb	Review of SOPs	The validator will confirm that samples were collected and analyzed in accordance with applicable SOPs.	Mr. Andrew Coenen, ERM Laboratory QA Officer
IIa	Documentation of Method QC Results	The validator will confirm that the appropriate number of QA/QC samples were collected by ERM and analyzed by the laboratory.	Mr. Andrew Coenen, ERM Laboratory QA Officer
IIa	Review Raw Data	The validator will review 10% of the raw laboratory data to confirm the laboratories calculations.	Mr. Andrew Coenen, ERM Laboratory QA Officer
IIa	Project Quantitation Limits	The validator will confirm that the sample results meet the project quantitation limits specified in the QAPP. If they do not, the laboratory will be contacted and possible reanalysis may be required.	Mr. Andrew Coenen, ERM Laboratory QA Officer

Groundwater monitoring samples only will undergo data validation. For each laboratory data deliverable the validator will prepare a Data Usability Report (DUSR). The DUSR will be prepared according to the guidelines established by Division of Environmental Remediation Quality Assurance Group and will review the following:

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

Once the data package has been reviewed and the above questions asked and answered the DUSR will describe the samples and the analytical parameters, data deficiencies, analytical protocol deviations, and quality control problems and their effect on the data. The DUSR shall also include recommendations on resampling/reanalysis if applicable. All data qualifications will be documented following the NYSDEC ASP '05 Rev. Guidelines.

QAPP Worksheet #36
Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria 1, 2, 3	Data Validator (Title and Organizational Affiliation)
IIa	Aqueous	VOCs	Low	USEPA Region II Data Review Standard Operating Procedure (SOP) Number HW-24, Revision 2, Validating Volatile Organic Compounds by SW-846 Method 8260B – October 2006	Mr. Andrew Coenen, ERM Laboratory QA Officer

1. The order in which the aforementioned guidance documents and/or criteria are listed does not imply a hierarchy of reliance on a particular document for validation.
2. The reviewer's professional judgment also plays a large role in the validation process.
3. The waste characterization parameters and Treatment Plant Vapor samples will not be validated.

QAPP Worksheet #37
Usability Assessment

The Data Usability Assessment will revisit the DQOs to ascertain whether the data collected is adequate in quantity and quality to meet the project objectives. Also, the usability assessment will be used to determine whether qualified data can be used to make project decisions.

The Data Usability Assessment will be performed by Mr. Chris Wenczel and Mr. Andrew Coenen. Mr. Wenczel will be responsible for information in the Usability Assessment. He will also be responsible for assigning task work to the individual task members who will be supporting the Data Usability Assessment. Note that the Data Usability Assessment will be conducted on validated data only. The results of the Data Usability Assessment will be presented in the final report. The following items will be assessed and conclusions drawn based on their results:

Precision – Results of all blind field duplicates will be discussed for each analysis. For each duplicate pair, the relative percent difference (RPD) will be calculated for each analyte whose original and duplicate values are either greater than or equal to the quantitation limit. The RPDs will be checked against the measurement performance criteria presented on Worksheet #12. The RPDs exceeding criteria will be identified. The discussion will summarize the results. Any conclusions about the precision of the analyses will be drawn and any limitations on the use of the data will be described.

Accuracy/Bias Contamination – Results for all laboratory method blanks and instrument blanks will be discussed for each analysis for Confirmatory Post Excavation and Post-Removal Ground water samples only. The results for each analyte will be checked against the measurement performance criteria presented on Worksheet #12. Results for analytes that exceed criteria will be discussed. The discussion will summarize the results of the laboratory accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Sensitivity – Results for all Lab Check Samples will be presented discussed for each analysis. The results for each analyte will be checked against the measurement performance criteria presented on Worksheet #12 and cross-checked against the quantitation limits presented on Worksheet #15. Results for analytes that exceed criteria will be discussed. The discussion will summarize the results of the laboratory sensitivity. Any conclusions about the sensitivity of the analyses will be drawn and any limitations on the use of the data will be described.

Completeness – A completeness check will be done on all of the data generated by the laboratory. Completeness criteria are presented on Worksheet #12. Completeness will be calculated for each analyte as follows. For each analyte, completeness will be calculated as the number of data points for each analyte that meets the measurement performance criteria for precision, accuracy/bias, and sensitivity, divided by the total number of data points for each analyte. A discussion will follow summarizing the calculation of data completeness. Any conclusions about the completeness of the data for each analyte will be drawn and any limitations on the use of the data will be described.

Reconciliation – Each of the Project Quality Objectives (PQOs) presented on Worksheet #12 will be examined to determine if the objective was met. This examination will include a combined overall assessment of the results of each analysis pertinent to an objective. Each analysis will first be evaluated separately in terms of the major impacts observed from the Data Validation, Data Quality Indicators, and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be determined. Based on the quality determined, the usability of the data for each analysis will be determined. Based on the combined usability of the data from all analyses for an objective, it will be determined if the PQO was met and whether project action limits were exceeded. The final report will include a summary of all the points that went into the reconciliation of each objective. As part of the reconciliation of each objective, conclusions will be drawn and any limitations on the usability of any of the data will be described.

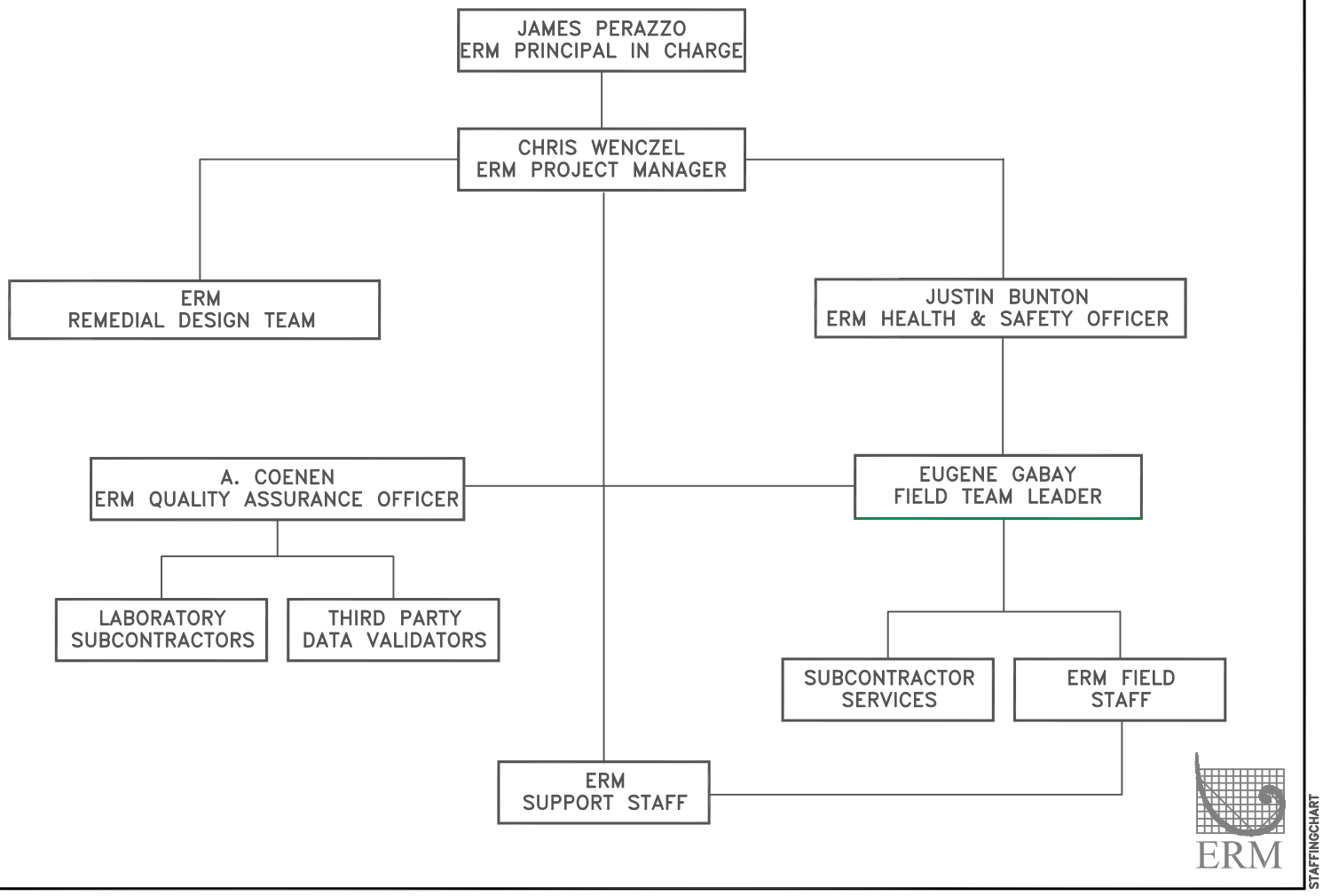
LIST OF ATTACHMENTS

ATTACHMENT A - ERM's Proposed Project Management Team

ATTACHMENT A

ERM PROJECT ORGANIZATION CHART

FULTON AVENUE SUPERFUND SITE OPERABLE UNIT 1 REMEDIAL DESIGN/REMEDIAL ACTION



ATTACHMENT B - Professional Profiles

James A. Perazzo, P.G.

Principal-In-Charge

Mr. Perazzo has 26 years of experience in the environmental field. His experience in formulating strategies to investigate legacy environmental problems, analyze data, and develop solutions to impaired assets enables client to manage environment risks and comply with disclosure obligations. He has directed projects to address CERCLA, RCRA, and TSCA and other federal and state obligations while evaluating environmental liability costs to assist sellers and purchasers in making business decisions. Mr. Perazzo brings a broad understanding of legacy problems to projects, which consider regulatory burdens, project life cycle, concept and engineering remedial estimates and operating cash flows. He has worked with clients to determine realistic cash flows as projects have matured into the remedial implementation phase enabling client's to establish proper reserves for legacy problems in conformance with financial reporting guidelines. Mr. Perazzo works with regulators and stakeholders to accurately communicate information and assist clients in meeting business goals. He routinely provides strategic guidance, conducts negotiations, and serves as an expert, giving testimony in private litigations and mediations.

Registrations & Professional Affiliations

- Professional Geologist in Pennsylvania
- New York State Council of Professional Geologists

Fields of Competence

- CERCLA RI/FS and removal actions
- RCRA (RFA, RFI CMS and CMI)
- UST assessment and hydrocarbon remediation
- Indirect/direct investigative techniques
- Soil and ground water investigations
- Hydrogeological assessments
- Regulatory negotiation and strategic guidance
- Expert witness

Education

- M.B.A. , Long Island University (C.W. Post), New York, 2006
- M.S. Earth Science, Adelphi University, New York, 1981
- B.S. Geology, The State University of New York at Stony Brook, 1978

Publications

"CERCLA - The Technical Perspective," Environmental Regulations Course, Executive Enterprises, Inc., June '95, October '95, and February '96.

"Remedial Investigation and Feasibility Study Process," New York Hazardous Regulation Course, Executive Enterprises, Inc., November 16-17, 1990.

"Groundwater Remediation; Performance Goals," Haztech International, Cleveland, Ohio, September 20-22, 1988.

"Remedial Design Needs to Consider in Planning Hazardous Waste Site Investigations," with J. Iannone and J. Mack; Haztech International, St. Louis, Missouri, August 26-27, 1987.

"Long Term Confidence in Ground Water Monitoring Systems," Groundwater Monitoring Review, Vol. 4, No. 4, all 1984.

Key Projects

Project Manager for large Superfund site containing lead. Project responsibilities included work plan preparation, RI implementation, coordination of human health risk and ecological assessments, a feasibility study, and remedial design and construction of the remediation action.

Provide expert testimony in toxic tort action involving alleged contribution of inorganic constituents from a former recycling operation to off-site parcels.

Provided expert testimony in matter involving the origin and subsequent migration of petroleum contamination as it related to on-site and off-site impacts.

Developed a tank management program for 36 locations in New York and Connecticut. Planned site assessments and remedial programs. Formulated monitoring programs for early warning of potential environmental problems. Negotiated financial estimates and justification for outstanding environmental liability allowing owner to divest with protection against future liabilities.

Provided expert testimony regarding waste characterization at a former septic disposal area and the alleged impacts to certain public and private supply wells.

Project Director for three removal actions pursuant to an ACO under 106 provisions at two separate Superfund sites that were in receivership. Performed removal of anhydrous ammonia vessel, ASTs, laboratory chemicals, drums, PCB oils, transformers, and closure of USTs. Also directed a radiological survey with a health physicist to locate and remove materials exhibiting anomalous levels of radiation. These efforts were done on behalf of a savings and loan in receivership.

Project Director for development and implementation of remedial system to extract VOCs from soil and ground water at State Superfund site. Coordinated program involving dewatering and vacuum extraction. Established basis for performance analysis and effectiveness evaluation to determine proper time for system termination.

Assessed alleged environmental liabilities at a commercial resort built on a former shipyard to facilitate a Chapter 11 bankruptcy work-out on Long Island, NY.

Conducted reviews and critiques of RI and RODs, the latter in support of petitions to amend. These efforts resulted in modifications to remedies that were consistent with the NCP and reduced client's financial exposure.

Assisted clients in securing approval for reimbursement of response costs from the Superfund

Negotiated with NYS DOL on behalf of two commercial financial institutions to secure the environmental conditions at three manufacturing facilities to allow assets to be removed as part of a Chapter 7 bankruptcy.

Developed technical approach to ongoing cases for the New York Sate Environmental Protection Bureau of the Attorney General's office. Prepared scientific reports and represented the Attorney General in adversarial discussions, public meetings, and court hearings.

As part of a multi-disciplined technical team, developed a comprehensive remedial program at a dioxin-contaminated landfill in western New York. The program involved collection and treatment of dissolved and non-aqueous phase liquids (NAPLs) in overburden and bedrock.

Technical representative for the government in developing a comprehensive soil and aquifer remediation project in Nassau County, New York. The project involved a soil and ground water remediation program including installation of a slurry wall via the vibrating beam technique, soil flushing system and staged ground water recovery from a shallow and deep aquifer. Maintained a key role in establishing performance criteria for cleanup and effectiveness monitoring.

Christopher W. Wenczel

Mr. Wenczel has more than 23 years of diversified experience in the environmental consulting field specializing in hydrogeology, hazardous waste management/remediation, and water supply. His diverse project experience includes work under CERCLA, RCRA, TSCA, NJDEP Site Remediation Program, NJPDES, NYSDEC Voluntary Cleanup Program, NYSDEC State Superfund Program, and NYSDEC Oil Spill Program.

Mr. Wenczel has experience in the development and implementation of complex remedial investigation and feasibility study (RI/FS) plans for USEPA and NYSDEC Superfund sites in both New York and New Jersey, which include 12 National Priority List (NPL) sites. He also has extensive experience in planning and performance of other compliance site investigations such as RCRA Corrective Action and property transfer due diligence environmental quality site assessments.

Mr. Wenczel's experience includes activities such as preparation of MOAs, preliminary site assessments, site investigations, remedial actions, and long-term monitoring programs at former landfills and manufacturing facilities.

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Registrations & Professional Affiliations

- State of New Jersey Certified Underground Storage Tank Investigator, License No. 0012475

Fields of Competence

- Site Investigation/Remediation Strategy & Implementation
- Ground Water Resource Development
- Multi-Media Sampling & Remediation
- Hydrogeologic Testing, Analyses and Interpretation
- Analysis of Surface & Ground Water Flow Systems
- Surface & Ground Water Quality Monitoring
- Applied Geophysics
- UST Assessment, Removal & Remediation
- Soil Vapor Extraction/Air Sparging

Education

- M.S. Earth Sciences/Hydrogeology, Adelphi University, New York, 1990
- B.S. Geology, State University of New York at Oneonta, 1985
- NJDEP UST License Renewal Course, New Jersey Society of Professional Engineers, 10 September 1998, 11 September 2001 & 9 September 2004
- State of New Jersey Certified Cleanup Star Program Participant, 2004
- 40-Hour OSHA 1910.120 Health and Safety Training, 1987, and 8-Hour OSHA Annual Refresher Training, 1987 – 2004
- 8-Hour OSHA Supervisory Training For Level B Activities, 1989

- International Symposium on Environmental Geotechnology, Lehigh University and the International Committee on Environmental Geotechnology, Allentown, PA, 21 -23 April 1986
- Theory and Application of Vadose Zone Monitoring, Sampling and Remediation, NGWA, Somerville, MA, 7-9 April 1992
- Assessment, Control and Remediation of LNAPL Contaminated Sites, API/USEPA, East Brunswick, NJ, 20 October 1994
- Environmental Horizontal Well Symposium, NGWA, Indianapolis, IA, 28-30 October 1995,
- Petroleum Hydrocarbons & Organic Chemicals in Ground Water: Prevention, Detection and Remediation, NGWA, Houston, TX, 13-15 November 1996
- NJDEP Technical Requirements For Site Remediation Seminar, Cook College @ Rutgers, 27 May 1998
- DNAPLs in Fractured Geologic Media: Monitoring, Remediation & Natural Attenuation, Univ. of Waterloo, San Francisco, CA, 8-10 December 1999
- Hydrogeology of Fractured Rock: Characterization, Monitoring, Assessment & Remediation, Fractured Rock Educational Services, Princeton, NJ, 19-22 May 2003

Key Projects

Project Manager responsible for execution of multiple projects at a major aeronautical systems manufacturing facility in Utica, New York. These projects include a NYSDEC RCRA Corrective Action program, facility relocation support and permitting, and implementation of multiple Interim Remedial Measures (IRM). The RCRA Corrective Action included the regulatory negotiation, development, and implementation of key program documents including the RCRA Facility Assessment and the RCRA Facility Investigation Work Plan. Both on-site and off-site investigations were required to characterize impacted media including soils, ground water, storm water, surface water, and building materials such as concrete and metals. Contaminants of concern at the facility included volatile organic compounds, semi-volatile organic compounds, polychlorinated biphenyls (PCBs), metals, and cyanide. IRMs included removal and disposal of structures, vent stacks, stormwater conveyance systems, soil, and concrete. Facility relocation support included procurement of permits/registrations for sanitary wastewater discharges, air discharges, petroleum bulk storage tanks, waste management, development of a spill control, containment and countermeasures plan (SPCC), and revisions to both waste management and emergency control procedure plans.

Project Manager responsible for execution of multiple projects at Brookhaven National Laboratory, Upton, NY, with revenues in excess of $2.8 million. These projects include extensive ground water delineation projects for volatile organic compounds, metals, and radionuclides. These ground water surveys include the High Flux Beam Reactor emergency response tritium delineation project conducted in March 1997. In a six-week period, ERM's team installed and sampled a total of 72 temporary ground water vertical profile wells to depths ranging between 200 and 300 feet below grade. In addition, these projects have included walk-over radiation surveys across the site, and geotechnical studies for BNL's sewage treatment plant.

Project Manager responsible for the implementation of an extensive RI/FS and Soil IRM at the Fulton Avenue Superfund site located in Garden City Park, NY. The Fulton Avenue site is listed on both the NYSDEC Registry of Inactive Hazardous Waste Sites and the USEPA NPL. Past discharges of chlorinated solvents (tetrachloroethene) have caused extensive ground water contamination in the Upper Glacial and Magothy aquifers. The ground water contaminant plume has allegedly migrated a distance of 2 miles from the site to depths of up to 500 feet to affect up to 5 public supply wells encompassing an area of approximately 5 square miles within Nassau County. The RI/FS focuses on a ground water vertical profiling task using temporary wells to further define the extent of ground water contamination within the upper glacial aquifer and the Magothy aquifer, and to select permanent ground water monitoring well locations and screen settings; installation of permanent conventional and multi-level

ground water monitoring wells to act as permanent monitoring and/or compliance points within the upper glacial aquifer and the Magothy aquifer; collection of ground water samples from over 60 ground water monitoring wells; collection of several rounds of synoptic ground water level data; a three-dimensional ground water flow computer model; a risk assessment for ground water; and a feasibility study for ground water. The soil IRM is comprised of a source area soil removal action, and the installation of a soil vapor extraction (SVE) and air sparging (AS) to remove contaminants from the vadose zone soils and the shallow ground water table. Since the SVE/as system went online in October 1998, approximately 10,000 pounds of tetrachloroethene has been removed from the ground. The post-IRM Site closure included indoor air sampling and installation of a sub-slab venting system beneath the building at the Site.

Project Manager/Senior Hydrogeologist responsible for the coordination and performance of a major off-site hydrogeologic investigation for a manufacturing facility and ISRA site (NJDEP Site Remediation) in South Brunswick, NJ. Conducted an extensive volatile organic compound plume delineation task in a dual aquifer ground water system which utilized the terrain conductivity, resistivity and VLF geophysical mapping techniques and the Hydropunch ground water sampling technique. Other site investigative activities have included: the phased installation of an extensive ground water monitoring well network, performance of multiple aquifer tests, characterization of the subsurface geologic and hydrogeologic regime, test pitting, soil sampling, an UST investigation, ground water sampling, performance of a soil vapor extraction pilot study, design/installation/testing of a ground water recovery well, data analyses/interpretation, and preparation of an Site Assessment Report, an extensive Pump Test Report, Soil and Ground Water Remedial Action Work Plans, a Comprehensive Hydrogeologic Report, a SVE Pilot Study Report. Remedial Action Work Plans proposed the use of SVE, biosparging, and pump and treat technologies. All three systems are currently in operation and effectively remediating soil and ground water contamination at the site.

Management and supervision of hydrogeologic investigation at an Ashland Drum Landfill Site, Fords, New Jersey (NJDEP Site Remediation). The investigation included: the installation of a ground water monitoring well network, characterization of the subsurface geologic and hydrogeologic regime, a study of tidal influence on ground water flow, test pitting, soil sampling, ground water sampling, drum sampling, data analyses and preparation of an RI Report.
Under the USEPA Superfund program, participated in RI/FS and Remedial Design (RD) programs at the following NPL Sites: the Lipari Landfill, the Port Washington Landfill, the Lone Pine Landfill, the Vestal Well Field RI and RD, the Sinclair Refinery site, Swope Oil Company site, the Metaltech/Robintech/National Pipe site, the Sarney Farm site, the Montclair/West Orange Radium site, and 150 Fulton Avenue.

Senior Hydrogeologist responsible for the coordination and supervision of a comprehensive RI at the Pfohl Brothers NYSDEC State Superfund site (120 acres) located in Williamsville, NY. The site investigation of Pfohl Brothers Landfill included: preparation of a RI work plan, Health and Safety Plan (HASP), a Quality Assurance Plan (QAPP), geophysical surveys using terrain conductivity, magnetometry and ground penetrating radar, soil borings, ground water monitoring well installation in both bedrock and overburden aquifers, soil sampling, sludge sampling, hydrologic monitoring of surface water bodies, surface water sampling, ground water sampling, landfill leachate sampling, test pitting and drum sampling. In addition to the overall site characterization, evaluated the presence of low-level radionuclide contamination on the site, delineated, and mapped over 450 radioactive "hot- spots" using scintillometers. Radionuclides found at the site included radium-226, thorium-232, cesium-132 and uranium-238 in the form of discarded machine parts, radioluminescent badges, and ore rocks.
Installation of ground water and landfill gas monitoring wells as part of an RI for the Port Washington Municipal Landfill NPL site, Port Washington, New York. Additionally, participated in the development and implementation of a landfill gas sampling program using flux boxes, landfill gas monitoring wells and summa canisters.

Senior Hydrogeologist responsible for the coordination and performance of a comprehensive environmental assessment at the former ESSO petroleum refinery, San Nicholas, Aruba, N.V. The investigation included: the installation of a ground water monitoring well network, characterization of the subsurface geologic and hydrogeologic regime, test pitting, soil sampling, an above ground storage tank investigation, ground water sampling, mapping of extensive LNAPL bodies, data analyses/interpretation, and preparation of an Site Assessment Report.
Participated in two NPL site RD programs, Vestal Well 1-1, Vestal, New York and the Lipari Landfill, Pitman, New Jersey. Activities for the Vestal Well 1-1 site included the preparation of a Remedial Design work plan, HASP and QAPP, performance of a soil boring program and design of a 1,000-gpm air stripper. Activities for the Lipari Landfill included the design of an automated extraction/injection well network and a 300-gpm production well.

Project Manager responsible for execution several major environmental investigative/cleanup tasks at the former Brooklyn Navy Yard (Brooklyn Navy Yard Industrial Park {BNYIP}), that have included:
Phase I & II Site Assessment/Investigation Services Related To a NYSDEC Voluntary Cleanup Agreement, Implementation of Interim Remedial Measures, and Investigation/Closure of Underground Storage Tanks

ERM performed a Phase I Preliminary Site Assessment data gathering and evaluation process in conjunction with a Phase II Site Investigation to address key data gaps for potential area and activity-specific sources of hazardous substances. The Phase I Preliminary Site Assessment included site inspections, review of all historic data/records, previous investigations performed at the BNYIP to date, inspection of BNYIP facilities, interviews of facility personnel regarding current and past operations.

The Phase II investigation included the sampling and characterization of environmental conditions at electrical substations/transformer areas, drum storage areas, dry docks, and facility-wide ground water characterization. The Phase II Investigative findings were then integrated with the Phase I Site Assessment information to prepare a Comprehensive Environmental Assessment Report (CEAR) for the BNYIP.

ERM provided complete turnkey services for investigation and closure of 10 underground petroleum storage tanks located in seven separate areas at the BNYIP. These services included pre-closure site investigations at each tank locations, preparation of all regulatory required work plan documents, notification of interested regulatory agencies (NYSDEC, NYCFD), procurement of necessary permits, closure by excavation and removal of the USTs and effected soils, complete restoration of each former tank location, and preparation of a final comprehensive UST Closure Report for submittal to NYSDEC.

ERM performed an Interim Remedial Measure (IRM) at former electrical substation to mitigate PCB contamination resulting from releases of electrical transformer dielectric fluids. The IRM included characterizing the extent of PCB contamination on concrete surfaces and soils/sediments associated with the former transformers. The IRM included the removal, containment and disposal of soils/sediments containing high levels of PCBs from a subsurface vault, cleaning, scarification, and final encapsulation of all effected concrete surfaces within the vault and other concrete surfaces associated with the former transformers. A Final Remediation Report was prepared and submitted to NYSDEC for review and official acknowledgment that "no further action" is required at this electrical substation.

Project Manager responsible for the implementation of an RI/FS at the NYSDEC Utility Manufacturing State Superfund site located in New Cassel, NY. The Utility Manufacturing site is listed on the NYSDEC Registry of Inactive Hazardous Waste Sites. Past discharges of chlorinated solvents have caused extensive ground water contamination in the Upper Glacial and Magothy aquifers affecting several deep public supply wells in the Bowling Green Water District. The RI features the off-site installation of soil borings to collect both lithologic samples to characterize off-site stratigraphic conditions, and groundwater samples using a Hydropunch to

characterize off-site groundwater quality/impacts (i.e. determine if site-related contaminants have migrated off-site); installation of groundwater monitoring wells to confirm the results of the Hydropunch sampling; and the collection of soil gas samples to evaluate potential risks from soil vapor migration.

Project Manager responsible for third-party oversight on behalf of ERM's client to ensure responsible parties (former owners) comply with all applicable NJDEP soil and ground water remediation standards and the NJDEP-approved Remedial Action Plan for an NJDEP ISRA site in Paramus, New Jersey. Additional activities include oversight of an asbestos removal action at the same site.

Eugene T. Gabay

Mr. Gabay has more than 6 years of diversified experience in the environmental consulting field specializing in hydrogeology, hazardous waste management andremediation. His diverse project experience includes work under CERCLA, RCRA, NJDEP Site Remediation Program, NJPDES, NYSDEC Voluntary Cleanup Program, NYSDEC State Superfund Program, and NYSDEC Oil Spill Program. Mr. Gabay is currently managing several New York State Department of Environmental Conservation projects, and has logged well over 5,000 hours organizing and performing various field activities. Mr. Gabay's field experience includes ground water and soil sampling, field parameter measurement, monitoring well installation and horizontal drilling activities, installation of vertical profile wells, logging of soil and bedrock, oversight of underground storage tank removals, subsurface and indoor air sampling, oversight of unexploded ordinance investigations and oversight of remediation activities at several manufactured gas plants in the State of New York.

Registrations & Professional Affiliations

- 40-hour Health and Safety Certification
- ExxonMobil Loss Prevention System-Certified

Fields of Competence

- Geologic and Hydrogeologic Correlation, Analysis, Interpretation and Assessments
- Soil and Ground Water Investigations
- Air Quality Investigations and Monitoring
- Multi-Media Sampling
- Underground Storage Tank Assessments, Removals, In-Place Closures and Hydrocarbon Remediation
- Applied Geophysics
- Aquifer Testing, Tidal Studies and Analysis
- Project Management and Client Liaison
- Project Planning and Scoping
- Project Budgeting and Scheduling
- Regulatory Agency Interaction
- Health and Safety Officer Of Site Investigations
- Field Management

Education

- M.S. Environmental Studies, C.W. Post University, New York, Currently Enrolled
- B.A. Environmental Planning and Resource Management, Plattsburgh State University, New York, 2000
- Associates Liberal Arts, Nassau Community College, New York, 1997

Key Projects

Mr. Gabay has logged over 5,000 hours as a Field Team leader for performance of investigative and remedial activities, and tasks associated with field activities.

Project Manager of New York State Department of Environmental Conservation (NYSDEC) Candlewood Road Superfund Site Investigation. Responsibilities included work plan design and implementation, oversight of field work, interpretation of laboratory data and report writing. Both on-site and off-site investigations were required to characterize impacted media including soils, soil vapor and groundwater. Contaminants of concern at the facility included chlorinated volatile organic compounds (VOCs). Investigative activities included sampling of soil, soil vapor and groundwater through the installation of Geoprobe soil borings, Geoprobe groundwater vertical profiling, and soil vapor sampling probes.

Project Manager of New York State Department of Environmental Conservation Levey Property Site. Responsibilities included work plan design and implementation, oversight of field work, interpretation of laboratory data and report writing. Both on-site and off-site investigations were required to characterize impacted media including soils, soil vapor and groundwater. Contaminants of concern at the facility included VOCs, semi-VOCs and metals. Investigative activities included sampling of soil, soil vapor and groundwater through the installation of Geoprobe soil borings, Geoprobe groundwater vertical profiling, and soil vapor sampling probes.

Project Manager of New York State Department of Environmental Conservation (NYSDEC) Pride Solvent Superfund Site. Contaminants of concern at the Site included VOCs, semi-VOCs and metals. Investigative activities included sampling of soil, soil vapor and groundwater through the installation of Geoprobe soil borings, Geoprobe groundwater vertical profiling, and soil vapor sampling probes.

Responsible for the coordination and implementation of a complex groundwater monitoring program at major petrochemical facility in Linden New Jersey since 2003. These responsilbilities include compliance with facility-specific health and safety programs, and receipt, review, evaluation and reporting of the monitoring data.

Managed and assisted in the selection and installation of multilevel groundwater monitoring systems such as the Solinst Waterloo, Westbay and the FLUTe multilevel sampling systems.

Field Manager and site health and safety officer at cable manufacturer in Yonkers, New York. Included soil sampling program, monitoring well installation, and ground water sampling.

Performed quarterly and annual water sampling rounds at a major petroleum storage and distribution terminal in Holtsville, New York.

Field Manager and site health and safety officer at a residence in Water Mill, NY. Project consisted of a vapor/fluid recovery system, and frequent groundwater monitoring and reporting.

Performed the duties of Field Manager and site health and safety officer at a NYSDEC site in West Babylon, New York. Responsibilities included ground water sampling, soil sampling, and leach field and septic system excavation and sampling.

Performed quarterly and annual groundwater monitoring activities several industrial manufacturing facilities in New Jersey.

Performed quarterly and annual water level and water sampling rounds at a cable-manufacturing site in New Brunswick, New Jersey.

Performed the duties of air sampling technician on numerous asbestos abatement projects in New York City, and Long Island.

Performed the duties of Asbestos Inspector in several buildings in Long Island. Responsibilities included sampling all suspect asbestos containing materials.

Flute installation

Andrew Coenen

Mr. Coenen has 13 years of general analytical chemistry experience, 6 years of analytical laboratory experience, and 7 years of environmental consulting experience, including analytical data validation, sampling and analysis programs, quality assurance programs, technical support, and QA oversight for fixed laboratory and field analysis. Mr. Coenen has knowledge of numerous analytical methodologies and experience in data validation of analytical data package deliverables for adherence to USEPA CLP and non-CLP, NYSDEC ASP, and NJDEP protocols. He is proficient with GIS/Key environmental management software and has operated a mobile gas chromatograph laboratory used to test soil and water samples for quick-turn volatile analysis.

Fields of Competence

- Analytical data review and validation
- Environmental database management (GIS/Key)
- Laboratory Subcontractor Management
- Analytical protocols for pollutants by USEPA methodologies
- Methods of analysis of organic and inorganic parameters
- Review and preparation of QA/QC plans
- Field analytical techniques
- Multi-Media Sampling

Education

- 8-Hour OSHA Annual Refresher Training, 1999 - current
- Rutgers University / Cook College - NJDEP Using GIS for Environmental Evaluations, October 1999
- 40-Hour OSHA [29 CFR 1910.120 (e) (2)] Health and Safety Training, 1998
- Computer Aided Drafting, 50-Hour Course, Island Drafting and Technical Institute, 1998
- Immunoassay Testing Training Program, Strategic Diagnostics Inc., 1998
- B.S. Chemistry, University of Michigan, 1991

Key Projects

Data validation for numerous projects located in New York, New Jersey, Pennsylvania, Illinois, Massachusetts, Indiana, and Wisconsin, involving evaluation of aqueous, soil, sediment, leachate and air samples analyzed by USEPA Contract Laboratory Protocols, New York State DEC Contract Laboratory and Analytical Services Protocols and SW-846 methodologies for organic, inorganic, wet chemistry parameters, TPH and various other analyses.

Reviewed sampling and laboratory chemical data for adherence to New Jersey Department of Environmental Protection protocols on numerous projects. Also constructed electronic deliverables for submission to NJDEP in required haz-deliverable format.

Database construction & management for numerous investigations utilizing GIS/Key software. Compiled field and laboratory data and generated result summary tables, contours, isopleths, contaminant plume maps, cross-sections and boring logs.

Prepared numerous Sampling and Analysis Plans (SAPs) and Quality Assurance Project Plans (QAPPs) for adherence to state and federal guidelines.

Project management and technical support for Special Analytical Services required to delineate low-level PAH contamination at a Superfund Site. This included method development and validation of a Selected Ion Monitoring (SIM) GC/MS technique.

Utilized Immunoassay test kits for field measurement of PCB contamination at the former Brooklyn Navy Yard, Brooklyn, New York. Performed data validation of all field analytical samples and off-site laboratory samples and compared off-site results to test kits.

Conducted subsurface investigations with a Geoprobe. Performed various field tests.

Supervision of tank removal and subsequent soils evaluation for contamination.

Justin M. Bunton, ASP

Mr. Bunton is a Staff Scientist within ERM, based in Hartford, CT. Justin is a degreed safety professional with experience in implementing and managing safety & health programs in warehousing operations. Prior to joining ERM in December 2007, Mr. Bunton worked as a Health & Safety Manager for a large grocery distributor. As an H&S manager, Justin has developed, implemented and managed a number of programs on a wide variety of topics, including:

- H&S Audits and Inspections
- Development/Implementation of H&S Programs, Policies and Procedures
- Job Hazard Analysis/Safe Work Practices
- H&S Training
- Accident/Incident Investigation & Recordkeeping
- First Aid/Injury Management
- Control of Hazardous Energy (Lockout/Tagout)
- Bloodborne Pathogens Exposure Control
- Emergency Action Plans
- Hazard Communication
- Personal Protective Equipment
- Industrial Hygiene
- Hearing Conservation
- Behavior-Based Safety
- Powered Industrial Trucks
- Ergonomics
- Machine Guarding
- Walking and Working Surfaces/Fall Protection
- Worker's Compensation

Education

- B.S. Occupational Safety Studies, Keene State College, 2006
- OSHA 511 General Industry Outreach Training
- OSHA 2250 Ergonomics Training
- OSHA HAZWOPER
- FEMA IS-00235 Emergency Planning Training
- American Red Cross CPR, First Aid, and AED Trained

Registrations & Professional Affiliations

- Associate Safety Professional (ASP)- Board of Certified Safety Professionals
- American Society of Safety Engineers (ASSE)

Fields of Competence

- Organization and administration of H&S programs
- H&S compliance
- H&S auditing
- Hazard analysis and control
- Safety management systems
- Competence & training
- Construction Management

Key Projects

Project Scientist for Industrial Hygiene Sampling Program for Confidential Natural Gas Utility– Conducted multiple rounds of employee exposure monitoring for a wide variety of constituents (such as cadmium, carbon black, asbestos, particulates and noise) associated with the processes at several facilities, to ensure compliance with applicable regulatory standards.

Compliance Audit, Confidential Food Distributor - Performed a two-day audit of production operations in multiple facilities to identify key health and safety issues, potential regulatory compliance issues and share best management practices (BMPs).

Industrial Hygiene Sampling, Confidential Plastic Color Concentrate Manufacturer– Performed industrial hygiene sampling to assess potential employee exposures to pigment dust and various metals associated with the facility's mixing operation to ensure compliance with applicable OSHA standards.

Compliance Audit for Confidential Locomotive Engine Manufacturer - Performed a comprehensive compliance audit of a major locomotive engine manufacturing facility to identify key health and safety issues, identify potential OSHA violations and assist with making recommendations for corrective measures.

On-Site Health and Safety Compliance Support for Large Food Manufacturing Company- Performed oversight of construction contractors to ensure compliance with OSHA CFR 1926 regulations during flood relief and recovery operations. Duties included: site walkthroughs, meeting with contractors to address and advise on H&S concerns, site safety training for new workers, reporting and correcting possible hazards, and ensuring the implementation of corrective actions.

General Facility Safety Assessment for Confidential Medical Solvent Laboratory- Performed a Safety and Health assessment of the laboratory's facility to identify any possible safety issues, OSHA violations and make recommendations for improvement or corrective measures.

Regulatory Applicability Review for Confidential Aerospace Component Manufacturer- Performed a review of the companies operations and written programs to determine applicable health and safety regulatory/legal requirements.

Industrial Hygiene Sampling, Confidential Plastic Food and Beverage Waste Processor– Performed industrial hygiene sampling to assess potential employee exposures to dust and various particles associated with the facility's grinding operation to ensure compliance with applicable OSHA standards.

Compliance Audit for Confidential Specialty Metal Alloy Manufacturer - Performed a comprehensive compliance audit of a specialty brass and stainless steel alloy manufacturing facility to identify key health and safety issues, identify potential OSHA violations and assist with making recommendations for corrective measures.

On Site Construction Manager for a 29,000 square foot replacement- Provided Health and Safety as well as project oversight at the facility for the removal and replacement of the roof. The project involved the removal and disposal of approximately 15,000 square feet of asbestos containing roofing materials before the new roof could be installed.

On Site Construction Manager for large excavation and UST removal- Provided Health and Safety as well as project oversight for the excavation of 800 tons of contaminated soil, backfill of the excavated area, removal of the existing UST, installation of a new AST, the construction of an enclosure around the AST, and was responsible for obtaining necessary construction and occupancy permits from the town.

John Mohlin, P.E.

Mr. Mohlin has more than 16 years of environmental engineering consulting experience with emphasis on remediation of contaminated soil and groundwater, remedial investigations, feasibility studies, operation of remedial systems, industrial and domestic wastewater treatment, and air emission permitting and control. He is experienced in conducting pilot studies to evaluate the feasibility of soil and groundwater treatment systems using: air sparging, soil vapor extraction, ozonation, carbon adsorption, chemical precipitation, filtration, and dissolved air flotation. He has also performed several vapor intrusion investigations, as well as pilot tested and designed mitigation systems.

Mr. Mohlin has prepared designs for air sparging, soil vapor extraction, groundwater treatment, and vapor intrusion mitigation systems. He has also prepared industrial air emissions surveys and the corresponding air permit applications, and performed construction oversight during remediation projects.

Mr. Mohlin is continuously involved in engineering oversight of several remediation systems, including those utilizing air stripping, UV peroxidation, soil vapor extraction, air sparging, metals removal, carbon adsorption, multiphase extraction, and catalytic oxidation. Oversight includes system trouble-shooting, constant air and water quality testing and evaluation of results, management of operation subcontractors, maintenance operations, preparation of reports, and design of system upgrades.

Registrations & Professional Affiliations

- Registered Professional Engineer in New York State
- American Society of Civil Engineers
- American Academy of Environmental Engineers

Fields of Competence

- Management of site investigation and remediation projects
- Design and engineering support of soil, groundwater, and wastewater treatment systems
- Design and operations of air sparging, soil vapor extraction, and other remediation systems
- Vapor intrusion investigation and mitigation
- Soil and groundwater remediation pilot studies
- Industrial wastewater treatment
- Development of feasibility studies
- Hazardous waste management
- Regulatory permitting and compliance for air and water
- Air quality engineering
- Construction oversight
- Health and safety monitoring

Education

- M.S. Environmental Engineering, Polytechnic University, New York, 1997
- B.S. Environmental Engineering, Florida Institute of Technology, 1993
- 40 hour OSHA 1910.120 Health and Safety Training
- Computer Aided Drafting, 50-hour Course, Island Drafting and Technical Institute, 2001

Languages

- English, native speaker

Key Projects

Designed a 50-gpm groundwater treatment system to remove metals and BTEX, using equalization, metals precipitation, UV peroxidation, and pH adjustment. Specified equipment and prepared an equipment layout. Developed a pipe arrangement. Calculated necessary head for pumps, and specified pumps. Determined process logic and prepared control narrative. Currently, managing long-term operation of this system and the groundwater monitoring program.

Served as Project Manager for the investigation of a groundwater PCE plume in a residential/commercial neighborhood. Investigation techniques included vertical profile borings using Hydropunch groundwater sampling, indoor air sampling, and sub-structure soil vapor sampling. Presented at public meeting and prepared Feasibility Study and Site Management Plan to address the plume.

Designed two sub-slab depressurization systems for 150,000+ sf warehouse operations in New York.

Evaluated the extent of vapor intrusion at seven homes in France, and proposed mitigation options. Building constraints included: heated floor slabs, 300+ year old home, multiple basement levels, and limited interior access.

Designed a sub-slab vapor mitigation system consisting of a spray-applied vapor barrier with recovery of sub-slab vapors using wind-driven ventilators.

Served as Project Manager for the evaluation of an industrial wastewater stream prior to shutdown of a production line. Reviewed raw materials, flow rates, and existing data, and predicted future wastewater characteristics. Recommended changes to the existing treatment process. Prepared a request to the local POTW for a modification in pretreatment limits, and provided justification for the change in limits.

As Project Manager, evaluated the unexpected presence of acetone in the wastewater of a vitamin manufacturer, and determined the source of the acetone.

Served as Project Manager for the investigation of soil impacts beneath two large buildings using horizontal drilling techniques.

Served as Project Manager for remedial investigation report and feasibility study for urban site with PCBs, metals, and SVOCs in soil and groundwater. Also managed the design for the removal of 4,000 cy of impacted soil.

Served as Project Manager for the remedial investigation of the property of an active airport. Developed scope of work and coordinated project team to perform soil borings, groundwater sampling, well installation, and test pit installation. Prepared a remedial investigation report based on the results.

Served as Project Manager for designing and constructing upgrades to an industrial wastewater treatment process to remove excess lint and powder from the water. Utilized an inline filter press with continuous recycle and an industrial vibrator on the existing clarifier.

Served as Project Manager for the development and implementation of a Remedial Action Work Plan for two former petroleum research facilities to address soil and groundwater, and the subsequent remediation activities, including: soil excavation, monitored natural attenuation, enhanced biodechlorination, soil capping, and soil mixing.

As Project Engineer, performed extensive pilot study for remediation of contaminated soil and groundwater at a major gasoline terminal. The study included operation of a soil vapor extraction/air sparging system and a catalytic oxidizer. Performed sampling of soil, soil vapor, and groundwater. Involved in the conceptual-level and full-scale designs of the soil vapor extraction/air sparging system. Pilot study included computer modeling to estimate remedial clean-up time.

Served as Project Engineer for engineering support for the water and wastewater systems for two summer camps in remote locations. Collected monthly compliance samples, recommended treatment system upgrades, performed sampling for Microscopic Particulate Analysis, and prepared application for a new drinking water supply well.

Assisted Health and Safety Officer and Field Engineer to provide site health and safety and engineering oversight of a $7,000,000 soil excavation.

As Project Engineer, evaluated the feasibility of using an in-situ iron treatment wall for the removal of chlorinated VOCs from groundwater. Developed a conceptual design for a wall that is 15 feet deep and 600 feet long.

Served as Project Engineer responsible for air emissions survey of a manufacturing facility with over 30 separate processes and emission points. The survey included an evaluation of each process such that mass balances could be used to calculate the emissions of each raw material. The emissions were then used to determine the potential annual impact and the short-term impact. These impacts were compared with guidance concentrations in order to determine the need for air emission controls. The resulting survey was used for the preparation of New York State applications for a permit to operate.

As Project Engineer, determined the capability of a domestic wastewater plant to accommodate an increased flow. Then, evaluated the potential and ultimately recommended the use of the treated domestic wastewater (i.e., "gray water") in an industrial cooling system. Assisted in the design of a gray water reuse system.

As Project Engineer, completed EPA Hazard Ranking System scoring for a site in Puerto Rico contaminated with mercury.

ATTACHMENT C - Standard Operating Procedures (SOPs)

ATTACHMENT C

STANDARD OPERATING PROCEDURES (SOPS)

Section	Standard Operating Procedure
C.1	SOP 1 Water Level Measurement Procedures
C.2	SOP 2 Groundwater Sampling Procedures
C.3	SOP 3 Field Blanks
C.4	SOP 4 Trip Blanks
C.5	SOP 5 Membrane Interface Probe Procedures
C.6	SOP 6 Waterloo Vertical Profile Borings with Groundwater Sampling Procedures
C.7	SOP 7 Pump Test Procedures
C.8	SOP 8 Geologic Boring Drilling Procedures
C.9	SOP 9 Temporary Well Installation and Sampling Procedures
C.10	SOP 10 Potable Water Blanks
C.11	SOP 11 Decontamination Procedures
C.12	SOP 12 Waste Management and Disposal

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C.0 STANDARD OPERATING PROCEDURES

C.1 WATER LEVEL MEASUREMENT PROCEDURES

The following procedure shall be used for water level measurements:

- Clean all water-level measuring equipment using appropriate decontamination procedures.
- Wear appropriate health and safety equipment as outlined in the Health and Safety Plan. In addition, samplers shall don new sampling gloves at each individual well prior to sampling.
- Visually examine the exterior of the monitoring well for signs of damage or tampering and record in the field logbook.
- Unlock well cap.
- Take and record in field logbook PID and/or OVA readings.
- Measure the static water level in the well with an electronic water level indicator. The water level indicator shall be rinsed with deionized water in between individual wells to prevent cross-contamination. Synoptic round of water level measurements shall all be completed on the same day.
- For wells located within the GCPIA, an interface probe will be used to check the bottom well sump for the presence of DNAPL. If it appears that DNAPL is present, an attempt will be made to collect a sample of the DNAPL using a discrete depth-sampling device such as a Bacon Bomb sampler. Groundwater samples will not be collected from any well containing DNAPL. Attach a pre-cleaned decontaminated discrete depth-sampling device to a new, dedicated length of polypropylene string. Set the sampler in the open position, and slowly lower the device to the bottom of the well. Upon reaching the well bottom, close the sampler using the wire-line or bottom actuated release mechanism to collect a sample. Slowly retrieve the sampler from the well, and collect a sample of the fluids into a sample jar for analysis and characterization.
- If DNAPL is not detected in the well, continue with the procedures described below.

C.2 SOP 2: GROUNDWATER SAMPLING PROCEDURES

Groundwater sampling will be performed using USEPA low-flow well purging/sample collection techniques. The following subsections present general preliminary well sampling procedures common to both techniques followed by low-flow sampling procedures, and if for some reason it is not possible to perform low-flow sampling, conventional procedures are also presented for reference.

The low-flow groundwater purging/sampling technique employs the use of a flow-through cell equipped with probes and a meter for measuring groundwater quality parameters such as pH, temperature, specific conductivity, dissolved oxygen and oxidation/reduction potential. One example of this equipment is the Horiba U-22 Flow-Through Cell and the specific manufacturer's calibration and operation instructions should be followed.

C.2.1 General Procedures

The following procedure will be used for all monitoring well groundwater sampling:

- Clean all water-level measuring equipment using appropriate decontamination procedures.
- Wear appropriate health and safety equipment as outlined in the HASP. In addition, samplers will don new sampling gloves at each individual well prior to sampling.
- Visually examine the exterior of the monitoring well for signs of damage or tampering and record in the field logbook.
- Unlock well cap.
- Take and record in field logbook PID and/or Organic Vapor Analyzer (OVA) readings.
- Measure the static water level in the well with a decontaminated steel tape or electronic water level indicator. The tape or water level indicator will be rinsed with deionized water in between individual wells to prevent cross-contamination. Synoptic round of water level measurements will all be completed on the same day.
- All wells will also be checked for the presence and thickness of Light or Dense Non Aqueous Phase Liquids (LNAPL/DNAPL).

- If LNAPL or DNAPL is encountered on the top of the water table at the time of sampling, a sample of the LNAPL or DNAPL will be collected for analysis if accumulations are sufficient. Measurement of the thickness of this layer will be taken using an interface probe. A sample of the LNAPL or DNAPL may be obtained using a dedicated bottom-loading bailer. The sample will be sent to the laboratory for analysis of its chemical composition and physical properties (e.g., specific gravity, and gas chromatograph (GC) fingerprint). Initially, no groundwater sample will be collected from wells that contain LNAPL or DNAPL.
- If LNAPL or DNAPL is not detected in the well, continue with the low-flow sampling procedures described below.

C.2.2 Low-Flow Sampling

The low-flow sampling procedure is intended to reduce the amount of purge water generated during groundwater monitoring well sampling.

Sample Equipment

- Adjustable-rate, positive displacement pumps (e.g., centrifugal or bladder pumps constructed of stainless-steel or Teflon®). The selected pump must be specifically designed for low-flow rates (i.e., use of a high volume pump that is adjusted down to a low flow setting is not permitted).
- Tubing used in purging and sampling each well must be dedicated to that well. Once properly located, moving the pump in the well should be avoided. Consequently, the same tubing should be used for purging and sampling. Teflon® and Teflon®-lined polyethylene tubing must be used to collect samples for organic analysis.
- Electronic water level measuring device, 0.01-foot accuracy.
- Flow measurement supplies (e.g., graduated cylinder and stop watch).
- Interface probe.
- Power or air source (generator, compressed air tank, etc.).
- In-line purge criteria parameter monitoring instruments - pH, turbidity, specific conductance, temperature, ORP, and dissolved oxygen.
- Decontamination supplies.
- Logbook and field forms.

- Sample bottles.
- Sample preservation supplies (as specified by the analytical methods).
- Sample tags or labels, chain of custody forms.
- Well construction data, location map, field data from last sampling event.

Sample Procedure

1) Lower pump, safety cable, tubing, and electrical lines very slowly into the well to a depth corresponding to the center of the saturated screen section of the well. The pump intake must be kept at least two feet above the bottom of the well to prevent mobilization of any sediment. Lowering the pump quickly, or even at a moderate rate, will result in disturbing sediment in the well. This is one of the most important steps in low flow sampling at the Site.

2) Measure the water level again with the pump in well before starting the pump. Start pumping the well at 100 to 500 milliliters per minute. Ideally, the pump rate should cause little or no water level drawdown in the well (less than 0.3 foot and the water level should stabilize).

- Measure and record the depth to water and pumping rate every 3 to 5 minutes (or as appropriate) during pumping. If purging continues for more than 30 minutes, readings will be recorded at approximately 10-minute intervals. However, once stabilization is indicated, a minimum of 3 consecutive readings at 3 to 5 minute intervals will be recorded prior to sample collection.
- Care should be taken not to cause pump suction to be broken or entrainment of air in the sample. Do not allow the groundwater level to go below the pump intake.
- Pumping rates should, if needed, be reduced to the minimum capabilities of the pump to minimize drawdown and/or to ensure stabilization of indicator parameters.

3) During purging, measure and record the field indicator parameters using the in-line meter (turbidity, temperature, specific conductance, pH, Eh, and dissolved oxygen) every 3 to 5 minutes (or as appropriate). If purging continues for more than 30 minutes, readings will be recorded at approximately 10-minute intervals. However, once stabilization is indicated, a minimum of 3 consecutive readings at 3 to 5 minute intervals will be recorded prior to sample collection.

- The well is considered stabilized and ready for sample collection once all the field indicator parameter values remain within 10 percent for 3 consecutive readings.
- If drawdown in the well is measured at 1 foot or more, continue to low flow purge until a minimum of the equivalent volume of 1 well casing volume is removed. Using the flow equation to calculate the volume of purge water. Then collect the ground water sample.

4) Before sampling, either disconnect the in-line cell or use a by pass assembly to collect groundwater samples before the in-line cell. All sample containers should be filled by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

5) Label the samples using waterproof labels, or apply clear tape over the paper labels. Place all samples in a cooler as described in the QAPP with bagged ice or frozen cold packs and maintain at 4°C for delivery to the laboratory.

6) Do not use ice for packing material; melting will cause bottle contact and possible breakage.

7) Measure and record well depth. Take final water quality reading using low flow cell.

8) Secure the well.

C.2.3 Standard Purging and Sampling Procedure

1) Calculate the volume of water in the well as follows:

$$\text{Volume (in gallons)} = 3.14r^2(h) \times 7.48 \text{ gal/ft}^3$$

Where

h - well depth (feet) - static water level (feet)

r = well radius (feet)

2) Lower the decontaminated submersible pump with new, dedicated lengths of polyethylene tubing into the well so the pump is set at the screen interval. Purge 3 to 5 volumes of water from the well, using the submersible pump.

3) Measure and record time, temperature, pH, turbidity, and specific conductance as each volume of well water is purged. Once the temperature, pH, and specific conductance have stabilized to within 10% for two successive well volumes and the turbidity is less than 50

4) After purging, allow static water level to recover to approximate original level.

5) Place polyethylene sheeting around well casing to prevent contamination of sampling equipment in the event equipment is dropped.

6) Obtain sample from well with a dedicated, factory pre-cleaned polyethylene Voss ™ bailer. The bailer will be suspended on a new, dedicated length of polypropylene string. The maximum time between purging and sampling will be three (3) hours. All the bailers for one day of sampling will be pre-cleaned and dedicated to each individual wells.

   Sample for VOCs first by lowering the bailer slowly to avoid degassing, then collect any other organic and inorganic samples by pouring directly into sample bottles from bailers.

   The sample preservation procedure will be to immediately place analytical samples in the cooler and chill to 4°C. Samples will be delivered to the appropriate laboratory within 24 hours. Samples will be maintained at 4°C until time of analysis.

7) Decontaminate the submersible pump and discard the pump discharge line.

8) Re-lock well cap.

Fill out field notebook, Well Sample Log Sheet, labels, Custody Seals and Chain-of-Custody forms.

C.3 SOP 3: FIELD BLANKS

Field blanks shall be taken to evaluate the cleanliness of groundwater sampling equipment, sample bottles and the potential for cross-contamination of samples due to airborne contaminants present in the air at the site and handling of equipment and sample bottles. Field blank samples shall be performed on the groundwater sample bailers and any filtering equipment. The frequency of field blanks taken shall be one per decontamination event for each type of sampling equipment, and each media being sampled (e.g., a groundwater bailer for groundwater, and a hand auger for soil sampling), at a minimum of one per equipment type and/or media per day.

Where required, field blanks shall be obtained prior to the occurrence of any analytical field sampling event by pouring deionized or potable water over a particular piece of sampling equipment and into a sample container. The analytical laboratory shall provide field blank water and sample jars with preservatives for the collection of all field blanks. Glass jars shall be used for organic blanks. The field blanks as well as the trip blanks shall accompany field personnel to the sampling location. The field blanks shall be analyzed for the same analytes as the environmental samples being collected that day and shall be shipped with the samples taken subsequently that day.

Field blanks shall be taken in accordance with the procedure described below:

(1) Decontaminate sampler using the procedures specified in this plan.

(2) Pour distilled/deionized water over the sampling equipment and collect the rinsate water in the appropriate sample bottles.

(3) The sample shall be immediately placed in a sample cooler and maintained at a temperature of 4°C until receipt by the laboratory.

(4) Fill out sample log, labels and chain-of-custody forms, and record in field notebook.

C.4 SOP 4: TRIP BLANKS

A laboratory supplied trip blank shall be an aliquot of distilled, deionized water which shall be sealed in a sample bottle prior to initiation of each day of field work. The trip blank shall be used to determine if any cross-contamination occurs between aqueous samples during shipment. Trip blanks are analyzed for aqueous VOCs only. Glass vials (40 ml) with teflon-lined lids shall be used for VOC blanks. A trip blank shall be prepared by the laboratory prior to each day of field sampling for aqueous volatiles. The sealed trip blank bottles shall be placed in a cooler with the empty sample bottles and shall be brought to the site by the laboratory personnel. If multiple coolers are required to store and transport aqueous VOC samples, then each cooler must contain an individual trip blank.

C.5 SOP 5: MEMBRANE INTERFACE PROBE (MIP) PROCEDURES

Vertical profile borings utilizing a Membrane Interface Probe (MIP) will be installed at sixteen (16) locations outside the Fulton property to a depth of approximately 130 feet. All boreholes will be advanced using direct push technologies. Specifically, a track mounted Geoprobe Model 6610DT. Continuous readings will be collected from ground surface to the completion depth. The objective is to create a real-time plan view and cross-sectional diagram of the VOC distribution in the subsurface.

C.5.1 Sample Collection

The profiler head screws into conventional AW drill rods and will be driven into the subsurface using direct push technology. The membrane interface probe head consists of a semi-permeable membrane comprised of a thin film polymer impregnated into a stainless steel screen for support. The membrane is approximately 6.35mm in diameter. The membrane is placed in a heated block attached to the probe. This block is heated to approximately 100-120 degrees C and is raised at the leading edge to protect the membrane. Heating the block accelerates diffusion of the contaminant through the membrane. A clean carrier gas sweeps behind the membrane and carries the contaminants to the gas phase detectors at the surface. Travel time from the membrane interface to the detector(s) is approximately 30-45sec (depending on the length of trunkline and flow rate). Teflon® tubing will be attached to the internal fitting using couplings.

At the surface the detectors will measure total VOCs in the carrier gas and provide this information in real-time as an instrument response. The detectors do not provide a quantitative concentration of VOCs in the groundwater or soil. However, the response level from the detector corresponds to the amount of VOCs present in the carrier gas, which is proportional to the amount of VOCs in the soil or groundwater at the MIP location. A greater response from the detector indicates greater VOC concentrations in the subsurface.

C.5.2 Source of Water

All water used during steam cleaning operations shall be from a potable source and so designated in writing. ERM's drilling subcontractor will be solely responsible for obtaining all permits from the local water purveyor and any other concerned authorities, and provision of any required back-flow prevention devices.

C.5.3 Borehole Abandonment

The borings will be properly abandoned to prevent an artificial conduit for vertical groundwater flow through any confining layers. After sampling, the borehole beneath the water table will be sealed by pumping a high-solids grout down the inside of the rods and out the bottom by displacing a disposable steel point. The grout is pumped down the rods while the rod assembly is withdrawn from the hole in a process known as retraction grouting. The rod assembly will then be removed from the borehole and the excess drill cuttings and the tailings from the unused portion of the samples will be placed back down the borehole. The remaining two feet will be filled with cement/bentonite grout, consisting of 5.0 pounds of high grade bentonite for each 94 pounds of Type I or Type II Portland cement mixed with 8.3 gallons of water for a target density of 13.9 pounds/gallon with an acceptable range of 13.4 to 14.5 pounds/gallon. Boreholes constructed in paved areas will then be repaired with an asphalt patch.

C.5.4 Work Site Restoration

Upon completion of the work, the drilling subcontractor shall restore all work areas/drilling locations to a pre-drilling condition. The drilling subcontractor shall remove and dispose of all debris, remove all equipment and materials from the each work site promptly and leave the location in a neat and orderly fashion to the satisfaction of ERM's representative. The restoration shall include repair of any holes, trenches, tire ruts, damage to pavement, etc. caused by the movement or operation of the drilling subcontractor's equipment.

C.6 SOP 6: WATERLOO VERTICAL PROFILE BORINGS WITH GROUNDWATER SAMPLING PROCEDURES

Vertical profile borings with groundwater sampling utilizing the Waterloo Groundwater profiler will be installed at eight (8) locations to characterize groundwater quality/impacts. The Vertical Profile, groundwater samples will be collected from up to 10 intervals per boring. The sampling intervals will be selected based on a combination of MIP data and relative permeability data generated using the Waterloo^{APS}. This technique will refine the understanding of the plume configuration.

All boreholes will be advanced using direct push technologies. Specifically, a track mounted Geoprobe Model 6610DT. The Profiler will be driven to the water table where continuous relative permeability data collection will begin. Up to ten (10) groundwater samples will be collected at each borehole at interval chosen based on a combination of MIP data and relative permeability data generated using the Waterloo^{APS}.

C.6.1 Sample Collection

ERM's drilling subcontractor will be responsible for provision of the Waterloo sampling tool and all necessary accessory items (reusable and disposable) to collect groundwater samples.

The effectiveness of the Waterloo Profiler is based on the premise that the device causes minimal drag down of contamination as it is driven through high contaminant zones into zones of little or no contamination.

The Profiler head consists of a stainless steel drive point with six 5/32-inch diameter circular ports fitted with stainless steel screens. Screen mesh sizes may be selected based on the grain size of the aquifer materials. The ports convey water into a common internal fitting tip. Teflon® tubing is attached to the internal fitting using couplings. The Teflon® tubing is used in a disposable continuous length. The small storage volume in the profiler and conduit tubing provides rapid transmission of the water sample to the surface. Sample bottles are fitted into stainless steel sampling caps in which an airtight seal is obtained. Because of the depth of the water table, groundwater samples will be collected using a nitrogen airlift pump. Purging of the sample containers prior to sampling ensures that formation water exists in the vials at the time of collection. The sampling tubing is protected in the AW drill rod used to drive the tip.

While the Profiler is being driven, contaminant free water, such as distilled water, is pumped down the tubing and out the small ports to purge the Profiler of formation water from the previous sampling interval and to prevent clogging of the ports. As the Profiler reaches sampling depth, the pump is reversed to begin pumping water to the surface, minimizing the introduction of foreign water to the zone to be sampled. Prior to collection, the ports are developed and the system is purged. The amount of water introduced into the formation is monitored during drilling. Samples are collected after the water introduced into the formation is recovered and field parameters such as oxidation-reduction potential, temperature, pH, dissolved oxygen and specific conductance stabilize.

Initially the continuous point sampler will be advanced to just above the designated sample depth starting with the upper most sample in the profile. The Profiler will be used to collect groundwater samples at multiple depths without tripping out the tool. The Profiler will be advanced in ten-foot intervals to collect the groundwater samples using the procedures presented above. Waterloo groundwater sample collection will continue to the borehole termination depth to be determined in the field. Once the desired numbers of sample have been obtained, the Profiler will be tripped out of the boring. If necessary, the Profiler will be properly decontaminated and re-introduced into the borehole to collect additional samples.

A New York State Department of Health (NYSDOH) Environmental Laboratory Accreditation Program (ELAP) -certified laboratory will analyze the groundwater samples obtained from these locations for USEPA SW-846 Method 8260.

C.6.2 Borehole Abandonment

The borings will be properly abandoned to prevent an artificial conduit for vertical groundwater flow through any confining layers. After sampling, the borehole beneath the water table will be sealed by pumping a high-solids grout down the inside of the rods and out the bottom by displacing a disposable steel point. The grout is pumped down the rods while the rod assembly is withdrawn from the hole in a process known as retraction grouting. The rod assembly will then be removed from the borehole and the excess drill cuttings and the tailings from the unused portion of the samples will be placed back down the borehole. The remaining two feet will be filled with cement/bentonite grout, consisting of 5.0 pounds of high grade bentonite for each 94 pounds of Type I or Type II Portland cement mixed with 8.3 gallons of water for a target

density of 13.9 pounds/gallon with an acceptable range of 13.4 to 14.5 pounds/gallon. Boreholes constructed in paved areas will then be repaired with an asphalt patch.

C.6.3 Work Site Restoration

Upon completion of the work, the drilling subcontractor shall restore all work areas/drilling locations to a pre-drilling condition. The drilling subcontractor shall remove and dispose of all debris, remove all equipment and materials from the each work site promptly and leave the location in a neat and orderly fashion to the satisfaction of ERM's representative. The restoration shall include repair of any holes, trenches, tire ruts, damage to pavement, etc. caused by the movement or operation of the drilling subcontractor's equipment.

C.7 SOP 7: PUMP TEST PROCEDURES

A pre-design hydraulic evaluation of groundwater flow dynamics within the capture field of Village of Garden City Public Supply Well Nos. 9, 13 & 14 will be preformed during the RD. These data will be used as a basis to better understand local groundwater response to pumping of the wells in constructing a preliminary groundwater flow model, identify data gaps, potential new monitoring well locations, and the design of the recovery wells.

Groundwater monitoring wells MWs 20A, 20B, 20C, 21A, 21B, 21C, 22A, 22B, 22C, 23A, 23B, 23C, GCP 14S, GCP-14D, and GCWD Well Nos. 9, 13 & 14 will be outfitted with water level data loggers. Presuming cooperation with the GCWD, the pumping of the three public supply wells during the time these aforementioned monitoring wells are recording water levels will be documented. Preferably, the Village of Garden City Water Department will coordinate pumping of GCWD Well Nos. 9, 13 and 14 during certain times to ensure the three public supply wells are pumped in various combinations as well as simultaneously for specified periods of time to optimize the quality of the data set obtained from this activity. The various pumping scenario combinations will, to the extent practicable, represent potential operating scenarios, e.g., 9 on/13 off/14 on, 9 on/13 on/14 off, etc. Again, the cooperation of the Village of Garden City Water Department will be required to effectively implement this pre-design task.

C.7.1 Transducer/Datalogger Installation

Pressure-sensitive transducer/datalogger, including one barometric pressure transducer/datalogger, that are compatible with both water quality and anticipated pressure-sensitivity range in a given well will be installed in the above mentioned monitoring wells during each pump test, and will record measurements at a frequency of one per minute.

The transducer/datalogger shall be installed by the geologist/environmental engineer in accordance with the manufacturer's instructions. The transducer/datalogger will be suspended inside the well casing by a length of polyethylene string and secured with plastic tie strips to the riser casing or protective well casing. The plastic tie strip shall be positioned such that it does not interfere with closing and locking of the wells protective casing.

C.7.2 Decontamination Procedures

Prior to installation and upon removal from the well each logger will be decontaminated in accordance with SOP 12A.

C.8 SOP 8: GEOLOGIC BORING DRILLING PROCEDURES

C.8.1 Borehole Construction

All temporary well boreholes will be constructed using 4.25 inch ID hollow stem augers. In order to reduce the potential for "running sands", a hydraulic head of potable water will be applied within the augers when the water table is encountered to maintain a positive hydrostatic head on subsurface materials. Each borehole will be advanced to the prescribed completion depth below grade. Cuttings generated from the construction of the boreholes will be contained in New York State Department of Transportation (NYSDOT) approved 55-gallon ring-top drums. The drums will be labeled according to the borehole/temporary well number. All drums will be staged within the fenced storage area along the southern boundary of the Fulton Avenue site.

C.8.2 Borehole Sampling

During the construction of each temporary well borehole, split-spoon samples will be obtained at 10-foot intervals beginning at the water table to the prescribed completion depth for geologic description to characterize the subsurface lithology beneath and downgradient of the Fulton Avenue site. Augers shall be advanced to the sample collection depth and a split-spoon sampler shall be deployed ahead of the lead auger following ASTM Method D1586. Split-spoons shall be advanced by either the wire-line method (downhole cable hammer) or with a cathead and standard 140 pound hammer simulating a free-fall of 30 inches. The soil samples shall be collected using a 2-foot by 2-inch carbon steel split-spoon sampler driven by a 140 lb. hammer dropped 30 inches repeatedly. An ERM Hydrogeologist shall examine and identify the sample immediately upon collection. The sample shall also be screened for volatile organic compound contamination using a hand-held PID total organic vapor analyzer.

C.8.3 Borehole Logging

The hydrogeologist, who shall use visual and field test criteria to classify the soils, shall examine each split-spoon sample. The cuttings brought to the surface during the drilling shall also be:

- screened for volatile organic compound contamination using a hand-held PID total organic vapor analyzer, and
- examined for any physical soil characteristics that may have varied between samples.

A standard "Geologic Log" shall be maintained for each boring that shall include all of the geological information gathered in the field, including the following:

- The structure of the soils sampled, including layering stratification features, and the dominant soil types.
- The color of soils, using Munsell Soil Color Charts.
- The moisture content of soils.
- Soil grain features, including grain sizes, degree of sorting or grading, angularity, and mineralogy. The soils shall be classified using the American Society for Testing and Materials (ASTM) Method D2488-84, a visual manual procedure.
- Identification of any rock fragments, organic material or other components.

The consistency of clay-dominated soils.

All of the soils information collected shall be recorded as a designation under the Unified Soil Classification System (USCS) along with additional observations for each distinctive soil type within each sample. All soil samples shall be stored in glass or plastic jars supplied by the drilling subcontractor. The hydrogeologist shall label the jars with well number, sample interval and date. In addition to the visual logging of the soils, a natural gamma geophysical log will be run inside of the augers upon completion of the boring. The results will be plotted on a linear graph in the field to confirm the geologic structure of subsurface soil.

C.9 SOP 9: TEMPORARY WELL INSTALLATION AND SAMPLING PROCEDURES

C.9.1 Temporary Well Construction

After the borehole has been completed it will be measured with a tape measure to ensure that no "running sands" have entered the augers and that the borehole is clear. Following this procedure a 5-foot, steam cleaned reusable stainless steel well screen attached to black steel riser pipe shall be lowered into the borehole and set at the prescribed completion depth. The augers shall then be withdrawn to allow the formation to collapse and create a seal around the well screen.

C.9.2 Temporary Well Sampling

Prior to purging and sampling, the static water level in the temporary well shall be measured and the volume of standing water in the well shall be calculated. A small diameter stainless steel submersible pump (variable speed Grundfos submersible pump) and dedicated lengths of new polyethylene tubing shall be lowered into the well casing and placed just above the well screen for purging and sampling the well. The submersible pump shall be decontaminated between each well location and each sampling interval utilizing an Alconox wash and potable water rinse followed by a deionized water rinse. At least three well casing volumes shall be purged until the pH, temperature, and specific conductance have stabilized to within 10% for two (2) successive well volumes before a groundwater sample is to be collected. Dissolved oxygen (DO) will also be measured at the conclusion of purging. The pump rate for sampling VOCs shall be less than or equal to 100 ml/minute. These samples shall be preserved by chilling to 4°C and held at this temperature until analyzed by the laboratory.

Following sample collection, the temporary well screen and riser pipe shall be withdrawn 10 feet to the next ground water sampling interval. The submersible pump will be lowered back into the well casing and another sample collected. All sampling measurements shall be recorded on a Field Sampling Log.

This procedure shall continue sequentially until all ground water quality screening samples (up to and including the water table) are collected. All of the samples will be analyzed for volatile organic compounds by USEPA Method 8260B. Duplicate samples will be collected at a frequency of one per twenty samples and will be analyzed for volatile organic compounds by NYSDEC ASP CLP Method 95-4 or 95-1 as described in Section 2.16.1.

Purge water will be collected in a tanker truck, transported to the Fulton Avenue site and pumped into a temporary frac tank. Analytical results from groundwater sampling and samples collected from the frac tank will determine the ultimate disposition of the purge water.

C.9.3 Temporary Well Borehole Abandonment

Once the temporary well has been sampled back to the water table, the remaining steel casing and screen will be withdrawn from the borehole. The borehole shall be allowed to collapse and will be backfilled with cuttings removed during construction of the borehole or clean sand. Grouting of the boreholes will not be required. Boreholes constructed in paved areas will then be repaired with an asphalt patch.

C.10 SOP 10: POTABLE WATER BLANKS

Quality Assurance samples of the potable water used for drilling operations shall be sent for analysis at the start of field activities and at least once every two weeks thereafter to demonstrate the water is analyte-free. If analytical results indicate the presence of a contaminant of concern in a quality assurance sample, then the analytical results of samples collected from those wells installed using the corresponding potable water shall be suspect. These samples shall be analyzed for the same compounds as the groundwater environmental samples.

The following procedure shall be used to collect potable water blanks:

- Pour potable water into an extra vial. The sample shall be immediately placed in a sample cooler and maintained at a temperature of 4°C until receipt by the laboratory.

- Complete sample log, labels, custody seals and Chain-of-Custody forms. Record in field notebook.

C.11 SOP 11: DECONTAMINATION PROCEDURES

As presented below, all drilling and field sampling equipment shall be decontaminated prior to use and/or sampling.

C.11.1 Decontamination of Sampling Equipment

Split-spoons shall be decontaminated between sampling intervals as follows:

- Potable water rinse.
- Alconox and water detergent and potable water scrub.
- Potable water rinse.
- Lay on or wrap equipment in clean polyethylene sheeting until use.

The submersible sampling pumps that are placed in the borehole shall be decontaminated with an Alconox detergent rinse and by pumping approximately 20 gallons of potable water through the pump. Since dedicated new lengths of polyethylene tubing shall be used for sampling each well, the tubing shall not be decontaminated. Unless otherwise specified, the submersible pumps shall be decontaminated prior to the sampling the first well and between each subsequent well as follows:

- Potable water rinse.
- Alconox detergent and potable water scrub.
- Potable water rinse.
- Distilled/deionized water rinse.
- Wrap in aluminum foil, shiny side facing out.

Unless otherwise specified, all non-detect sampling equipment utilized to obtain groundwater environmental samples for chemical analyses (e.g., stainless steel bailers) shall be decontaminated between sampling points as follows:

- Potable water rinse.
- Alconox and water detergent and potable water scrub.
- Potable water rinse.
- Methanol (at least pesticide grade) rinse: Light spray to minimize material used. Segregate and store rinsate separately.
- Distilled/deionized water rinse.
- Air dry.
- Wrap or cover in aluminum foil shiny side facing out.

C.11.2 Driller's/Heavy Equipment Decontamination

All drilling equipment and the back of the drilling rig shall be decontaminated by steam cleaning prior to performance of the first boring/well installation and between all subsequent borings/well installations. This shall include all hand tools, casing, augers, drill rods and bits, tremie pipe and other related tools and equipment. The steam cleaning equipment shall be capable of generating live-steam with a minimum temperature of 212°F.

All water used during drilling and/or steam cleaning operations shall be from a potable source and so designated in writing. The drilling contractor is responsible for obtaining all permits from the local potable water purveyor and any other concerned authorities, and provision of any required back-flow prevention devices. The equipment shall be cleaned to the satisfaction of the ERM Hydrogeologist.

All well casing and screen shall be steam cleaned, wrapped in clean polyethylene sheeting and stored until the time of well construction.

Extraneous contamination and cross-contamination shall be controlled by wrapping the sampling equipment with aluminum foil when not in use and changing and disposing of the sampler's gloves between samples. Decontamination of sampling equipment shall be kept to a minimum in the field, and wherever possible, dedicated sampling equipment shall be used. Personnel directly involved in equipment decontamination shall wear appropriate protective equipment.

C.12 SOP 12: WASTE MANAGEMENT AND DISPOSAL

The following section describes the handling and ultimate disposal of solid and liquid wastes generated during the field activities. Waste generated is expected to consist of trash (boxes, paper, etc.), auger cuttings, decontamination wash water, purge water, and used protective clothing.

The PCE in soil and ground water at the Fulton Avenue site is a listed hazardous waste. Accordingly, its derived-from solid and liquid wastes are considered hazardous for handling and disposal purposes. In regards to disposal, disposal options for generated wastes will depend on contaminant levels in the waste. The following standards and regulations have been identified as being applicable, relevant and appropriate to any removal, management, and off-site or on-site disposal of Fulton Avenue site RI generated waste materials:

NYSDEC's RCRA TAGM #3028 on "Contained-In Criteria for Environmental Media" {November 30, 1992};

- 40 C. F.R. Part 262 (Standards Applicable to Generators of Hazardous Waste);
- 40 C. F. R. Part 263 (Standards Applicable to Transporters of Hazardous Waste;
- 40 C. F. R. Part 264 (Standards for Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities); and
- 40 C. F. R. Part 268 (Land Disposal Restrictions)

Accordingly, handling and disposal will be as follows:

- Non-contaminated trash and debris will be placed in a trash dumpster and disposed of by a local garbage hauler.
- Non-contaminated protective clothing will be packed in plastic bags and placed in a trash dumpster for disposal by a local garbage hauler.
- Cuttings from soil borings and monitoring well installations will be collected on plastic sheeting and stored in a roll-off container or drums staged in the secure, fenced area along the southern boarder of the Fulton Avenue site established for that purpose. Subsequent

sampling of the material will be conducted to determine its classification for disposal purposes. The soils will then be disposed of in accordance with any applicable federal and state regulation in addition to those referenced above by a waste subcontractor.

- Liquids generated from equipment decontamination, temporary well purging and permanent ground water monitoring well development will be collected in a tanker truck at the point of generation, transported to the Fulton Avenue site, and stored in a temporary frac tank. Purge water from sampling the permanent ground water wells will be collected in 55-gallon drums or a tanker truck, hauled to the Fulton Avenue site for transfer into the frac tank at the Fulton Avenue site. Subsequent sampling of the material will be conducted to determine its classification for disposal purposes. It is intended that these liquids will not be stored in the frac tank for more than 90 days in order to comply with applicable RCRA storage regulations.

- Used protective clothing and equipment that is suspected to be contaminated with hazardous waste will be placed in plastic bags, packed in 55-gallon ring-top drums, and disposed of in accordance with any applicable federal and state regulation in addition to those referenced above by a waste subcontractor.

ATTACHMENT D - Laboratory SOPs

Accutest Laboratories
Standard Operating Procedure

Lab Manager_____

QA Manager_____

Effective Date:_____

TITLE: **PURGE-AND-TRAP EXTRACTION FOR AQUEOUS SAMPLES**

REFERENCES: **SW-846 5030B (Revision 2, December 1996)**

Revised Sections: Added 6.2, 7.6, 7.7, 8.2, 10.2.5.1.1, 10.2.7.2, 10.2.8 & 12.0; Modified 1.0, 3.0, 4.0, 5.3, 6.0, 7.5, 8.0, 10.0 & 11.0

1.0 SCOPE AND APPLICATION

1.1 This method describes a purge-and-trap procedure for the analysis of volatile organic compounds (VOCs) in aqueous samples, water miscible liquid, and high-level soil and waste sample extracts prepared in Method 5035. This method is used in conjunction with gas chromatographic determinative methods SW8015, SW8021, and SW 8260.

1.2 Method 5030 can be used for most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

1.3 Water samples can be analyzed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water can be determined by direct injection of the sample into the chromatographic system or by dilution of the sample prior to the purge-and-trap process.

2.0 SUMMARY OF METHOD

2.1 Aqueous Samples: An inert gas is bubbled through a portion of the aqueous sample at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.

2.2 High Concentration Extracts from Method 5035: An aliquot of the extract prepared in Method 5035 is combined with organic free reagent water in the purging chamber. It is then analyzed by purge-and-trap GC or GC/MS following the normal aqueous method.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Not applicable. See determinative method.

4.0 DEFINITIONS

BATCH - a group of samples which are similar with respect to matrix and testing procedures being employed and which are processed as a unit. A sample batch is a maximum of 20 samples which can be prepared and analyzed over a period of one day.

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank; Instrument Blank, Storage Blank, and Sulfur Blank.

MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards, that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

REAGENT WATER - water in which an interferant is not observed at or above the minimum detection limit of the parameters of interest.

TRIP BLANK - a sample of analyte free matrix taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures.

5.0 HEALTH & SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor/Health and Safety Officer.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses.

5.3 Primary standards of the toxic compounds should be prepared in a hood. A NIOSH/Mass approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Collect samples in 40 ml glass screw-cap VOA vials with Teflon-faced silicone septum. The vials and septum should be washed and rinsed with distilled deionized water, then baked in oven at 105 °C for approximately one hour. Do not heat the septum for more than one hour, because the silicone begins to slowly degrade at 105 °C.

6.2 Test all samples for residual chlorine using test paper for free and total chlorine. If samples contain residual chlorine, three milligrams of sodium thiosulfate should be added for each 40 ml of water sample.

6.3 Preserve samples with HCl to pH $\leq$2. Samples received unpreserved must be so noted on the chain of custody. The sample manager must notify the project manager of the non-conformance, who in turn notifies the client for additional instructions. The non-conformance is documented in the report narrative.

6.4 Store samples with minimum headspace, at 4 ^{o}C or less in an area free of solvent fumes. The size of any bubble caused by degassing upon cooling the sample should not exceed 5 - 6 mm. When a bubble is present, also observe the cap and septum to ensure that a proper seal was made at time of sampling. If the sample was improperly sealed, the sample should be discarded.

6.5 Samples must be analyzed within 14 days of collection.

7.0 APPARATUS AND MATERIALS

7.1 Microsyringes - 10-µL, 25-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL.

7.2 Syringe valve - Two-way, with Luer ends.

7.3 5-mL glass hypodermic syringes with Luer-Lok tip.

7.4 Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers.

7.5 Purge-and-trap device.

7.5.1 The following autosampler models are used for purging, trapping, and desorbing the sample into GC column.

- O.I. Model 4560 sample concentrator with 4551 vial multi-sampler.
- O.I. Model 4560 sample concentrator with 4552 Water/Soil multi-sampler.

7.5.2 The sample purge chamber accepts 5 ml samples with a water column at least 3 cm deep.

7.5.3 The Archon auto sampler is equipped with a heater capable of maintaining the purge chamber at 40 °C (± 1°C) is to be used for low level soil/sediment analysis, but <u>not</u> for water or medium level soil/sediment analyses.

7.5.4 The trap is 25 cm long with an inside diameter of 0.105 inch, which is purchased from a commercial vendor. It contains Tenax, silica gel, and a carbon molecular sieve. Before initial use, the trap should be conditioned at 180°C for 30 minutes by backflushing with at least 20 ml/minute flow of helium.

7.5.4.1 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analyte of interest. Whichever trap is employed, it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in this method.

7.5.5 The desorber should be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 210 °C during bakeout mode.

7.6 Vials - 40 ml with Teflon lined septa.

7.7 pH papers.

8.0 REAGENTS

8.1 Reagent Water

8.1.1 Reagent water is defined as water in which an interferant is not observed at the method detection limit of the parameters of interest.

8.1.2 Reagent water is generated by using a multi-element deionizing system consisting of a particulate filter, twin mixed bed ion exchange resin columns, and a carbon-polishing column.

8.2 Methanol - purge-and-trap grade or equivalent.

8.3 Refer to the determinative method SOP for specifications on internal and surrogate standards.

9.0 INTERFERENCES

9.1 The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

9.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

9.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to cross contamination from highly concentrated samples. Frequently bake-out and purge the entire system on a routine basis.

9.4 Special precautions must be taken to control methylene chloride sources. All GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Organics prep staff whose clothing is routinely exposed to methylene chloride and other solvent vapors during sample preparation are banned from wearing their lab coat in the GC/MS laboratory.

10.0 PROCEDURE

10.1 The purge-and-trap technique for aqueous samples is found in Sec. 10.2 and guidance for analysis of solvent extracts from the High Concentration Method in Method 5035 is found in Sec. 10.3. The samples prepared by this method may be analyzed by Methods 8015, 8021, and 8260. Refer to these methods for appropriate analysis conditions.

10.2 This section provides guidance on the analysis of aqueous samples and samples that are water miscible, by purge-and-trap analysis.

10.2.1 Condition the trap according to manufacturer's instructions upon installation. Routine Daily maintenance must be performed before any tuning, calibration or sample analysis activities are initiated. These include checks of the following items:

- Clean & bake purge tube.
- Bake trap and transfer lines.
- Check or refill internal/surrogate spike solution on SIM/SAM vials.
- Clean/replace syringe (if necessary).
- Change and refill rinse bottle.
- Empty and rinse waste bottle.

10.2.2 Prior to using this introduction technique, the GC system must be calibrated. The procedures for the determinative methods give details on preparation of standards for initial and daily calibrations. The GC/MS methods require instrument tuning prior to proceeding with calibration.

10.2.2.1 Transfer and fill up (no air space) each standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray.

10.2.2.2 Program the autosampler to add surrogate and internal standard spiking solution to each standard.

10.2.2.3 Follow the purge-and-trap analysis as outlined in Sec. 10.2.4.

10.2.2.4 Calculate response factors (RF) or calibration factors (CF) for each analyte of interest. Follow the specific calibration requirements as described in the determinative method SOP.

10.2.3 Sample Screening

10.2.3.1 Screening of the sample prior to purge-and-trap analysis may provide guidance on whether sample dilution is necessary and may prevent contamination of the purge-and-trap system.

10.2.3.2 The Tekmar 7000/7050 headspace autosampler is utilized by Accutest for screening. The system functions by direct sampling of the heated sample headspace by direct injection onto the GC column equipped with a flame ionization detector (Method 3810).

10.2.4 Sample Introduction and Purging

10.2.4.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

10.2.4.2 Adjust the purge gas flow rate to 25-40 mL/min on the purge-and-trap device. Once the flow is optimized, it is not necessary to set the flow daily, although periodic checking is recommended.

10.2.4.3 Using O.I.Model 4560 sample concentrator with 4551 or 4552 vial multisampler,

- Place the 40 ml vial in the tray, or
- Load 5ml sample into purge tube if sample volume limited.

10.2.4.4 Program the autosampler to inject the internal standard (if applicable) and surrogate solution into the robotic syringe used to withdraw sample from the 40 ml vial.

10.2.4.5 Purge the sample for the time and at the temperature specified in the determinative method SOP.

10.2.5 Sample Desorption

10.2.5.1 Place the purge-and-trap system in the desorb mode, and rapidly heat the trap to the temperature specified in the determinative method SOP while backflushing with helium for the method specific time. Simultaneously begin the temperature program of the gas chromatograph and start the data acquisition.

10.2.5.1.1 Desorb time may require performance optimization between 2.0 and 4.0 minutes as dictated by trap manufacturer's specifications or instrument characteristics.

10.2.5.2 Program the purge and trap system to automatically rinse purge tube at least twice with heated reagent water between analyses to avoid carryover of target compounds. For samples containing large amounts of water-soluble materials, suspended solids, high-boiling compounds, or high purgeable

levels, it may be necessary to wash out the purging device with methanol solution between analyses, rinse it with distilled water.

10.2.6 Trap Reconditioning

10.2.6.1 Recondition the trap by returning the purge-and-trap device to the purge mode. Maintain the trap temperature and bake time as specified in the method SOP.

10.2.7 Sample Dilution

10.2.7.1 If the concentration of any target compound in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and reanalyzed.

10.2.7.2 Establish the dilution of sample in order to fall within the calibration range:

- Use FID screen data.
- Employ data from undiluted sample analysis.
- Use historical site data.
- Use sample characteristics (i.e. appearance, odor) as initial guidance.

10.2.7.3 The following procedure is appropriate for diluting purgeable samples. Until the dilute sample is in a sealed sample vial, all steps in the dilution procedure must be performed without delay.

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10.2.7.3.1 Dilutions may be made in volumetric flasks (10-mL to 100- mL). Intermediate dilutions may be necessary for extremely large dilutions.

10.2.7.3.2 Calculate the approximate volume of reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask.

10.2.7.3.3 Inject the proper aliquot of samples from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times.

10.2.7.3.4 Fill a 40 ml sample vial and seal with a Teflon baked silicon septa, load the diluted sample into the autosampler and analyze.

10.2.8 pH Verification.

10.2.8.1 Once the sample analysis is completed or before preparing sample dilution, the pH of the sample must be determined to ensure that all VOA samples were properly preserved in the field. Put one or two drops of sample directly on a piece of pH paper. Check the pH. Any sample with a pH greater than 2 will be recorded accordingly on the instrument run log and will be reported with a footnote indicating the sample preservation deficiency.

10.2.9 Analysis of water-miscible liquids

10.2.9.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with organic-free reagent water.

10.3 This section provides guidance on the analysis of solvent extracts from High Concentration Samples prepared by Method 5035.

10.3.1 Low-level soil method.

10.3.1.1 Weigh out 5 g of each sample into a labeled vial. Add 5 ml of reagent water and cap the vial quickly. Transfer the 40 ml vial to the autosampler tray. Stir and heat the sample at the time of analysis.

10.3.2 Medium-level soil method.

10.3.2.1 Select the volume of methanol extract to add to the 50 ml of reagent water for analysis. If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-concentration analysis to determine the appropriate volume.

10.3.2.2 Aliquot 5ml of the solution into the 40 ml vial and cap with Teflon septum, then place the vial onto the autosampler.

10.3.3 Proceed with the analysis as outlined in the specific determinative method. Analyze all reagent blanks on the same instrument as that used for the samples. The standards and blanks should also contain the same amount of methanol to simulate the sample conditions.

11.0 QUALITY CONTROL

11.1 Analyze a method blank and blank spike at a rate of one per day or every twenty samples, whichever is more frequent.

11.2 A matrix spike/matrix spike duplicate is required for every 20 samples. Client specific requirements may specify a MS/MSD per day.

12.0 DOCUMENTATION

12.1 The Analytical Logbook records the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

12.1.1 If samples require reanalysis, a brief explanation of the reason must be documented in the Comments section.

12.2 Standards Preparation Logbook must be completed for all standard preparations. All information must be completed; the page must be signed and dated by the appropriate person.

12.2.1 The Accutest lot number must be cross-referenced on the standard vial.

12.3 Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

12.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

12.5 Unused blocks of any form must be X'ed and Z'ed by the analyst before submitting the data for review.

12.6 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information should also be verified during this review.

13.0 POLLUTION PREVENTION & WASTE MANAGEMENT

13.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 13.2.

13.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, ESM003. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

13.2.1 Non hazardous aqueous wastes.

13.2.2 Hazardous aqueous wastes

13.2.3 Chlorinated organic solvents

13.2.4 Non-chlorinated organic solvents

13.2.5 Hazardous solid wastes

13.2.6 Non-hazardous solid wastes

Lab Manager:_____

QA Manager:_____

Effective Date:

TITLE: TCLP - VOLATILES EXTRACTION

METHOD REFERENCES: SW846 1311

Revised Sections: 13.2

1.0 SCOPE AND APPLICATION

1.1 The Toxicity Characteristic Leaching Procedure (TCLP) utilizes a zero-headspace extraction device to evaluate the presence and mobility of volatile organics for waste characterization.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes containing less than 0.5 % solids, the sample is filtered through a 0.6 to 0.8 um filter and the filtrate is defined as the TCLP leachate. All filtrations are done in a zero headspace extractor/filtration device to minimize exposure of the sample to the air.

2.2 For solid samples, the solid portion of the sample is extracted by adding extraction fluid equal 20 times the weight of the sample and rotating the sample for 18 $\pm$ 2 hours at 30 rpm. All filtrations are done in a zero headspace extractor/filtration device to minimize exposure of the sample to the air. After leaching, the sample is filtered through 0.6 to 0.8 um filter paper and the filtrate is analyzed for volatile organics.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Not applicable for this method. Refer to determinative methods for reporting limit and method detection limit information.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to

generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent should be used.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

ORGANIC-FREE REAGENT WATER: For semivolatiles and nonvolatiles, all references to water in the methods refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10

times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIME

6.1 The samples are stored at 4^{o}C prior to extraction. The samples should be collected with no headspace. No preservatives should be added to the samples for TCLP volatile extraction. The TCLP extract should be stored at 4^{o}C after filtration and prior to analysis.

6.2 All volatiles must be leached within 14 days of the date of collection.

7.0 INTERFERENCES

7.1 Refer to the individual methods for the analytes of interest for discussion of interferences.

8.0 APPARATUS

Below is a summary of the apparatus to be used for extraction of samples for volatile analyses.

8.1 Zero Headspace Extractor (ZHE)- Analytical Testing and Consulting Services, or equivalent. A pump is normally required to add the extraction fluid to the vessel. This vessel should have an internal volume of 500 to 600 ml and be able to accommodate a 90-110 mm filter. The vessel must be gas tight and free of organic contaminants. It is

strongly recommended that all ZHE's be fitted with a pressure valve to check for leaks. The o-rings must be checked and replaced on a regular basis.

8.1.1 ZHE extractors must be leak tested before first being placed into use and whenever a loss of pressure during an extraction is observed. To leak test an extractor, pressurize the ZHE to 50 psi and allow it to stand unattended for one hour, and recheck the pressure.

8.1.1.1 If pressure is lost, check all fittings and inspect and replace o-rings and repeat the leak test process.

8.1.1.2 If the pressure still does not hold in a range from 45 to 50 psi, then pressurize the extractor to 10 psi and allow it to stand unattended for one hour and recheck the pressure. If it does not hold at 10 psi, the ZHE must be removed from all service until the problem is resolved.

8.1.1.2.1 Any pressure of < 45 psi is considered failing the 50 psi check and must be also checked at the 10 psi level.

8.1.1.2.2 Any pressure of < 10 psi is considered failing the 10 psi check and must be taken out of service until it is repaired.

8.1.1.3 If the ZHE will hold pressure at 10 psi and can maintain a filtering pressure of 50 psi when gas pressure is applied, then it can be used for extractions. Notify a team leader or supervisor that further maintenance is required on this extractor.

8.2 Rotary Agitation Device - Analytical Testing and Consulting Services or equivalent. Must be capable of rotating the extraction vessel in an end-over-end fashion at 30 $\pm$ 2 rpm. The rotation rate should be checked and recorded at least once per week on the analysis worksheets.

8.3 Filters - Whatman GF/F; 90-110 mm, or equivalent. The filters should be borosilicate glass fiber filters with an effective pore size of 0.6 to 0.8 um. Pre-filters should not be used.

8.4 pH meter - capable of reading $\pm$ 0.05 pH units

8.5 Balance - capable of weighing $\pm$ 0.01 g and with a range up to approximately 150 g or higher.

8.6 Glass, gas-tight syringes.

8.7 40 ml volatile vials

8.8 Filtration device - Millipore Corp., YT 3014214w; 142 mm, or equivalent. For use in the determination of the solids content of the sample. This device must have a minimum

internal volume of 300 ml and be equipped to handle a 142 mm diameter filter. It must be made of inert materials which will not leach or absorb waste components.

8.9 Filters - Whatman GF/F; 142 mm, or equivalent. For use in the determination of the solids content of the sample. The filters should be borosilicate glass fiber filters with an effective pore size of 0.6 to 0.8 um. Pre-filters should not be used.

8.10 Thermometer, calibrated against an NIST traceable thermometer. To record room temperature.

8.11 Variable speed mechanical pump - Environmental Express Model TP1200, or equivalent.

9.0 REAGENTS

All reagents should be prepared from reagent grade chemicals unless otherwise specified.

9.1 Organic Free Water: ASTM Type II or equivalent. Use DI water from the taps in the GC lab for all volatile extractions.

9.2 Sodium Hydroxide, 1N; Dissolve 40.0 grams of NaOH in 500 ml DI water in a 1 liter flask. Dilute to volume and mix. CAUTION: The solution will become warm.

9.3 Glacial Acetic Acid, CH_3CH_2OOH, Reagent Grade

9.4 Extraction Fluid #1: Add 5.7 ml of glacial acetic acid to 500 ml of DI water in a 1 liter flask. Add 64.3 ml of 1N sodium hydroxide, and dilute to 1 liter. The pH of this solution must be within the range of 4.93 $\pm$ 0.05 to be used. The expiration date for this extraction fluid is 2 weeks from the date of preparation.

10.0 PROCEDURE

Below is the procedure to be followed for the TCLP extraction of volatile analytes.

10.1 If the waste will obviously yield no liquid when subjected to pressure filtration, proceed to Section 10.3. For example, for samples that are solids with no free liquids, proceed to Section 10.3.

10.2 If the waste is a liquid or multiphasic, proceed as follows, using the pressure filtration device.

10.2.1 Pre-weigh the filter and the container that will hold the sample. Document all weights on the leachate form.

10.2.2 Assemble the filtering apparatus as per the manufacturer's instructions.

10.2.3 Weigh a 100 gram subsample of the waste and record the weight. Note: There may be limited sample volume available for the volatile extraction and analysis. In that case, the aliquot used to determine the solids content of the sample should be taken from one of the bottles intended for non-volatile analysis.

10.2.3.1 If there is insufficient volume to do this characterization with 100 g, then proceed with a smaller sample aliquot and note on the extraction log that a smaller aliquot was used for percent solids characterization.

10.2.4 Quantitatively transfer the subsample to the filtering apparatus. Slurries may be allowed to settle and the liquid portion filtered prior to transferring the solid portion of the waste. NOTE: If waste material has adhered to the sample container, obtain the weight of this residue and subtract from the total weight of the waste.

10.2.5 Complete the assembly of the filtration device, and gradually apply pressure until fluid is expelled. If no fluid is expelled, gradually increase the pressure in 10 psi increments to a maximum of 50 psi. If no fluid is expelled in a 2 minute period, stop the filtration. Shut off the pressurizing gas and vent the filtration system using the side port. If the pressure is taken too high and the filter breaks, start the procedure again will a new sample aliquot. Never use more than one filter for a sample aliquot. CAUTION: Do not remove flange clamps while system is pressurized! Serious injury may result!

10.2.6 The material in the filtration apparatus is defined as the solid phase. This material may either be a solid or a high viscosity liquid such as an oil or paint.

10.2.7 Remove the solid portion of the waste sample and the filter from the filtration apparatus. Then dry the filter and solids at 100^0 C $\pm$ 20 to a constant weight (2 successive weights within 1%). Determine the percent solids as shown below. (This drying is only required when there is filtrate entrained in the filter, but it is recommended for all wet samples.)

$$\% \text{ solids} = \frac{(W - F) \times 100}{T}$$

where W = weight of sample remaining on filter
F = weight of filter
T = initial weight of sample used

10.2.8 If the sample contains <0.5% dry solids, the filtrate is defined as the sample leachate.

10.3 Zero Headspace Extraction

10.3.1 If the waste contains <0.5% dry solids, charge the zero headspace extractor (ZHE) with 200 ml of the sample, insert the filter and supports, and seal the vessel. Raise the piston to remove any headspace present. Attach a 50 ml glass gas tight syringe to the ZHE, and raise the piston to expel approximately 45 ml of filtrate. Transfer the filtrate to a 40 ml VOA vial with a minimum of agitation, and seal the vial. Make sure that there is no headspace in the vial. Repeat the sampling to obtain three vials of filtrate. Store at 4^{o}C until analysis.

10.3.2 If the waste is 100% solid, weigh a 10 to 25 gram subsample and quickly transfer to the ZHE. (Before weighing and loading the sample, make sure that the base of the ZHE has been adjusted upwards so that the volume open on sealing the vessel will be no more than the volume needed for the sample and the extraction fluid.) Install the filter and supports, and seal the vessel. Check the pH of the extraction fluid to ensure that it is within the proper range (4.93 $\pm$ 0.05). Then add 20 times the sample weight of extraction fluid into a class A graduated cylinder, and attach the pump and the transfer line to the ZHE. Open the value to the transfer line and then turn on the pump and pump in the appropriate volume of extraction fluid. When the fluid has been transferred, remove the transfer line and close the valve. Pressurize the ZHE to 5 to 10 psi and then rotate the ZHE 2-3 times. Open the valve and expel any residual headspace. Stop at the first sign of liquid expulsion. Check the ZHE carefully for leaks and make sure that the pressure is holding to within 2 psi of the initial pressure. If not, discard the sample and set up a new aliquot in a different ZHE. Make sure to record the initial pressure on the extraction sheet. Proceed to section 10.3.4.

10.3.3 If the waste is biphasic, charge the ZHE with enough sample to obtain 10-25 grams of solid using the formula shown below. Install the filter and supports, and seal the unit. Pressurize the ZHE by raising the piston and expelling any headspace. Attach a 50 ml syringe to the ZHE and continue to raise the piston to expel the liquid phase. Transfer the liquid to 40 ml VOA vials for storage at 4^{o}C until analysis. Check the pH of the extraction fluid to ensure that it is within the proper range (4.93 $\pm$ 0.05). Charge the ZHE with extraction fluid equivalent to 20 times the sample dry weight, (as described in 10.3.2 above) and pressurize the ZHE at 5 to 10 psi. Check the ZHE carefully for leaks. Record the initial pressure on the extraction sheet.

$$\text{final g of sample (10 to 25)} = \frac{\text{x g of sample}}{\text{\% solids}/100}$$

10.3.4 Turn on the rotary agitator and allow the extraction to proceed 18 $\pm$ 2 hrs at a rotation rate of 30 $\pm$ 2 rpm and a temperature of 23 $\pm$ 2°C.

10.3.5 At the end of the extraction period, check the pressure reading on each ZHE. If the pressure has decreased by more than 2 psi from the initial pressure reading of 5 to 10 psi, then the sample must be discarded and releached using a different ZHE. (The leaking ZHE must be successfully leak tested as outlined in Section 8.1.1 before reuse with another sample.)

10.3.5.1 Note: an increase in pressure is not a problem.

10.3.6 If the pressure has held over the course of the extraction, then attach a 50 ml syringe to the ZHE on the three-way valve, open the valve, and remove three 50

ml aliquots of the leachate. Transfer the aliquots to 40 ml VOA vials and store with no headspace at 4°C until analysis.

10.3.6.1 For biphasic samples, if a compatible liquid was obtained in Section 10.3.3, then the extract must be combined with this liquid proportionally before analysis. For example, if there was 50 ml of original filtrate and 300 ml of extract, then a 40 ml aliquot of sample to be analyzed should contain 5.7 ml of the original filtrate and 34.3 ml of the filtered extract. Check with the area supervisor or manager for further assistance when 2 extracts are obtained.

10.3.6.2 For biphasic samples, if a non-compatible liquid was obtained in Section 10.3.3, then each separate aliquot must be analyzed. The results should be combined mathematically after analysis according to the following formula:

$$\text{Concentration} = \frac{V_1C_1 + V_2C_2}{V_1 + V_2}$$

Where: V_1 = Volume of first phase (liters)
C_1 = Concentration of analyte, ug/l, in first phase.
V_2 = Volume of second phase (liters)
C_2 = Concentration of analyte, ug/l, in second phase.

10.3.7 Open the ZHE and remove the remaining leachate and sample. Remove the piston and clean the vessel with hot soapy water, rinse with deionized water, and dry in an oven at 110°C. If the ZHE contained a sample with high levels of organics (i.e. oily appearance or strong smell), then Contrad soap (available in the organic extractions area) should be used to clean the vessel. After approximately 10 extractions, or after a dirty sample is leached, the o-rings on the ZHE should also be changed.

10.4 Make sure that all documentation is complete and have the paperwork checked by the general chemistry lab supervisor or manager. Then make copies for the organics analysis department and distribute the sample fractions with the paperwork.

11.0 QC REQUIREMENTS

11.1 A method blank must be done for every 20 extractions that are done in an extraction vessel. A different container should be used for the method blank for each batch. The blank containers should be rotated so that all of the containers are used for a blank over time. It is recommended that a method blank be prepared on each different analysis day. In addition, method blanks must be prepared for each type of extraction fluid used for a batch. If the sample does not need leaching due to low percent solids, then DI water is provided as the method blank associated with that sample.

11.2 An aliquot of the appropriate extraction fluid must be provided to each analytical area to use to prepare a leachate spike blank for each batch. Extraction fluid #1 is provided for all extracted samples and DI water is provided for samples that are filtered only.

11.3 For each matrix type extracted, (soil, water, sludge, etc.) a leachate spike must be performed. Various unique matrices may require their own leachate spikes. Check with the lab supervisor or manager to find out the leachate volume required for a given sample. A minimum of one leachate spike must be performed for every 20 samples of a specific matrix.

12.0 DOCUMENTATION

12.1 A minimum of once per week, check and record the rotation speed of the TCLP agitation apparatus on the sample worksheet. The rotator should turn at a rate of 30 $\pm$ 2 rpm.

12.2 Record all extraction information on the data worksheet, including the characterization, the sample weights used, the extraction fluid pH, the final extract pH, the initial and final pressures, etc. Make sure to record all pH values to 2 places past the decimal.

12.3 Record all the reagent information, including manufacturers, lot numbers, and expiration dates, in the TCLP reagent log.

12.4 Record the temperature at the beginning and end of each leaching period. Make sure to use a calibrated thermometer to measure the temperature.

12.5 Record all leak testing and maintenance on the ZHE extractors and rotators in the TCLP maintenance log.

13.0 DATA REVIEW AND REPORTING

13.1 All samples should be updated to QC batches in the LIMS system. The analyst is responsible for reviewing all data for compliance with the QC outlined in this SOP. They are responsible for making sure that the raw data is fully documented and it is updated and entered into the LIMS system.

13.2 After the analyst review is completed, the supervisor or a designated reviewer shall review the data for technical compliance to the SOP. Additional reviews are periodically done by the manager of the department for technical completeness. The raw data is then electronically filed by the report generation department.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment

must be followed. All method users must be familiar with the waste management practices described in section 14.2.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

13.2.1 Non hazardous aqueous wastes.

13.1.2 Hazardous aqueous wastes

13.1.3 Chlorinated organic solvents

13.1.4 Non-chlorinated organic solvents

13.1.5 Hazardous solid wastes

13.1.6 Non-hazardous solid wastes

14.0 ADDITIONAL REFERENCES

14.1 No additional references are required for this method.

Lab Manager_____

QA Manager_____

Effective Date_____

TEST NAME: METHOD 8260B, VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (GC/MS)

METHOD REFERENCE: SW846 8260B (Revision 2, December 1996)

Revised Section: 2.8

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the analytical procedures, which are utilized by Accutest to acquire samples for analysis of volatile organic compounds by gas chromatographic/mass spectrometric (GC/MS) following purge and trap utilizing the internal standard technique. The compounds in Table 1 may be determined by this method. An option has been included for the analysis of 1,4-Dioxane by selected ion monitoring GC/MS (GC/SIM-SIM).

1.2 This analytical method is designed for nearly all types of samples, regardless of water content, including ground water, aqueous sludges, liquors, waste solvents, oily wastes, tars, filter cakes, sediments and soils.

1.3 The applicable concentration range of this method is compound, matrix, and instrument dependent. Volatile water-soluble compounds can be included in this analytical technique. However, for some low-molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides, quantitation limits are approximately ten times higher because of poor purging efficiency. Determination of some structural isomers (i.e. xylenes) may also be hampered by coelution.

2.0 SUMMARY OF METHOD

2.1 Volatile compounds are introduced into the gas chromatograph by purge-and-trap (Method 5030/5035). Method 5030 may be used directly on ground water samples. Method 5035 is used for low-concentration and medium-concentration soils, sediments, and wastes. Medium concentration samples are preserved and stored in methanol prior to purge-and-trap analysis.

2.2 An inert gas is bubbled through a 5 ml sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic (GC) column.

2.3 The volatile compounds are separated by the temperature programmed GC column and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.4 The peaks detected are qualitated by comparison to characteristic ions and retention times specific to the known target list of compounds.

2.5 Once identified the compound is quantitated by comparing the response of major (quantitation) ion relative to an internal standard technique with an average response factor generated from a calibration curve.

2.6 Additional unknown peaks with a response > 10 % of the closest internal standard may be processed through a library search with comparison to a database of approximately 75,000 spectra. An estimated concentration is quantitated by assuming a response factor of 1.

2.7 Water soluble volatile organic and other poor purging compounds maybe analyzed using this methodology, however this method is not the method of choice for these compounds and the laboratory's ability to achieve all calibration and quality control criteria for this method cannot be guaranteed. These compounds are noted as (pp) in Table 7.

2.8 The method includes an analytical option for the analysis of 1,4-Dioxane by GC/MS-SIM. The selected ions that are characteristic of the analytes of interest are analyzed using lower concentrations of calibration standards under the same MS conditions. SIM analysis is performed upon client request and is documented in the report.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve and may vary depending on matrix interferences, sample volume or weight and percent moisture. Detected concentrations below this concentration cannot be reported without qualification. See Table 10.

3.1.1 Compounds detected at concentrations between the reporting limit and MDL are quantitated and qualified as "J", estimated value. Program or project specifications may dictate that "J" qualified compounds are not to be reported.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method.

3.2.2 Process all raw data for the replicate analysis in each MDL study. Forward the processed data to the QA group for archiving.

4.0 DEFINITIONS

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank, Instrument Blank, Storage Blank, Cleanup Blank and Sulfur Blank.

4-BROMOFLUOROBENZENE (BFB) - the compound chosen to establish mass spectral instrument performance for volatile (VOA) analyses.

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CONTINUING CALIBRATION - analytical standard run every 12 hours to verify the initial calibration of the system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - used herein synonymously with the terms continuous extraction, continuous liquid extraction, and liquid extraction. This extraction technique involves boiling the extraction solvent in a flask and condensing the solvent above the aqueous sample. The condensed solvent drips through the sample, extracting the compounds of interest from the aqueous phase.

EXTRACTED ION CURRENT PROFILE (EICP) - a plot of ion abundance versus time (or scan number) for ion(s) of specified mass (Es).

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer to the target compounds.

INTERNAL STANDARDS - compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatiles), and sample extract (for semivolatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD DETECTION LIMITS (MDLs) - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLs should be determined approximately once per year for frequently analyzed parameters.

PERCENT DIFFERENCE (%D) - As used in this SOP and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

PRIMARY QUANTITATION ION - a contract specified ion used to quantitate a target analyte.

REAGENT WATER - water in which an interferant is not observed at or above the minimum detection limit of the parameters of interest.

RECONSTRUCTED ION CHROMATOGRAM (RIC) - a mass spectral graphical representation of the separation achieved by a gas chromatograph: a plot of total ion current versus retention time.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

INSTRUMENT BLANK – a system evaluation sample containing lab reagent grade water with internal standards and surrogate standards added. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which include the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 The following analytes covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichlorethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Primary standards of these toxic compounds must be prepared in a hood. A

NIOSH/Mass approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 INTERFERENCES

6.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

6.2 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

6.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

6.4 Contamination by carry-over can occur whenever high level and low-level samples are sequentially analyzed.

6.4.1 Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of an instrument blank to check for cross contamination. Refer to Table 11 for compounds that may cause carryover for this method.

6.4.2 It may be necessary to wash the purging device with methanol, rinse it with organic-free water, and then dry the purging device in an oven at 105^0 C. Follow the instrument manual for instructions on cleaning. Document the occurrence in the maintenance log and notify the manager/supervisor.

6.4.2.1 Clean and bake purging tube.

6.4.2.2 Clean or replace purge needle.

6.4.2.3 Clean and bake sample filter or sparge filter.

6.4.2.4 Clean and bake sample loop.

6.4.2.5 Replace trap if necessary.

6.4.2.6 Replace water management module if necessary.

6.4.2.7 Rinse transfer line with methanol. Caution: disconnect the trap before rinsing.

6.4.3 In extreme situations, the entire purge-and trap device may require dismantling and cleaning. Follow the instrument's manual for instructions on disassembly. Document the occurrence in the maintenance log and notify the

manager/supervisor. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples.

6.4.4 If the contamination has been transferred to gas chromatograph, any of the following approaches may be used to cleanup the instrument.

6.4.4.1 Baking out the column between analyses.

6.4.4.2 Change the injector liner to reduce the potential for cross-contamination.

6.4.4.3 Remove a portion of the analytical column in the case of extreme contamination.

6.4.5 The oven temperature program must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are stripped from the chromatographic column.

6.4 Special precautions must be taken during the analysis to avoid contamination from methylene chloride and other common laboratory solvents.

6.5.1 The sample storage and analytical area should be isolated from all atmospheric sources of methylene chloride or other common solvents.

6.5.2 Laboratory clothing worn by the analyst should be clean and used in designated areas only. Clothing previously exposed to solvent vapors in the organics sample preparation laboratory can contribute to sample contamination.

7.0 SAMPLE HANDLING AND PRESERVATION AND HOLDING TIME

7.1 HANDLING and PRESERVATION

7.1.1 Water samples

7.1.1.1 Container - 40 ml glass screw-cap VOA vial with Teflon-faced silicone septum. The 40-ml glass VOA vials are pre-cleaned and certified.

7.1.1.2 Collect all samples in duplicate. Test all samples for residual chlorine using test paper for free and total chlorine. If samples contain residual chlorine, three milligrams of sodium thiosulfate should be added for each 40 ml of water sample.

7.1.1.3 Fill sample bottles to overflowing, but do not flush out the dechlorinating agent. Sample should be taken with care so as to prevent any air or bubbles entering vials creating headspace.

7.1.1.4 Adjust the pH of all samples to $\leq$ 2 at the time of collection, but after dechlorination, by carefully adding two drops of 1:1 HCl for each 40 ml of sample. Seal the sample bottles, Teflon face down, and mix for one minute.

Note: Do not mix the sodium thiosulfate with the HCl in the sample bottle prior to sampling.

7.1.1.5 The samples must be protected from light and refrigerated at 2 - 6 °C from the time of receipt until analysis.

7.1.2 Soil Samples

7.1.2.1 Refer to the SOP for SW846 Method 5035 for preservation requirement of non-aqueous solids. ForOhio VAP freezing is not allowed; samples must be preserved with sodium bisulfate.

7.2 HOLDING TIME

7.2.1 Water Samples.

7.2.1.1 All samples are to be analyzed within 14 days of sampling (HCl preserved for aqueous sample) unless otherwise specified by the contract. If aqueous samples are received unpreserved, the client is notified of the deficiency and the samples must be analyzed within 7 days. The sample preservation deficiency is noted on the chain of custody.

7.2.2 Soil Samples

7.2.2.1 Refer to the SOP for SW846 Method 5035 for holding time requirement of non-aqueous solids.

7.2.2.2 All samples are analyzed within 14 days of sampling unless otherwise specified.

8.0 APPARATUS AND MATERIALS

8.1 SYRINGE

8.1.1 10, 25, 50, 100, 500 and 5000 µl graduated syringes, manually held (Hamilton/equiv.).

8.1.2 5 ml and 50 ml glass gas tight syringes with Luerlok end, if appropriate for the purging device.

8.2 BALANCE

8.2.1 Analytical balance capable of weighing 0.0001 gram.

8.2.2 Top loading balance capable of weighing 0.1 gram.

8.3 PURGE AND TRAP DEVICES

8.3.1 The autosampler models are used for purging, trapping and desorbing the sample into GC column.

- O.I. Model 4560 sample concentrator with 4551 vial multi-sampler
- O.I. Model 4560 sample concentrator with 4552 Water/Soil multi-sampler

8.3.2 The sample purge vial must be designed to accept 5 ml samples with a water column at least 3 cm deep.

8.3.3 The auto-sampler is equipped with a heater capable of maintaining the purge chamber at 40 °C to improve purging efficiency. The heater is to be used for low level soil/sediment analysis, but not for water or medium level soil/sediment analysis.

8.3.4 The OI #10 trap is 42 cm with an inside diameter of 0.105 inches. The trap must be packed to contain the following absorbents (3-ring) and should be conditioned at 180 °C for 30 minutes by backflushing with a Helium gas flow at least 20 ml/min before initial use.

- Tenax (2,6-Diphenylene oxide polymer).
- Silica gel.
- Carbon Molecule Sieve (CMS).

8.3.5 The desorber should be capable of rapidly heating the trap to 190^0 C for desorption. Do not exceed 210^0 C during bake-out mode. Alternatively, follow manufacturer's instructions.

8.3.6 The response of chloromethane and bromonethane can be tracked for thermal decomposition products formed. If levels over the calibration requirement, the trap must be replaced and the system re-calibrated after the manager/supervisor been notified.

8.4 GAS CHROMATOGRAPH/MASS SPECTROMETER SYSTEM

8.4.1 Gas Chromatograph.

8.4.1.1 An analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases.

8.4.1.2 The injection port should be suitable for split or splitless with appropriate interface.

8.4.1.3 The narrow bore capillary column is directly coupled to the source for HP-6890 model.

8.4.1.4 The wide bore capillary column is interfaced through a jet separator to the source for HP-5890 model.

8.4.2 Column.

- 75 m x 0.53mm ID x 3 μm film thickness capillary column coated with DB-624 (J&W Scientific), or equivalent. Condition as per manufactures directions.
- 105 m x 0.53mm ID x 3 μm film thickness capillary column coated with HP-VOA, or equivalent. Condition as per manufactures directions.
- 60 m x 0.25mm ID x 1.4 μm film thickness capillary column coated with DB-624 (J&W Scientific), or equivalent. Condition as per manufactures directions.
- 60 m x 0.45mm ID x 1.7 μm film thickness capillary column coated with DB-VRX (J&W Scientific), or equivalent. Condition as per manufactures directions.

8.4.3 Mass Spectrometer.

8.4.3.1 HP5973 or HP5970 is capable of scanning from 35 to 300 amu every 2 seconds or less, utilizing a 70 volt (nominal) electron energy in the electron impact ionization mode.

8.4.3.2 The mass spectrometer must be capable of producing a mass spectrum which meets all the criteria in Table 3 when injecting or purging 50 ng of the GC/MS tuning standard - Bromofluorobenzene (BFB).

8.4.3.3 SIM Mode – Capable of selective ion grouping at specified retention times for increased compound sensitivity (Table 2a).

8.5 DATA SYSTEM

8.5.1 Data Acquisition and Instrument Control (HP Chemstation) **-** A computer system is interfaced to the mass spectrometer, which allows the continuous acquisition and storage on a machine-readable media (disc) of all mass spectra obtained throughout the duration of the chromatographic program.

8.5.2 Data Processing (HP Enviroquant) - The software accommodates searching of GC/MS data file for target analytes which display specific fragmentation patterns. The software also allows integrating the abundance of an EICP between specified time or scan number limits. The data system includes the recent version of the EPA/NBS or NIST98 mass spectral library for qualitative searches of non-target compounds present in the chromatogram. The data system flags all data files that have been edited manually by laboratory personnel.

8.5.3 Off line Magnetic Tape Storage Device (Lagato Networker) - The magnetic tape storage device copies data for long-term, off-line storage.

9.0 REAGENTS AND STANDARDS

9.1 Solvent

9.1.1 Methanol: purge-and-trap grade quality or equivalent. Store separately, away from the other solvents.

9.2 Reagent Water

9.2.1 Reagent water is defined as water in which an interferant is not observed at the method detection limit of the parameters of interest.

9.2.2 Reagent water is generated by either passing tap water through a bed of approximately one pound of activated carbon or by using the water purification system at Accutest that is a series of deionizers and carbon cartridges.

9.3 Stock Standard Solutions

9.3.1 Commercially prepared standards used.

9.3.1.1 EPA Method 524.2 Volatiles (78 components): Absolute (or equivalent) at 200 μg/ml or 2,000 μg/ml concentration.

9.3.1.2 Custom Volatiles Mix A: Restek (or equivalent) at 2,000 μg/ml concentration.

9.3.1.3 Custom Volatiles Mix B: Restek (or equivalent) at 2,000 - 100,000 μg/ml concentration.

9.3.1.4 VOC Gas Mixture: Ultra (or equivalent) contains 200 μg/ml or 2,000 μg/ml of the following compounds in methanol.

- Bromomethane
- Chloroethane
- Chloromethane
- Dichlorodifluoromethane
- Trichlorofluoromethane
- Vinyl Chloride

9.3.1.5 Multiple neat compounds.

9.3.1.6 Surrogate standard mixture: Ultra (or equivalent) at a concentration of 2,500 μg/ml each surrogate compound.

- 1,2-Dichloroethane-d_4
- Dibromofluoromethane
- Toluene-d_8
- 4-Bromofluorobenzene

9.3.1.7 Internal standard mixture: Ultra (or equivalent) at a concentration of 2,000 μg/ml for all the compounds except Tert Butyl Alcohol-d_9, which is from Absolute (or equivalent) at a concentration of 50,000 μg/ml. The following five internal standards are used that exhibit similar analytical behavior to the compounds of interest.

- 1,4-Dichlorobenzene-d_4
- 1,4-Difluorobenzene
- Chlorobenzene-d_5
- Pentafluorobenzene
- Tert Butyl Alcohol-d_9

9.3.1.8 1,4-Dioxane Solution for SIMS : Ultra (or equivalent) at 100 μg/ml in methanol .

9.3.2 Unopened stock standard (ampoules) must be stored according to manufacturer's documented holding time and storage temperature recommendations (usually placed on the ampoule).

9.3.3 After opened, stock standards, internal standards, and surrogate solutions must be replaced after 6 months (one month for purgeable gases standard) or sooner if manufacture expiration date come first or comparison with quality control check samples indicates degradation.

9.3.4 Store all stock standards in vials with minimal headspace and Teflon lid liners after open, protect from light, and refrigerate to –10°C or colder or as recommended by the standard manufacturer.

9.3.5 Return the standards to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

9.4 Internal Standard and Surrogate Solution

9.4.1 Five internal standard and surrogate spiking solutions are prepared in methanol per Table 8.A.

9.4.1.1 25 μg /ml internal standard and surrogate mixture.

9.4.1.2 250 μg /ml internal standard and surrogate mixture.

9.4.1.3 100 μg/ml surrogate mixture.

9.4.1.4 25 μg /ml internal standard mixture.

9.4.1.5 250 μg /ml internal standard mixture.

9.4.2 A calibration range must be constructed for the surrogate compounds. Accordingly, appropriate amounts of surrogates are mixed with each calibration solution to define a range similar to the target compounds.

9.4.3 Each 5 ml sample, QC sample, and blank undergoing analysis should be spiked with any one of the above spiking solutions (depending upon the type of standards addition modules used), resulting in a concentration of 50 μg/l of each compound.

9.4.4 Prepare fresh internal standard and surrogate spiking solutions every six months, or sooner, if manufacturer's expiration dates come first or if the solution has degraded or evaporated.

9.5 Secondary Dilution Standards

9.5.1 Using stock standard solutions, prepare secondary dilution standards in methanol containing the compounds of interest, either singly or mixed together.

9.5.1.1 100 μg /ml V8260 mixture: prepared from 2,000 μg /ml stock solution. (see Table 8-C)

9.5.1.2 100 μg /ml V8260 custom mixture: prepared from 2,000 μg /ml stock solution. (see Table 8-C)

9.5.1.3 100 μg /ml Gas mixture: prepared from 2,000 μg /ml stock solution. (see Table 8-C)

9.5.2 Replace after one month for non-gas mixtures (one week for gas mixtures) or sooner if manufacture expiration date come first or comparison with quality control check samples indicates degradation.

9.5.3 Store all secondary dilution standards in vials with no headspace and Teflon lid liners, protect from light, and refrigerate to – 10°C or colder or according to manufacturer's storage temperature recommendation.

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9.5.4 Return the standards to the freezer as soon as preparation is finished to prevent the evaporation of volatile compounds.

9.6 Aqueous Calibration Standard Solutions

9.6.1 Initial Calibration Standards

9.6.1.1 Prepare a minimum of five aqueous calibration standard solutions containing the surrogate compounds as Table 8-D.1 or 8-D.2.

9.6.1.2 To prepare a calibration standard, add a measured volume of secondary dilution standard solutions and the surrogate spiking solution to an aliquot of reagent water in the flask. Use a micro-syringe and rapidly inject the methanol standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Bring to volume. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask.

9.6.1.2.1 1,4-Dioxane for SIMS analysis is prepared from primary stock standard (100ppm).

9.6.2 Continuing Calibration Standard

9.6.2.1 A continuing calibration standard at a concentration of 50 μg/l is prepared as the scheme outlined in Table 8-E.

9.6.3 Aqueous standards are not stable and may be stored up to 24 hours if held in Teflon sealed screw-cap vials with zero headspace at 4^0C ($\pm$ 2^0C). Protect the standards from light. If not so stored, they must be discarded after use, unless they are set up to be purged by an autosampler.

9.6.4 When using an autosampler, standards may be retained up to 12 hours if they are in purge tubes connected via the autosampler to the purge and trap device.

9.7 Second Source Calibration Check Standard (ICV)

9.7.1 Prepare the second source calibration check standards from separate sources of stock standards from the calibration curve following the procedures in Section 9.6. At a minimum, an ICV should be analyzed with every initial calibration.

9.7.2 For 1,4-Dioxane via SIMS: Prepare the second source calibration check standard using 2.5 µl of a 1000ppm (Absolute or equivalent) to 50 mL of reagent water which yields a 50 ppb standard.

9.8 4-Bromofluorobenzene (BFB) Standard

9.8.1 Two BFB solutions are prepared in methanol per Table 8-B.

9.8.1.1 25 μg /ml solution for direct injection.

9.8.1.2 250 μg /ml solution for purging.

9.8.2 The solution must be replaced after 6 months or sooner if mass spectrum indicates degradation or if manufacture expiration date comes first.

10.0 CALIBRATION

10.1 Daily Maintenance. Routine Daily maintenance must be performed before any tuning, calibration or sample analysis activities are initiated. These include checks of the following items:

<u>Purge and Trap Device:</u>

Clean & bake purge tube
Bake trap and transfer lines
Check or refill internal/surrogate spike solution on SIM/SAM vials
Clean/replace syringe (if necessary)
Change and refill rinse bottle
Empty and rinse waste bottle

GC Oven: (if necessary)

Change septum
Change liner
Clip column, indicated by carbon build-up

10.2 Initial Calibration

10.2.1 The calibration range covered for routine analysis under RCRA, and SIM, employs standards of 1(specified compounds only), (2)*, 5, 10, 20, 50, 100, 200,(300 or 400)* μg/l. (*instrument dependent). A minimum of five standards must be run sequentially. The low calibration standard defines the reporting limit. Lower concentration standards (1.0 or 2.0 μg/l) may be needed to meet the reporting limit requirements of state specific regulatory programs. Refer to Table 8-D-1 and 8-D-2 for calibration standard preparation.

10.2.2 A calibration range must be constructed for each surrogate compound. Accordingly, add appropriate amounts of each surrogate compound to the calibration solution to define a range similar to the target compounds.

10.2.2.1 For most samples and spikes both the internal standard and the surrogate are added automatically. When doing an initial calibration surrogates are added manually. In order to compensate for the difference between the automatic and manual surrogate additions a correction factor must be applied to the amount of surrogate added in Table 8-D. To determine the correction factor divide the surrogate concentration from an automatic injection by the surrogate concentration from a manual injection for each of the surrogates. Average the result for each of the surrogates to determine the correction factor. Finally multiply the correction factor by the appropriate amount of surrogate from Table 8-D and add this amount to the standard.

10.2.3 For water and medium-level soil calibration: Transfer and fill up (no air space) each standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray.

10.2.4 For low-level soil calibration: Transfer 5 ml of each standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray.

10.2.4.1 When calibrating for Method 5035 low-level samples, if the sodium bisulfate option was used, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of each standard into vial otherwise do not add sodium bisulfate. This is equivalent to the amount of sodium bisulfate added to the samples and

will maintain a consistent purging efficiency of the compounds. Cap the vial with Teflon septum and place it into O.I sample tray.

10.2.5 The linear range covered by this calibration is the highest concentration standard.

10.2.6 Program the autosampler to add internal standard mixture to each standard. This results in a concentration of 50 μg/l for each internal standard.

10.2.6.1 For O.I. SIM spiker: Automatically adds 10 μl of 25 μg/ml internal standard solution **(**Section 9.4.1.4) to each standard.

10.2.6.2 For O.I. SAM spiker: Automatically adds 1 μl of 250 μg/ml internal standard solution (Section 9.4.1.5) to each standard.

10.2.7 Analyze the standard solutions using the conditions established in Section 11.0. Whenever the highest concentration standard is analyzed, it is usually followed by the analyses of two reagent water blanks. Further analysis may not proceed until the blank analysis is demonstrated to be free of interferences.

10.2.8 Each analyte is quantitatively determined by internal standard technique using the closest eluting internal standard and the corresponding area of the major ion. See Table 7.

10.2.9 The Response Factor (RF) is defined in Section 13.1. Calculate the mean RF for each target analyte using minimum of five RF values calculated from the initial calibration curve.

10.2.10 For the initial calibration to be valid, the following criteria must be met.

10.2.10.1 Five compounds (System Performance Check Compounds, SPCCs) are checked for a minimum average response factor. The minimum mean response factors are listed in Table 6. If the initial calibration criteria for SPCCs are not achieved, perform corrective action before completing the calibration

10.2.10.2 The % RSD for each individual Calibration Check Compound (CCC) must be less than 30 %. This check is used to identify gross instrument operating problems. If the initial calibration criteria for CCCs are not achieved, perform corrective action before completing the calibration.

10.2.10.3 The percent relative standard deviation (% RSD) (see Section 13.2) of all target analytes must be less than 15 %.

10.2.10.4 If the average response factor criteria cannot be achieved, and if the problem is associated with one or more of the standards, reanalyze the standards and recalculate the RSD. The instrument logbook should have clear documentation as to what the suspected problem was.

10.2.10.4.1 A calibration standard is allowed to be repeated only once; if the second trial fails, a new initial calibration must be performed. Notify the team leader/manager. Document this occurrence in the instrument log.

10.2.10.5 Alternately, if the average response factor criteria cannot be achieved, the calibration range can be narrowed by dropping the low or high point of the curve.

10.2.10.5.1 The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end.

10.2.10.6 If the average response factor criteria still cannot be achieved, employ an alternative calibration linearity model. Specifically, linear regression using a least squares approach may be employed.

10.2.10.6.1 If Linear regression is employed select the linear regression calibration option of the mass spectrometer data system. Do not force the regression line through the origin and do not employ 0,0 as a sixth calibration standard.

10.2.10.6.2 The correlation coefficient (r value) must be $\geq$0.99 for each compound to be acceptable.

10.2.10.6.3 Perform corrective action and recalibrate if the calibration criteria cannot be achieved.

10.2.10.7 The initial calibration criteria for this method applies to all additional compounds of concern specified by the client.

10.2.10.8 The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units.

10.3 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

10.3.1 The calibration is verified with a calibration check standard at 50 µg/l from an external source (Section 9.7). It must be analyzed immediately following the initial calibration.

10.3.2 The percent difference (% D) (Section 13.3) for this standard must meet the criteria of 20% for all the target compounds.

10.3.2.1 If % D is greater than 20%, reanalyze the second source check. If the criteria cannot be met upon re-injection, re-prepare the second source solution using a fresh ampoule and repeat the process.

10.3.2.2 If the %D criteria cannot be achieved after re-preparation of the second source, prepare a third source and repeat the process. Make fresh

calibration standards using one of the two standard sources that matches each other and repeat the initial calibration.

10.4 Continuing Calibration Verification Standard(CCV)

10.4.1 A continuing calibration verification standard at a concentration near mid-level of the initial calibration range (50 μg/l) must be acquired every 12 hrs or at the beginning of each analytical batch.

10.4.1.1 For water and medium level soil analysis: Transfer and fill up (no air space) the calibration verification standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray. Analyze as per Section 11.7.

10.4.1.1.1 Vary the concentration of the continuing calibration verification standard on alternate verifications (i.e. every other calibration verification) using an alternative concentration standard. The standard selected must be lower than the midpoint calibration standard.

10.4.1.2 For low-level soil analysis: Transfer 5 ml of the calibration verification standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray. Analyze as per Section 11.7.

10.4.1.2.1 When calibrating for Method 5035 low-level samples, if the sodium bisulfate option was used add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of the calibration verification standard into vial, otherwise do not use sodium bisulfate. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds. Analyze as per Section 11.7.

10.4.1.3 A continuing calibration standard is analyzed whenever the analyst suspects that the analytical system is out of calibration. If the calibration cannot be verified, corrective action is performed to bring the system into control. Analysis may not continue until the system is under control.

10.4.2 For the continuing calibration to be valid, all of the following specified criteria must be met.

10.4.2.1 The minimum RF for SPCC compound is shown on Table 6. Each SPCC compound in the calibration verification standard must meet its minimum response factor.

10.4.2.2 The percent difference (% D, see Section 13.3) for CCC must be less than 20%.

10.4.2.2.1 If the CCCs and SPCCs are not required analytes, such as the shortlist analysis for BTEX only, then all required project analytes must meet the 20 %D.

10.4.3 If the first continuing calibration verification does not meet criteria, a second standard may be injected after notify the team leader/manager and checking the system for defects.

10.4.3.1 A continuing calibration check is allowed to be repeated only once; if the second trial fails, a new initial calibration must be performed. In situations where the first check fails to meet the criteria, the instrument logbook should have clear documented notations as to what the problem was and what corrective action was implemented to enable the second check to pass.

10.4.3.2 If the calibration verification is being performed using an auto sampler for night batch, two (2) vials of standard solution are placed in the device for analysis. The second standard must meet continuing calibration criteria and is used for calibration verification. The second check may be discarded because of a purge failure or incorrect spike concentration provided the first calibration standard meets the requirement. In this case, the first calibration standard is used as calibration verification following team leader/manager approval. Document this occurrence on instrument log.

10.4.4 If the verification criteria cannot be achieved, a new initial calibration must be performed.

10.4.5 If any of the internal standard areas change by a factor of two (- 50% to + 100%) or the retention time changes by more than 30 seconds from the midpoint standard of the last initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate.

10.4.5.1 Reanalyze the continuing calibration standard. New initial calibration is required if reanalyzed standard continues to fail the internal standard requirements.

10.4.5.2 All samples analyzed while the system was out of control must be reanalyzed following corrective action.

10.5 Corrective Action Maintenance For Failed Tuning and Calibration Procedures

10.5.1 Inability to achieve criteria for instrument tuning or calibration may indicate the need for instrument maintenance. Maintenance may include routine system cleaning and replacement of worn expendables or the need for outside service if the scope of the repair exceeds the capability of the staff.

10.5.2 If maintenance is performed on an instrument, return to control must be demonstrated before analysis can continue. Return to control is demonstrated as follows:

10.5.2.1 Successful instrument tune using PFTBA.

10.5.2.2 Successful tune verification by the analysis of 4-bromofluorobenzene.

10.5.2.3 Successful initial calibration or continuing calibration.

11.0 PROCEDURE

11.1 Instrument conditions.

11.1.1 Recommended instrument conditions are listed in Table 2 and 2a (SIM only). Modifications of parameters specified with an asterisk are allowed as long as criteria of calibration are met. Any modification should be approved by team leader/manger.

11.1.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, use the same GC conditions for the analysis of all standards, blanks, samples, and QC samples.

11.2 Purge and Trap Device conditions.

11.2.1 See Table 2.

11.2.2 Daily Maintenance. Routine Daily maintenance must be performed before any tuning, calibration or sample analysis activities are initiated. These include checks of the following items:

Purge and Trap Device:

- Clean & bake purge tube.
- Bake trap and transfer lines.
- Check or refill internal/surrogate spike solution on SIM/SAM vials.
- Clean/replace syringe (if necessary).
- Change and refill rinse bottle.
- Empty and rinse waste bottle.

11.3 Step 1: Daily GC/MS performance check.

11.3.1 Every 12 hours, either

- Inject 2 μl (50 ng) of BFB solution directly on column or
- Purge 10 μg/l of 5ml (50ng) to GC column.

11.3.2 The GC/MS system must be checked to verify acceptable performance criteria are achieved (see Table 3).

11.3.3 This performance test must be passed before any samples, blanks or standards are analyzed. Evaluate the tune spectrum using three mass scans from the chromatographic peak and a subtraction of instrument background.

11.3.3.1 Select the scans at the peak apex and one to each side of the apex.

11.3.3.2 Calculate an average of the mass abundances from the three scans.

11.3.3.3 Background subtraction is required. Select a single scan in the chromatogram that is absent of any interfering compound peaks and no more than 20 scans prior to the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tuning compound peak.

11.3.4 If all the criteria are not achieved, the analyst must retune the mass spectrometer with team leader/manager and repeat the test until all criteria are met.

11.3.4.5 Alternatively, an additional scan on each side of the peak apex may be selected and included in the averaging of the mass scans. This will provide a mass spectrum of five averaged scans centered on the peak apex. NOTE: The selection of additional mass scans for tuning may only be performed with supervisory approval on a case by case basis.

11.3.5 The injection time of the acceptable tune analysis is considered the start of the 12-hour clock.

11.3.6 Until performance check is acceptable, then calibration check (step 2) can be analyzed.

11.4 Step 2 : Daily calibration check

11.4.1 Initial calibration

11.4.1.1 Refer to Section 10.2.

11.4.1.2 An initial calibration must be established (or reestablished) on each instrument:

- Prior to any sample analyses;
- Whenever a new column is installed;
- Whenever instrument adjustments that affect sensitivity are made; and
- Whenever a continuing calibration standard fails to meet the specified acceptance criteria, on the second trial.

11.4.2 Initial Calibration Verification - Second Source Calibration Check Standard

11.4.2.1 This standard is only analyzed when initial calibration provided. Refer to Section 10.3.

11.4.3 Continuing Calibration verification standard

11.4.3.1 Refer to Section 10.4.

11.4.4 The method blank (step 3) cannot be analyzed until the continuing calibration verification meets the criteria.

11.5 Step 3 : Method blank

11.5.1 The acceptable method blank must be analyzed for every 12-hour time period or sooner.

11.5.1.1 Water and medium-level soil samples - Place a 40 ml vial, filled with DI water onto the autosampler.

11.5.1.2 Low-level soil samples without sodium bisulfate - Transfer 5 ml of DI water to a 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray.

11.5.1.2.1 Low-level soil samples with sodium bisulfate (Method 5035) - Add 1g of sodium bisulfate to a 40 ml vial before aliquot 5 ml of DI water into the vial and cap with Teflon septum, then place the vial onto the autosampler.

11.5.2 Program the autosampler to add internal standard and surrogate solution to the method blank for a concentration of 50 µg/l for each internal standard and surrogate.

11.5.2.1 For O.I. SIM spiker: Automatically adds 10 µl of 25 µg/ml internal standard and surrogate solution (Section 9.4.1.1) to the method blank.

11.5.2.2 For O.I. SAM spiker: Automatically adds 1 µl of 250 µg/ml internal standard and surrogate solution (Section 9.4.1.2) to the method blank.

11.5.3 No compound can be present above the laboratory's MDL. If common laboratory solvents (i.e. methylene chloride, acetone) are present in the sample between the MDL and RL, the analyst must determine if the contamination will negatively impact data quality. If the contamination impacts data quality, all affected samples must be re-analyzed.

11.5.4 Surrogates must meet recovery criteria specified in house limits.

11.5.5 If the method blank does not meet surrogate criteria or contains target analytes above the MDL, then

11.5.5.1 All samples analyzed following an out of control method blank must be reanalyzed.

11.5.5.2 Check for the potential of contamination interference from the following areas. Make sure all items are free contamination.

- the analytical system,
- dust and vapor in the air,
- glassware and
- Reagents.

11.5.5.3 Re-analyze the method blank following the system evaluation. In this situation, the instrument logbook should have clear documented notations as to what the problem was and what corrective action was implemented to enable the second blank to pass.

11.5.5.4 If re-analyzed method blank remains out of control, notify team leader or manager.

11.5.6 If two consecutive method blanks are analyzed during unattended operations, the second analysis must meet criteria for the subsequent sample analysis to be valid. Always report the second method blank. The second analysis can only be discarded because of a purge failure provided that the first blank meets the requirement. In this case, the first blank is reported following team leader/manager approval. Document this occurrence on the instrument log.

11.5.7 The blank spike (BS) (step 4) cannot be analyzed until the method blank meets criteria.

11.6 Step 4: Blank spike (BS)

11.6.1 An acceptable blank spike must be analyzed with every analytical batch. The maximum number of samples per analytical batch is twenty.

11.6.2 Spike 50 ml of reagent water with appropriate amount of the standards to prepare a blank spike containing 50 μg/L of each analyte. In situations where lower detection limits are required, a blank spike at 20 μg/L may be prepared. The stock solution for the BS must be from a different source than the initial calibration solution. Refer to Table 8-F for the preparations of the blank spikes.

11.6.2.1 Water and medium-level soil samples - Place a 40 ml vial, filled with DI water onto the autosampler.

11.6.2.2 Low-level soil samples without sodium bisulfate - Aliquot 5 ml of the blank spike into vial and cap with Teflon septum, then place the vial into O.I. sample tray.

11.6.2.2.1 Low-level soil samples with sodium bisulfate for Method 5035 - Add 1g of sodium bisulfate to labeled 40 ml vial before aliquot 5 ml of the blank spike into vial and cap with Teflon septum, then place the vial into O.I. sample tray.

11.6.3 Initiate auto addition of internal standard and surrogate into the syringe per 11.5.2.

11.6.4 Compare the percent recoveries (% R) (see Section 13.5) to the in house limits acceptance criteria. If a blank spike is out of control, all the associated samples must be reanalyzed. The exception is if the blank spike recovery is high and no hits reported in associated samples and QC batch. In that case, the sample results can be reported with footnote (remark) and no further action is required.

11.6.5 Do not analyze samples and MS/MSD (step 5) unless the BS meets acceptance criteria.

11.7 Step 5: Samples /MS/MSD analysis

11.7.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

11.7.2 Select the sample dilution factor to assure the highest concentration analyte is above the calibration range midpoint, but below the upper limit of the range depend on project requirements. See Table 9 for dilution guideline.

- Utilize FID screen data.
- Utilize acquired sample data.
- Utilize the history program.
- Sample characteristics (appearance, odor).

11.7.3 Water samples.

11.7.3.1 Using O.I.Model 4560 sample concentrator with 4551 or 4552 vial multisampler,

- Place the 40 ml vial in the tray, or
- Load 5ml sample into purge tube if sample volume limited.

11.7.4 Sediment/ soil sample

11.7.4.1 Low-level soil method

11.7.4.1.1 Collect the sample using the procedures detailed in the SOP for SW846 Method 5035 low - level soil samples.

11.7.4.1.2 Weigh out 5 g of each sample into a labeled vial. Add 5 ml of reagent water and cap the vial quickly. Transfer the 40ml vial to the autosampler tray. Stir and heat the sample at the time of analysis.

11.7.4.2 Medium-level soil method

11.7.4.2.1 Collect the sample using the procedures detailed in the SOP for SW846 Method 5035 medium - level soil samples.

11.7.4.2.2 Select a methanol aliquot of appropriate volume (see Table 9) determined via screening and transfer to 40 ml of reagent water.

11.7.5 Program the autosampler to inject the internal standard and surrogate solution into the robotic syringe used to withdraw sample from the 40 ml vial. This addition to 5 ml of sample is equivalent to a concentration of 50 μg/L of each internal standard and surrogate.

11.7.5.1 For O.I. SIM spiker: Automatically adds 10 μl of 25 μg/ml internal standard and surrogate solution (Section 9.4.1.1) to each sample.

11.7.5.2 For O.I. SAM spiker: Automatically adds 1 μl of 250 μg/ml internal standard and surrogate solution (Section 9.4.1.2) to each sample.

11.7.6 Purge the sample for 11 minutes with Helium.

11.7.6.1 Low-level soil sample must be performed at 40 °C while the sample is being agitated with the magnetic stirring bar or other mechanical means.

11.7.6.2 To improve the purging efficiency of water-soluble compounds, aqueous samples may also be purged at 40 °C as long as all calibration standards, samples and QC samples are purged at the same temperature and acceptable method performance is demonstrated.

11.7.7 One sample is randomly selected from each analytical batch of similar matrix types and spiked in duplicate to determine whether the sample matrix contributes bias to the analytical results. A matrix spike and matrix spike duplicate are performed by spiking the sample for a concentration of 50 μg/l or 50 μg/kg based on 5 g dry weight. In situations where lower detection limits are required, a blank spike at lower concentration may be prepared.

11.7.8 Desorb the sample for 4 minutes by rapidly heating the trap to 190 °C while backflushing with Helium. Desorb time may require performance optimum between 2.0 and 4.0 minutes as dictated by trap manufacturers specifications or instrument characteristics.

11.7.9 Program the purge and trap system to automatically rinse purge tube at least twice with heated organic-free water (reagent water) between analyses to avoid carryover of target compounds. For samples containing large amounts of water-soluble materials, suspended solids, high-boiling compounds, or high purgeable levels, it may be necessary to wash out the purging device with methanol solution between analyses, rinse it with distilled water.

11.7.10 Bake the trap at least 10 minutes at 210 °C to remove any residual purgeable compounds.

11.7.11 If the initial analysis of the sample or a dilution of the sample has a response for any ion of interest that exceeds the working range of the GC/MS system, the sample must be reanalyzed at a higher dilution.

11.7.11.1 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

11.8 Sample dilutions

11.8.1 Using Screening Data to Determine Dilution Factors

11.8.1.1 Dilution for High Concentration Analytes Exceeding The Calibration Range

11.8.1.1.1 The highest concentration target compound detected in the screen data is compared to the highest concentration calibration standard used for determinative volatile organics analysis.

11.8.1.1.1.1 Divide the calibration concentration of the highest concentration calibration standard by the screen concentration.

11.8.1.1.1.2 If the result is >1, sample dilution is considered.

11.8.1.1.2 The result from step 11.8.1.1.1 determines the dilution factor. The dilution factor is targeted to assure that the highest concentration diluted analyte is at the mid-range concentration of the calibration curve for the determinative analysis.

11.8.1.1.3 In all cases a conservative approach to dilution is applied to minimize the increase of detection and reporting limits

11.8.1.2 Dilution for High Concentration Matrix Interferences

11.8.1.2.1 The peak height of the background is compared to the peak height of the later eluting calibration standards from the screening analysis.

11.8.1.2.1.1 A rough estimate of background concentration is calculated by dividing the background peak height by the peak height of the selected screening standard and multiplying by its concentration.

11.8.1.2.2 If the result is >1, sample dilution is considered.

11.8.1.2.3 The result from step 11.8.1.2.1 determines the dilution factor. The dilution factor is targeted to avoid Carry-over contamination between samples and facilitate qualitative and quantitative analysis of target compounds present in the sample.

11.8.1.2.4 In all cases a conservative approach to dilution is applied to minimize the increase of detection and reporting limits

11.8.2 If the concentration of any target compound in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and re-analyzed. Until the diluted sample is in a sealed sample vial, all steps in the dilution procedure must be performed without delay.

11.8.3 Water Samples.

11.8.3.1 Prepare all dilutions of water samples in volumetric flasks (10 ml to 100 ml). Intermediate dilutions may be necessary for extremely large dilutions.

11.8.3.2 Calculate the approximate volume of reagent water, which will be added to the volumetric flask, and add slightly less than this quantity to the flask. Refer to Table 9 for dilution guideline.

11.8.3.3 Inject the proper sample aliquot from a syringe into the volumetric flask. Dilute the flask to the volume mark with reagent water. Cap the flask and invert the flask three times.

11.8.3.4 Fill a 40 ml sample vial and seal with a Teflon baked silicon septa, load the diluted sample into the autosampler and analyze according to Section 11.7.

11.8.3 Low-level Soil Samples.

11.8.3.1 The screening data are used to determine which is the appropriate sample preparation procedure for the particular sample, the low-level soil method or the medium-level soil method.

11.8.3.2 If any target compound exceeds the initial calibration range from the analysis of 5 g sample, a smaller sample size must be analyzed. However, the smallest sample size permitted is 0.5 g. If smaller than 0.5 g sample size is needed to prevent any target compounds from exceeding the initial calibration range, the medium level method must be used.

11.9 Data interpretation

11.9.1 Qualitative identification.

11.9.1.1 The targeted compounds shall be identified by analyst with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound.

11.9.1.2 The characteristic ions for target compounds that can be determined are listed in Table 7. Table 4 and Table 5 list the characteristic ions for internal standards and surrogate compounds respectively.

11.9.1.3 The criteria required for a positive identification are listed below.

11.9.1.3.1 The sample component must elute at the same relative retention time (RRT) as the daily standard. Criteria are the RRT of sample component must be within ± 0.06 RRT units of the standard component.

11.9.1.3.2 The relative intensities of these ions must agree within ± 30 % between the daily standard and sample spectra. (Example: For an ion with an abundance of 50 % in the standard spectra, the corresponding sample abundance must be between 20 and 80 %.)

11.9.1.3.2.1 Compounds can have secondary ions outside criteria from co-eluting compounds and/or matrix effect that can contribute to ion abundances. The interference on ion ratios can't always be subtracted out by software programs resulting in qualified compound identification.

11.9.1.3.2.2 Quantitation reports display compounds that have secondary ions outside the ratio criteria with a "#" flag.

11.9.1.3.2.3 Any quant reports with compounds that are deemed to be reportable despite the "#" flag, will be initialed in the "#" column by the analyst. Further review to the reporting of qualified compounds will be done by a supervisor or team leader and initialed on the quantitation.

11.9.1.3.3 Structural isomers that produce very similar mass specrtra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25 % of sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

11.9.2 Quantitative analysis

11.9.2.1 Once a target compound has been identified, its concentration (Section 13.4) will be based on the integrated area of the quantitation ion, normally the base peak (Table 7). The compound is quantitated by internal standard technique with an average response factor generated from the initial calibration curve.

11.9.2.2 If the sample produces interference for the primary ion, use a secondary ion to quantitate (see Table 7). This is characterized by an excessive background signal of the same ion, which distorts the peak shape beyond a definitive integration. Also interference could severely inhibit the response of the internal standard ion. This secondary ion must also be used to generate new calibration response factors.

11.10 Library search for tentatively identified compounds.

11.10.1 If a library search is requested, the analyst should perform a forward library search of NBS or NIST98 mass spectral library to tentatively identify 15 non-reported compounds.

11.10.2 Guidelines for making tentative identification are listed below.

11.10.2.1 These compounds should have a response greater than 10 % of the nearest internal standard. The response is obtained from the integration for peak area of the Total Ion Chromatogram (TIC).

11.10.2.2 The search is to include a spectral printout of the 3 best library matches for a particular substance. The results are to be interpreted by analyst.

11.10.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.10.2.4 Relative intensities of major ions in the reference spectrum (ions > 10 % of the most abundant ion) should be present in the sample spectrum.

11.10.2.5 The relative intensities of the major ions should agree within ± 20 %. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must between 30 and 70%).

11.10.2.6 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

11.10.2.7 Ions present in the reference spectrum but not in the sample spectrum should be verified by performing further manual background subtraction to eliminate the interference created by coeluting peaks and/or matrix interference.

11.10.2.8 Quantitation of the tentatively identified compounds is obtained from the total ion chromatogram based on a response factor of 1 and is to be tabulated on the library search summary data sheet.

11.10.2.9 The resulting concentration should be reported indicating: (1) that the value is estimate, and (2) which internal standard was used to determine concentration. Quantitation is performed on the nearest internal standard.

11.11 An instrument blank is a system evaluation sample containing lab reagent grade water with internal standards and surrogates. An instrument blank is used to remove and or evaluate residual carryover from high level standards, spike samples and field samples. Since target compound lists have expanded to overlap some volatile and semi-volatile compounds, instrument blanks are necessary to remove carryover contamination.

11.11.1 The compounds that may exhibit carryover for this method are listed in Table 11.

11.11.2 If instrument blanks following a standard or spike sample exhibits carrry-over effect, then any samples that show the same carryover profile, after a comparable concentration must be considered suspect and rerun for confirmation. For example, if an instrument blank has 1ppb detected after a 200ppb standard, then any sample following a sample containing 200ppb or above of the same compound must be confirmed for possible carryover.

11.11.3 If an Instrument Blank(s) was run following suspect high concentration samples and it exhibits the same carryover profile after a comparable concentration must be considered suspect and rerun for confirmation.

11.11.4 In some cases, several instrument blanks may have to be run to eliminate contamination from over loaded samples.

11.11.5 The analytical system is considered free of carryover, when no target analytes can be detected above the MDL.

11.12 Selected Ion Monitoring (SIM) Option Selected Ion Monitoring (SIM) Option

11.12.1 Instrument Set-Up: Modify the method for SIM analysis and define ion groups with retention times, ions and dwell times to include base peak ion for the target compounds of interest, surrogates, and internal standards (Table 2a.) Select a mass dwell time of 50 milliseconds for all compounds.

11.12.2 Calibration: Calibrate the mass spectrometer in the selected ion monitoring mode using 6 calibration standards of 5, 10, 20, 50, 100, 200 ug/l. Spike each standard with the SIM specific internal standard solution at 4ug/ml. Calculate individual response factors and response factor RSDs.

11.12.3 Initial Calibration Verification. Verify the initial calibration after its completion using a 50 ug/l calibration standard purchased or prepared from a second standards reference materials source. The initial calibration verification must meet the criteria of Section 10.2.

11.12.4 Continuing Calibration Verification. Verify the initial calibration every 12 hours using a 50 ug/l calibration. The continuing calibration verification must meet the criteria of Section 10.4.

11.12.5 Surrogate Standard Calculation.. Report surrogate spike accuracy for the surrogates spiked for the full scan GC/MS analysis.

12.0 QUALITY CONTROL

12.1 QC Requirements Summary

BFB	Beginning of the analytical shift and every 12 hours
Second Source Calibration Check Standard	Following initial calibration
Calibration Verification Standard	Every 12 hours
Method Blank	Every 12 hours
Blank Spike	One per analytical batch*
Matrix Spike	One per analytical batch*
Matrix Spike Duplicate	One per analytical batch*
Surrogate	Every sample and standard
Internal Standard	Every sample and standard

*The maximum number of samples per analytical batch is twenty.

12.2 Daily GC/MS Performance Check - BFB

12.2.1 Refer to Section 11.3.

12.3 Second Source Calibration Check Standard

12.3.1 Refer to Section 10.3.

12.3.2 Calibration Verification Standard

12.3.3 Refer to Section 10.4.

12.5 Method Blank

12.5.1 Refer to Section 11.5.

12.6 Blank Spike

12.6.1 Refer to Section 11.6.

12.7 Matrix Spike (MS)/Matrix Spike Duplicate (MSD)

12.7.1 One sample is selected at random from each analytical batch of similar matrix types and spiked in duplicate to check precision and accuracy.

12.7.2 Assess the matrix spike recoveries (Section 13.5) and relative percent difference (RPD) (Section 13.6) against the control limits..

12.7.3 If the matrix spike recoveries do not meet the criteria, check the blank spike recovery to verify that the method is in control. If the blank spike did not meet criteria, the method is out of control for the parameter in question and should be reanalyzed or qualified with an estimate of potential bias. Otherwise, matrix interference is assumed and the data is reportable. No further corrective action is required.

12.8 Surrogates

12.8.1 All standards, blanks, samples, and matrix spikes contain surrogate compounds, which are used to monitor method performance. If the recovery of any surrogate compound does not meet the control limits, the result must be flagged and:

12.8.1.1 The calculation must be checked.

12.8.1.2 The sample must be reanalyzed if the recovery of any one surrogate is out of control limit.

12.8.2 If the sample exhibits matrix interference, defined as excessive signal levels from target or non-target interfering peaks. In this case, reanalysis may not be required following team leader/manager approval.

12.8.3 If surrogate recoveries are acceptable upon reanalysis, the data from the reanalysis is reported. If the reanalysis date did not meet the hold time, then both sets of data must be submitted with the reanalysis reported.

12.8.4 If surrogates are still outside control limits upon reanalysis, then both sets of data should be submitted with the first analysis reported.

12.9 Internal Standard

12.9.1 Retention time for all internal standards must be within ± 30 seconds of the corresponding internal standard in the latest continuing calibration or 50 μg/l standard of initial calibration.

12.9.2 The area (Extracted Ion Current Profile) of the internal standard in all analyses must be within 50 to 200 % of the corresponding area in the latest calibration standard (12 hr. time period).

12.9.3 If area of internal standard does not meet control limits, the calculations must be checked. If a problem is not discovered, the sample must be reanalyzed.

12.9.4 If areas are acceptable upon reanalysis, the reanalysis data is reported.

12.9.5 If areas are unacceptable upon reanalysis, then both sets of data are submitted with the original analysis reported.

13.0 CALCULATION

13.1 Response Factor (RF)

$$RF = \frac{As \times Cis}{Ais \times Cs}$$

where:
As = Area of the characteristic ion for the compound being measured.
Ais = Area of the characteristic ion for the specific internal standard.
Cs = Concentration of the compound being measured (ug/l).
Cis = Concentration of the specific internal standard (ug/l).

13.2 Percent Relative Standard Deviation (% RSD)

$$\%RSD = \frac{SD}{RFav} \times 100$$

where:
SD = Standard Deviation
RFav = Average response factor from initial calibration.

13.3 Percent Difference (%D)

$$\%D = \frac{(RFav - RFcv)}{RFav} \times 100$$

where:
RFcv = Response factor from Calibration Verification standard.
RFav = Average response factor from initial calibration.

13.4 Concentration (Conc.)

For water:

$$\text{Conc. } (\mu g/l) = \frac{Ac \times Cis \times Vp}{Ais \times RF \times Vi}$$

For soil/sediment low level (on a dry weight basis):

$$\text{Conc. } (\mu g/kg) = \frac{Ac \times Cis \times Vp}{Ais \times RF \times Ws \times M}$$

For soil/ sediment medium level (on a dry weight basis)

$$\text{Conc. } (\mu g/kg) = \frac{Ac \times Cis \times Vp \times Vt}{Ais \times RF \times Vme \times Ws \times M}$$

Where:
Ac = Area of characteristic ion for compound being measured.
Ais = Area of characteristic ion for internal standard.
Cis = Concentration of internal standard
RF = Response factor of compound being measured(from initial calibration)
Vi = Initial volume of water purged (ml)
Vp = 5 ml (Total Purge Volume)
Vme = Volume of Methanol aliquot
Vt = Ml Solvent + ((100-% solid)/100 x Ws)
Ws = Weight of sample extracted (g).
M = (100 - % moisture in sample) / 100 or % solids / 100

13.5 Percent Recovery (% R)

$$\%\ R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

13.6 Relative Percent Difference (RPD)

$$RPD = \frac{|\ MSC - MSDC\ |}{(1/2)\ (MSC+MSDC)} \times 100$$

Where:
MSC = Matrix Spike Concentration
MSDC = Matrix Spike Duplicate Concentration

13.7 Linear regression by the internal standard technique.

$$C_s = \left(\frac{A_s}{A_{is}} - b \right) \times C_{is}$$

Where:
Cs = concentration of target analyte
As = Area of target analyte
Cis = concentration of the internal standard
b = Intercept
a = slope of the line

$$a = \frac{N \Sigma xy - \Sigma x \Sigma y}{N \Sigma x^2 - (\Sigma x)^2}$$

$$b = \frac{\Sigma y - a \Sigma x}{N}$$

N = number of points
x = amount of analyte
y = response of instrument

13.8 Correlation Coefficient

$$r = \frac{\Sigma(x - \bar{x})(y - \bar{y})}{\sqrt{\Sigma(x - \bar{x})^2 \Sigma(y - \bar{y})^2}}$$

Where r = correlation coefficient
x = amount of analyte
y = response of instrument

$\bar{x}$ = average of x values

y = average of y values

14.0 DOCUMENTATION

14.1 The Analytical Logbook records the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

14.1.1 If samples require reanalysis, a brief explanation of the reason must be documented in the Comments section.

14.2 Standards Preparation Logbook must be completed for all standard preparations. All information must be completed; the page must be signed and dated by the appropriate person.

14.2.1 The Accutest lot number must be cross-referenced on the standard vial.

14.3 Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

14.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

14.5 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information should also be verified during this review.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non hazardous aqueous wastes

15.2.2 Hazardous aqueous wastes

15.2.3 Chlorinated organic solvents

15.2.4 Non-chlorinated organic solvents

15.2.5 Hazardous solid wastes

15.2.6 Non-hazardous solid wastes

Table 1. TARGET COMPOUNDS		
Acetone	1,3-Dichlorobenzene	Methyl Methacrylate
Acetonitrile	1,4-Dichlorobenzene	4-Methyl-2-pentanone (MIBK)
Acrolein	Dichlorodifluoromethane	Methylene Bromide
Acrylonitrile	1,1-Dichloroethane	Methylene Chloride
Allyl Chloride	1,2-Dichloroethane	1-Methylnaphthalene (1)
Benzene	1,1-Dichloroethene	2-Methylnaphthalene (1)
Benzyl chloride	cis-1,2-Dichloroethene	Naphthalene
Bromobenzene	trans-1,2-Dichloroethene	2-Nitropropane (1)
Bromochloromethane	1,2-Dichloropropane	Pentachloroethane
Bromodichloromethane	1,3-Dichloropropane	Propionitrile
Bromoform	2,2-Dichloropropane	Propyl Acetate (1)
Bromomethane	1,1-Dichloropropene	n-Propylbenzene
2-Butanone (MEK)	cis-1,3-Dichloropropene	Styrene
Butyl Acetate (1)	trans-1,3-Dichloropropene	Tert Butyl Alcohol
n-Butyl Alcohol (1)	1,4-Dioxane	tert-Amyl Methyl Ether
n-Butylbenzene	Epichlorohydrin (1)	tert-Butyl Ethyl Ether
sec-Butylbenzene	Ethyl Acetate	1,1,1,2-Tetrachloroethane
tert-Butylbenzene	Ethyl Ether	1,1,2,2-Tetrachloroethane
Carbon Disulfide	Ethyl Methacrylate	Tetrachloroethene
Carbon Tetrachloride	Ethylbenzene	Tetrahydrofuran
Chlorobenzene	p-Ethyltoluene (1)	Toluene
Chlorodifluoromethane (1)	Freon 113	trans-1,4-Dichloro-2-Butene
Chloroethane	Heptane (1)	1,2,3-Trichlorobenzene
2-Chloroethyl Vinyl Ether	Hexachlorobutadine	1,2,4-Trichlorobenzene
Chloroform	Hexachloroethane	1,1,1-Trichloroethane
Chloromethane	Hexane (1)	1,1,2-Trichloroethane
Chloroprene (2-chloro-1,3-butadiene)	2-Hexanone	Trichloroethene
o-Chlorotoluene	Iodomethane (Methy iodide)	Trichlorofluoromethane
p-Chlorotoluene	IsoAmyl Alcohol (1)	1,2,3-Trichloropropane
Cyclohexane (1)	Isobutyl Alcohol	1,2,4-Trimethlylbenzene
Cyclohexanone	Isopropyl Acetate (1)	1,3,5-Trimethylbenzene
di-Isobutylene (1)	Isopropylbenzene	Vinyl Acetate
di-Isopropyl Ether	p-Isopropyltoluene	Vinyl Chloride
1,2-Dibromo-3-Chloropropane	Methacrylonitrile	Vinyltoluene (1)
Dibromochloromethane	Methyl Acetate (1)	m,p-Xylene
1,2-Dibromoethane	3 Methyl-1-Butanol (1)	o-Xylene
Dibromomethane (1)	Methyl Tert Butyl Ether	Ethanol
1,2-Dichlorobenzene	Methylcyclohexane (1)	Methyl Acrylate

(1) NELAC Accreditation is not offered for this compound. Results may not be useable for regulatory purposes in States where this accreditation option is not offered.

Table 2. RECOMMENDED OPERATING CONDITION	
Gas Chromatograph/ Mass Spectrometer	
Carrier Gas (linear velocity)	Helium at *30 cm/sec
Mass range	35 – 300 amu
Electron Energy	70 volts (nominal)
Scan time	not to exceed 2 sec. per scan
Injection port temperature	200 - 225 °C
Source temperature	200 - 250 °C
Transfer line temperature	220 - 280 °C
Analyzer temperature	220 - 250 °C
Gas Chromatograph temperature program*	
Initial temperature	*40 °C
Time 1	*3 minutes
Column temperature rate	*8 degrees/min.
Final temperature	*220 °C.- 240 °C
Total run time	*25 – 50 mins
Purge and Trap Device	
Purge time	9 min. (at 40 °C for low-level soil) SIM – 6 min @ 50 °C
Desorb**	4 min. at 190 °C
Bake	>10 min. at 210 °C
Transfer line	100 - 130 °C
Valve temperature	approx. transfer line temperature

* Parameter modification allowed for performance optimization provided operational and QC criteria is achieved.(must be approved by team leader/manager)

** Desorb time may require performance optimum between 2.0 and 4.0 minutes as dictated by trap manufacturers specifications or instrument characteristics

Table 2a – SIM Group Parameters		
Group No.	**Retention Time (minutes)**	**Ions**
1	0 – 10.8	58, 65, 66, 88
2	10.8 – 16.0	95, 174, 176, 98, 100, 70

Table 3. BFB KEY IONS AND ION ABUNDANCE CRITERIA	
Mass	**Ion Abundance Criteria**
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5-9% of mass 174
176	>95% and <101% of mass 174

177	5-9% of mass 176	

Table 4. INTERNAL STANDARD QUANTITION IONS	
Internal Standard	**Primary/Secondary Ions**
1,4-Difluorobenzene	114 / 63,88
Chlorobenzene-d5	117 / 82, 119
Pentafluorobenzene	168
1,4-Dichlorobenzene-d4	152 / 115, 150
Tert Butyl Alcohol-d9	65/66
Internal Standard (SIM)	
Tert Butyl Alcohol-d9	65/66

Table 5. SURROGATE QUANTITION IONS	
Surrogate Compound	**Primary/Secondary Ions**
1,2 Dichloroethane – d_4	102
Dibromofluoromethane	113
Toluene-d8	98
4-Bromofluorobenzene	95 / 174, 176

Table 6. CRITERIA FOR CCC AND SPCC		
Initial Calibration	Maximum % RSD for CCC is 30 %	
Continuing Calibration	Maximum % D for CCC is 20 %	
Calibration check compounds (CCC)	**Volatile Compound**	
	Vinyl chloride 1,1-Dichloroethene Chloroform 1,2-Dichloropropane Toluene Ethylbenzene	
System Performance Check Compounds (SPCC)	**Compound Name**	**Minimum RF**
	Chloromethane	0.1
	1,1-Dichloroethane	0.1
	Bromoform	0.1
	1,1,2,2-Tetrachloroethane	0.3
	Chlorobenzene	0.3

Table 7. Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)	Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)
Tert Butyl Alcohol-d9			Dibromomethane	93	95, 174
Tert Butyl alcohol	59	57	Di-isobutylene	57	
Ethanol	45	46	Epichlorohydrin (pp)	57	57, 49, 62, 51
Pentafluorobenzene			Ethyl methacrylate	69	69, 41, 99, 86, 114
1,1,1-Trichloroethane	97	99, 61	Heptane	57	
1,1-Dichlorethane	63	65, 63	Hexane	56	
1,1-Dichloroethene	96	61, 63	Isopropyl acetate	43	
2,2-Dichloropropane	77	97	Methyl cyclohexane	83	
2-Butanone (pp)	72	43, 72	Methyl methacrylate	69	69, 41, 100, 39
Acetone (pp)	58	43	n-Butanol (pp)	56	41
Acetonitrile (pp)	41	41, 40, 39	Propyl Acetate	43	
Acrolein (pp)	56	55,58	tert Amyl Methyl Ether	73	
Acrylonitrile (pp)	53	52, 51	Toluene	92	91
Allyl Chloride	41		Toluene-d_8	98	
Bromochloromethane	128	49, 130	trans-1,3-Dichloropropene	75	77, 39
Bromomethane	94	96	Trichloroethene	95	97, 130, 132
Carbon disulfide	76	78			
Chlorodifluouromethane	51	86	**Chlorobenzene-d5**	117	82,119
Chloroethane	64	66	1,1,1,2-Tetrachloroethane	131	133, 119
Chloroform	83	85	1,2-Dibromoethane	107	109, 188
Chloromethane	50	52	1,3-Dichloropropane	76	78
Chloroprene	53	53, 88, 90, 51	Bromoform	173	175, 254
cis-1,2-Dichloroethene	96	61, 98	Butyl Acetate	56	
Cyclohexane	84		Chlorobenzene	112	77, 114
Dibromofluoromethane	113		Dibromochloromethane	129	127
Dichlorodifluoromethane	85	87	Ethylbenzene	91	106
Dichloroethane-d_4	102	65	m-Xylene	106	91
Diethyl ether	74	45, 59	o-Xylene	106	91
Diisopropyl ether	45	102	p-Xylene	106	91
Ethyl acetate (pp)	88	43, 45, 61	Styrene	104	78
Ethyl tert Butyl Ether	59		Tetrachloroethene	164	129,131,166
Freon 113	151				
Iodomethane	142	127, 141	**1,4 Dichlorobenzene-d4**	152	115,150
Isobutyl alcohol (pp)	43	43, 41, 42, 74	1,1,2,2-Tetrachloroethane	83	131, 85
Methacrylonitrile (pp)	41	41, 67, 39, 52, 66	1,2,3-Trichlorobenzene	180	182, 145
Methyl Acetate	43	74	1,2,3-Trichloropropane	75	77
Methylene chloride	84	86, 49	1,2,4-Trichlorobenzene	180	182, 145
Methyl-t-butyl ether	73	57	1,2,4-Trimethylbenzene	105	120
Propionitrile (ethyl cyanide)(pp)	54	54, 52, 55, 40	1,2-Dibromo-3-chloropropane(pp)	75	155, 157
Tetrahydrofuran	42		1,2-Dichlorobenzene	146	111,148
trans-1,2-Dichloroethene	96	61, 98	1,3,5-Trimethylbenzene	105	120
Trichlorofluoromethane	151	101, 153	1,3-Dichlorobenzene	146	111, 148
Vinyl acetate	43	86	1,4-Dichlorobenzene	146	111, 148
Vinyl chloride	62	64	2-Chlorotoluene	91	126
Methyl Acrylate	55	85	4-Bromofluorobenzene	95	174, 176
1,4 Difluorobenzene	114	63, 88	4-Chlorotoluene	91	126

Table 7. Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)	Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)
1,1,2-Trichloroethane	83	97, 85	Benzyl chloride	91	91, 126, 65, 128
1,1-Dichloropropene	75	110, 77	Bromobenzene	156	77, 158
1,2 Dichloroethane	62	98	Cyclohexanone	55	
1,2 Dichloropropane	63	112	Hexachlorobutadiene	225	223, 227
1,4-Dioxane (pp)	88	88, 58, 43, 57	Hexachloroethane (pp)	201	166, 199, 203
2-Chloroethyl-vinylether (pp)	63	65, 106	Isopropylbenzene	105	120
2 – Hexanone	43	58,57,100	Naphthalene	128	-
2-Hexanone (pp)	43	58, 57, 100	n-Butylbenzene	91	92, 134
2-Nitropropane	46	-	n-Propylbenzene	91	120
3 Methyl –1 butanol	55		Pentachloroethane (pp)	167	167,130,132,165,169
4-Methyl-2-pentanone (pp)	100	43, 58, 85	p-isopropyltoluene	119	134,91
Benzene	78	-	sec-Butylbenzene	105	134
Bromodichloromethane	83	85, 127	tert-Buytlbenzene	119	91, 134
Carbon tetrachloride	117	119	trans-1,4-Dichloro-2-butene (pp)	53	88, 75
cis-1,3-Dichloropropene	75	77, 39			
			(pp) = Poor Purging Efficiency		

Table 7-1 SIM - Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)
Tert Butyl Alcohol-d9		
1,4-Dioxane	88	58
Toluene –d8	98	100
4-BFB	95	174, 176

Table 8. STANDARDS PREPARATION

A) Internal standard and Surrogate mixtures:

	a) 25/250 μg/ml	b) 250/2,500 μg/ml
Internal Standard Mixture (2,000 μg/ml)	1.25 ml	1 .25 ml
Tert Butyl Alcohol-d_9 (50,000 μg/ml)	0.5 ml	0.5 ml
Surrogate Mixture (2,500 μg/ml)	1 ml	1 ml
Methanol	97.25 ml	7.25 ml
Total	100 ml	10 ml

- 25/250 μg /ml internal standard and surrogate mixture: The mixture is prepared by measuring 1.25ml of 2,000 μg /ml Internal Standard Mixture (Ultra or equivalent), 0.5 ml of 50,000 μg/ml TBA-d_9 (Absolute or equivalent), 1 ml of 2,500 μg /ml Method 8260A Surrogate Standard Mixture (Ultra or equivalent) and bringing to 100 ml with methanol.

- 250/2,500 μg /ml internal standard and surrogate mixture: The mixture is prepared by measuring 1.25 ml of 2,000 μg /ml Internal Standard Mixture (Ultra or equivalent), 0.5 ml of 50,000 μg/ml TBA-d_9 (Absolute or equivalent), 1 ml of 2,500 μg /ml Method 8260A Surrogate Standard Mixture (Ultra or equivalent) and bringing to 10 ml with methanol.

- 100 μg/ml surrogate mixture: The solution is prepared at 100 μg/ml by measuring 0.4 ml of 2,500 μg/ml Method 8260A Surrogate Standard Mixture (Ultra or equivalent) and bringing to 10 ml with methanol.

- 25/250 μg /ml internal standard mixture: The solution is prepared by measuring 1.25 ml of 2,000 μg /ml Internal Standard Mixture (Ultra or equivalent), 0.5 ml of 50,000 μg/ml TBA-d_9 (Absolute or equivalent), and bringing to 100 ml with methanol.

- 250/2,500 μg /ml internal standard mixture: The solution is prepared by measuring 1.25 ml of 2,000 μg /ml Internal Standard Mixture (Ultra or equivalent), 0.5 ml of 50,000 μg/ml TBA-d_9 (Absolute or equivalent), and bringing to 10 ml with methanol.

B) Bromofluorobenzene (BFB):

	a) 25 μg/ml	b)) 250 μg/ml
BFB (25,000 μg/ml)	0.1 ml	0.1 ml

Methanol	ml	99.9	ml	9.9
Total	ml	100	ml	10

- 25 μg /ml solution for direct injection: The BFB is prepared at 25 μg /ml by measuring 0.1 ml of 25,000 μg /ml (Absolute Stock or equivalent) and diluting to 100 ml with methanol.

- 250 μg /ml solution for purging: The BFB is prepared at 250 μg /ml by measuring 0.1 ml of 25,000 μg /ml (Absolute Stock or equivalent) and diluting to 10 ml with methanol.

Table 8. STANDARD PREPARATION (Continued)

C) Secondary dilution standards:

2nd Dilution Standards	Stock Solution	Concentration (μg/ml)	Volume Added (μl)	Final Volume in Methanol (ml)	Final Concentration (μg/ml)
	EPA Method 524.2 Volatiles	2,000	2,500	50	100
	Acrolein	Neat (90%)	66.2		1,000
V8260 Mixture	Acrylonitrile*	Neat	25		500+
	Propionitrile**	Neat	58.9		1,000++
	Di-iso Butylene	Neat	7.1		100
	Cyclohexane	Neat	6.5		100
	Cyclohexanone	Neat	52.9		1,000
	Custom Volatiles Mix A	2,000	2,500	50	100
	Custom Volatiles Mix B	2,000 - 100,000	2,500		100 - 5,000
	Epichlorohydrin	Neat	21.4		500
V8260 Custom Mixture	Iso-Amyl alcohol	Neat	125		2,000
	2-Chloroethyl vinyl ether	Neat	20.1		500
	Ethyl tert-butyl ether	Neat	6.8		100
	Tert-Amyl methyl ether	Neat	6.56		100
	Benzyl chloride	Neat	4.6		100
Gas Mixture	VOC Gas Mixture	2,000	1,000	20	100

- 100 μg /ml V8260 mixture: The mixture is prepared at 100 μg /ml by measuring 2 ml of 2,000 μg /ml EPA Method 524.2 Volatiles stock standard, appropriate amount of some neat compounds, and bringing to 50 ml with methanol.
  * Acrylonitrile = 400 μg /ml (Neat) + 100 μg /ml (EPA Method 524.2 Volatiles)
  ** Propionitrile = 900 μg /ml (Neat) + 100 μg /ml (EPA Method 524.2 Volatiles)

- 100 μg /ml V8260 custom mixture: The mixture is prepared at 100 - 5,000 μg /ml by measuring 2.5ml of 2,000 μg /ml Custom Volatiles Mix A, 2.5 ml of 2,000 - 100,000 μg/ml Custom Volatiles Mix B, appropriate amount of some neat compounds, and bringing to 50 ml with methanol.

- 100 μg /ml gas mixture ***: The mixture is prepared at 100 μg /ml by measuring 1 ml of 2,000 μg /ml stock standard and bring to 20 ml with methanol.
  ******* Gas mixture should be prepared weekly.

Table 8. STANDARD PREPARATION (Continued)

D).1 Initial Calibration Standards: using DI water bring to 50 ml final volume: all mixtures used should be **secondary dilution** standards at **100 ppm**.

Standard and Surrogate Concentration	V8260 Mix (100 ppm)	V8260 Custom Mix (100 ppm)	Gas compound Mix (100 ppm)	Surrogate Mix (100ppm)
1 ppb	0.5 µl	0.5 µl	0.5 µl	0.5 µl#
2 ppb *	1.0 µl	1.0 µl	1.0 µl	1.0 µl#
5 ppb	2.5 µl	2.5 µl	2.5 µl	2.5 µl#
10 ppb *	5 µl	5 µl	5 µl	5 µl#
20 ppb	10 µl	10 µl	10 µl	10 µl#
50 ppb	25 µl	25 µl	25 µl	25 µl#
100 ppb	50 µl	50 µl	50 µl	50 µl#
200 ppb	100 µl	100 µl	100 µl	100 µl#
300 ppb *	150 µl	150 µl	150 µl	150 µl#
400 ppb *	200 µl	200 µl	200 µl	200 µl#

* depending upon the instrument.
\# See Section 10.2.2.1 for correction factor.

- When calibrating for Method 5035 low-level soil samples, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of each standard into vial. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds.

D).2 Initial Calibration Standards for 1,4-Dioxane using SIMS

Standard and Surrogate Concentration (ppb)	1,4-Dioxane Solution (100ppm)	Surrogate Mix (100ppm)	DI Water – Final Volume (ml)
2	2 µl	1 µl	100
5	5 µl	2 µl	100
25	25 µl	5 µl	100
50	25 µl	2.5 µl	50
100	50 µl	5 µl	50
200	100 µl	10 µl	50
400	200 µl	20 µl	50

Table 8. STANDARD PREPARATION (Continued)

E) Continuing Calibration Standard: using DI water bring to 50 ml final volume: All mixtures used are secondary dilution standards at 100 ppm.

Concentration	V8260 Mix (100 ppm)	V8260 Custom Mix (100 ppm)	Gas compound Mix (100 ppm)
50 ppb	25 µl	25 µl	25 µl

- When calibrating for Method 5035 low-level soil samples, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of the continuing calibration standard into vial. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds.

F) Blank Spike (BS): using DI water bring to 50 ml final volume: All mixtures used are 100 ppm secondary dilution standards.

Concentration	V8260 Mix (100 ppm)	V8260 Custom Mix (100 ppm)	Gas compound Mix (100 ppm)
50 ppb	25 ul	25 ul	25 ul

For lower detection level required (test code: V8260LL)

Concentration	V8260 Mix (100 ppm)	V8260 Custom Mix (100 ppm)	Gas compound Mix (100 ppm)
20 ppb	10 ul	10 ul	10 ul

- When calibrating for Method 5035 low-level soil samples, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of the blank spike into vial. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds.

Table 9. GUIDELINE FOR DILUTION PREPARATION

Water Sample

Dilution	Sample amount taken	Final volume A (volumetric)	Take from final volume A	Final volume B (volumetric)
1:2	25 ml	50 ml		
1:5	10 ml	50 ml		
1:10	5 ml	50 ml		
1:20	2.5 ml	50 ml		
1: 25	2 ml	50 ml		
1:50	1 ml	50 ml		
1:100	0.5 ml	50 ml		
1:200	250 µl	50 ml		
1:250	200 µl	50 ml		
1:500	100 µl	50 ml		
1:1000	50 µl	50 ml		
1:2000	25 µl	50 ml		
1:2500	20 µl	50 ml		
1:5000	10 µl	50 ml		
1:10000	0.5 ml	50 ml	0.5 ml	50 ml
1:20000	0.5 ml	50 ml	250 µl	50 ml
1:25000	0.5 ml	50 ml	200 µl	50 ml
1:50000	0.5 ml	50 ml	100 µl	50 ml
1:100000	0.5 ml	50 ml	50 µl	50 ml

Soil-Low level (Non-Encore sample)

Dilution	Sample amount taken	Final volume
1:2	2.5 gram	5 ml
1:5	1 gram	5 ml
1:10	0.5 gram	5 ml

Soil-medium level

Additional Dilution	Sample in Methanol amount taken	Final volume (volumetric)
1:1	1 ml	50 ml
1:2	0.5 ml	50 ml
1:5	200 µl	50 ml

1:10	100 µl	50 ml
1:20	50 µl	50 ml
1: 25	40 µl	50 ml
1:50	20 µl	50 ml
1:100	10 µl	50 ml
1:200	5 µl	50 ml
1:250	4 µl	50 ml
1:500	2 µl	50 ml

Table 10. REPORTING LIMITS

Compound	Water	Soil	Compound	Water	Soil
	µg/l	µg/kg		µg/l	µg/kg
Chlorodifluoromethane	5	5	Chloroform	5	5
Dichlorodifluoromethane	5	5	Freon 113	5	5
Chloromethane	5	5	Methacrylonitrile	10	10
Vinyl chloride	5	5	Butyl Acetate	5	5
Bromomethane	5	5	1,1,1-Trichloroethane	5	5
Chloroethane	5	5	Heptane	5	5
Trichlorofluoromethane	5	5	n-Propyl acetate	5	5
Ethyl ether	5	5	2-Nitropropane	10	10
Acrolein	50	50	Tetrahydrofuran	10	10
1,1-Dichloroethene	2	2	2-Chloroethyl Vinyl Ether	20	20
Tertiary butyl alcohol	50	50	n-Butyl alcohol	250	250
Acetone	5	5	Cyclohexane	5	5
Methyl acetate	5	5	Carbon Tetrachloride	1	1
Allyl chloride	5	5	1,1-Dichloropropene	5	5
Acetonitrile	100	100	Isopropyl Acetate	5	5
Iodomethane	25	25	Benzene	1	1
Iso-butyl alcohol	50	50	1,2-Dichloroethane	2	2
Carbon disulfide	5	5	Trichloroethene	1	1
Methylene chloride	2	2	Methyl methacrylate	10	10
Methyl tert butyl ether	1	1	1,2 Dichloropropane	1	1
Trans-1,2-Dichloroethene	5	5	Di-isobutylene	5	5
Di-isopropyl ether	5	5	Dibromomethane	5	5
2-Butanone	5	5	1,4 Dioxane	125	125
1,1-Dichloroethane	2	2	Bromodichloromethane	1	1
Hexane	5	5	cis-1,3-Dichloropropene	1	1

Chloroprene	5	5	4-Methyl-2-pentanone	5	5
Acrylonitrile	5	5	Toluene	1	1
Vinyl acetate	10	10	trans-1,3-Dichloropropene	1	1
Ethyl acetate	5	5	Ethyl methacrylate	10	10
2,2-Dichloropropane	5	5	1,1,2-Trichloroethane	3	3
Cis-1,2-Dichloroethene	5	5	2-Hexanone	5	5
Bromochloromethane	5	5	Cyclohexanone	5	5

Table 10. REPORTING LIMITS (Continued)

Compound	Water	Soil	Compound	Water	Soil
	µg/l	µg/kg		µg/l	µg/kg
Tetrachloroethene	1	1	4-Chlorotoluene	5	5
1,3-Dichloropropane	5	5	1,3,5-Trimethylbenzene	5	5
Dibromchloromethane	5	5	tert-Butylbenzene	5	5
1,2-Dibromoethane	2	2	1,2,4 Trimethylbenzene	5	5
Chlorobenzene	2	2	sec-Butylbenzene	5	5
1,1,1,2-Tetrachloroethane	5	5	1,3-Dichlorobenzene	5	5
Ethylbenzene	1	1	p-Isopropyltoluene	5	5
M,p-Xylene	1	1	1,4-Dichlorobenzene	5	5
o-Xylene	1	1	1,2-Dichlorobenzene	5	5
Styrene	5	5	n-Butylbenzene	5	5
Bromoform	4	4	1,2-Dibromo-3-choropropane	10	10
Isopropylbenzene	2	2	1,2,4-Trichlorobenzene	5	5
Bromobenzene	5	5	Hexachlorobutadiene	5	5
1,1,2,2-Tetrachloroethane	2	2	Naphthalene	5	5
Trans-1,4-Dichloro-2-butene	5	5	1,2,3-Trichlorobenzene	5	5
1,2,3-Trichloropropane	5	5	Epichlorohydrin	100	100
n-Proplybenzene	5	5	3-Methyl-1-butanol	5	5
2-Chlorotoluene	5	5	Hexachloroethane	5	5
Ethanol	50	--	Methyl Acrylate	5	--
Benzyl Chloride	1	--			

Table 11. COMPOUNDS THAT MAY EXHIBIT CARRYOVER

Compound
1,2,4-Trichlorobenzene
Hexachlorobutadiene
Naphthalene
1,2,3-Trichlorobenzene

Accutest Laboratories
Standard Operating Procedure

Lab Manager:_____

QA Manager:_____

Effective Date:_____

TITLE: PERCENT SOLIDS and TOTAL SOLIDS in SOIL/SOLID MATRICES

METHOD REFERENCES: SM18 2540G , ASTM Method D4643-00

Revised Sections*: 1.3, 6.2*

1.0 SCOPE AND APPLICATION

1.1 This method is for the measurement of the percent solids or total solids in samples of soil, sludge, or other solid material. This method is based on a method SM18 2540G and on ASTM method 4643-93.

1.2 The oven drying techniques for percent solids as described in this SOP can be applied to any sample type where percent solids can be determined. The microwave drying technique for percent solids described in this SOP can only be applied to samples which do not fall into the categories listed: lower percent solids (<50%), sludges or any other sample with a high organic content, samples containing high amounts of hydrated materials or larger clumps or particles, and samples with high TDS in the pore water (i.e. marine deposits). In addition, samples designated for hexavalent chromium analysis or NYASPB reporting cannot be dried in the microwave.

1.3 Sludge samples for regulatory reporting must follow the sludge procedure outlined in this SOP. These samples are tracked using the product code SLDGSOL

2.0 SUMMARY OF METHOD

2.1 A homogeneous aliquot of sample is placed in a tared dish and weighed. The wet sample is then dried to constant weight. The difference between the initial and final weight indicates the amount of water in the sample. Percent solids are calculated using the dry weight of the sample divided by the total weight of the sample. Percent moisture is calculated using the weight of water in the sample divided by the total weight of the sample.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Not applicable.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing

procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE (DUP): A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed.

5.0 HEALTH AND SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the Accutest Laboratory Chemical Hygiene Plan. Always wear a lab coat and glasses and the appropriate gloves when analyzing samples for percent solids.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIME

6.1 No specific holding time is in place for the calculation of percent solids in soil samples. The samples should be stored at 0 to 4°C until the time of analysis.

6.2 Sludge samples for regulatory reporting must be analyzed within 7 days from time of collection.

7.0 APPARATUS AND MATERIALS

The items listed below are needed for the sample analysis.

7.1 Crucibles. Capable of heating to 105°C and capable of being heated in a microwave.

7.2 Two or three place balance. All balances must have their calibration verified with Class S weights daily before the analysis of each batch of samples. Note: A 3 place balance is required for CLP samples.

7.3 Stainless Steel or Teflon coated Spatulas

7.4 Drying oven capable of maintaining a constant temperature at 103 to 105°C.

7.5 Variable temperature microwave oven.

7.6 Dessicator with dessicant.

8.0 STANDARDS AND REAGENTS

8.1 No special standards or reagents are required for this method.

9.0 INTERFERENCES

9.1 When heated at 103 to 105°C, samples will lose moisture, but can also lose certain volatile

components and ammonium carbonate. When heated in the microwave, there is a possibility that the soil is overheated if the microwave is not cycled properly. Microwave heating should not be used for any samples with high organic content (black or oily appearance), with low percent solids (50% or less), or on soils containing significant amounts of hydrated materials or soils in which the pore water contains high dissolved solids (for example, marine deposits).

10.0 SAMPLE CLASSIFICATION PROCEDURE.

10.1 Each sample must first be classifed as one of the following three types; a) a soil or solid that can be analyzed using the microwave method, b) a soil or solid that can be analyzed using the oven %solids method, or c) a sludge being analyzed for regulatory purposes that must use the sludge method. Classify the sample for analysis by microwave or oven. Certain sample types and certain protocols do not allow for the use of the microwave method. Samples which fall into any of the categories listed below should not be analyzed using the microwave.

10.1.1 Soils or solids to be analyzed by the microwave method must not be in any of the following categories.

10.1.1.1 Any NYASPB samples.

10.1.1.2 Any samples requiring analysis for hexavalent chromium (XCr).

10.1.1.3 Any samples with high organic content.

10.1.1.4 Any samples containing significant amounts of hydrated materials or with larger clumps or particles.

10.1.1.5 Any samples with low percent solids (< 50%).

10.1.1.6 Any samples in which the pore water contains high dissolved solids (for example marine deposits.)

10.1.1.7 Any sludge samples being reported for regulatory purposes.

10.1.2 Soils or solids to be analyzed by the oven %solids method can be any soils or solids that are not sludge samples being reported for regulatory purposes.

10.1.3 Sludges to be reported for regulatory purposes must be analyzed following the sludge %solids/total solids method.

11.0 MICROWAVE ANALYSIS METHOD FOR PERCENT SOLIDS/PERCENT MOISTURE

11.1 Dry the crucibles to be used at 103 to 105°C for a minimum of 1 hour or for 20 minutes at 50 to 70 percent power in the microwave. Allow to cool in a dessicator.

11.2 Tare the balance to zero. Before weighing any samples, make sure that the balance calibration has been checked with at least 2 class S weights covering the range of use before calibration and meets the specifications listed in front of the balance log. Normally 3 weights are used.

11.3 After the balance has been tared, weight the crucible(s) and record the weights. This may be done electronically or manually.

11.4 Mix the sample well using a stainless steel or teflon coated spatula. Do not just mix the top of the sample, but make sure that the whole sample is well mixed. Remove any stones, twigs, etc. from the sample.

11.4.1 If the sample jar is too full or the sample is difficult to mix, then empty the sample into a large stainless steel or ceramic bowl and stir well there before taking a sample aliquot.

11.5 Remove approximately a 5 to 20 g aliquot from the mixed sample and place it in the tared crucible. For at least one sample of every 20, set up a sample duplicate.

11.5.1 For any samples where it is difficult to obtain a representative sample aliquot, increase the sample aliquot size to 25 to 50 g.

11.5.2 If limited sample is available, smaller weights may be used if the aliquots are well homogenized.

11.5.3 Using the spatula, crush any large lumps in the aliquot to be weighed and dried.

11.6 Place a batch of 20 samples in the microwave and dry them for approximately 10 minutes at 50 to 70 percent power. Let the samples cool for 5 minutes and then dry them for an additional 10 minutes at 50 to 70 percent power.

11.6.1 Power levels and times will need to be adjusted for smaller batches of samples. Check with area supervisors for assistance.

11.7 Remove the samples and let cool for 5 minutes. If they are going to be out for longer than 10 minutes, place them in a dessicator before weighing.

11.8 Weigh the samples on the balance and record the final weights. This can be done manually or electronically. Take at least 2 samples and stir them with a spatula, making sure not to retain any sample particles on the spatula. The 2 samples used for this check should be the 2 that have the highest moisture content (lowest percent solids) in the batch. Any samples with < 50% solids must be submitted for redo using the oven drying method.

11.9 Place these 2 samples back in the microwave and microwave for 3 to 5 minutes at 50 percent power. Remove the samples and let cool for 5 minutes.

11.10 Weigh the samples on the balance and record the final weights. Again, this can be done

either manually or electronically.

11.10.1 If the sample weights on the redry differ by more than 0.05 g or 4%, whichever is less, from the previous weights, then then the whole batch should be stirred and redried until all percent solids agree within this criteria.

12.0 OVEN ANALYSIS METHOD FOR PERCENT SOLIDS/PERCENT MOISTURE (NOT TO BE USED FOR SLUDGES).

12.1 Dry the crucibles to be used at 103 to 105°C for a minimum of 1 hour. Allow to cool in a dessicator.

12.2 Tare the balance to zero. Before weighing any samples, make sure that the balance calibration has been checked with at least 2 class S weights covering the range of use before calibration and meets the specifications listed in front of the balance log. Normally 3 weights are used.

12.3 After the balance has been tared, weight the crucible(s) and record the weights. This may be done electronically or manually.

12.4 Mix the sample well using a stainless steel or teflon coated spatula. Do not just mix the top of the sample, but make sure that the whole sample is well mixed. Remove any stones, twigs, etc. from the sample.

12.4.1 If the sample jar is too full or the sample is difficult to mix, then empty the sample into a large stainless steel or ceramic bowl and stir well there before taking a sample aliquot.

12.5 Remove approximately a 5 to 20 g aliquot from the mixed sample and place it in the tared crucible. For at least one sample of every 20, set up a sample duplicate.

12.5.1 For any samples where it is difficult to obtain a representative sample aliquot, increase the sample aliquot size to 25 to 50 g.

12.5.2 If limited sample is available, smaller weights may be used if the aliquots are well homogenized.

12.5.3 Using the spatula, crush any large lumps in the aliquot to be weighed and dried.

12.6 Place a batch of samples in the oven at 103 to 105°C. The samples must remain in the oven for a minimum of 1 hour and up to 24 hours. Drying times should not exceed 24 hours unless specific project instructions are being followed. .Record the drying time.

12.6.1 Samples with high organic content must stay in the oven for a minimum of 8 to 12 hours.

12.6.2 Remove the samples and let cool for 5 minutes. If they are going to be out for longer than 10 minutes, place them in a dessicator before weighing.

12.6.3 Weigh the samples on the balance and record the final weights. This can be done manually or electronically.

12.6.3.1 If the samples are in the oven less than 12 hours, then place all of the samples back in the oven for a minimum of 1 hour at 103 to 105°C.

12.6.3.2 If the sample weights on the redry differ by more than 0.05 g or 4%, whichever is less, from the previous weights, then then the whole batch must be stirred and redried until all percent solids agree within this criteria.

12.7 Percent solids are calculated using the equation shown below.

Percent moisure = 100 – percent solids.

$$\text{Percent solids} = 100 \times \frac{\text{(final dry weight + crucible) - crucible tare weight}}{\text{(wet weight + crucible) – crucible tare weight}}$$

13.0 ANALYSIS METHOD FOR PERCENT SOLIDS/PERCENT MOISTURE/TOTAL SOLIDS FOR SLUDGE MATRICES.

Below is a step-by-step procedure for the analysis of sludge samples for percent solids and percent moisture and total solids.

13.1 Dry the crucibles to be used at 103 to 105°C for a minimum of 1 hour. Allow to cool in a dessicator.

13.2 Tare the balance to zero. Before weighing any samples, make sure that the balance calibration has been checked with at least 2 class S weights covering the range of use before calibration and meets the specifications listed in front of the balance log. Normally 3 weights are used.

13.3 After the balance has been tared, weight the crucible(s) and record the weights. This may be done electronically or manually.

13.4 Mix the sample well using a stainless steel or teflon coated spatula. Do not just mix the top of the sample, but make sure that the whole sample is well mixed. Remove any stones, twigs, etc. from the sample.

13.4.1 If the sample jar is too full or the sample is difficult to mix, then empty the sample into a large stainless steel or ceramic bowl and stir well there before taking a sample aliquot.

13.5 Remove approximately a 25 to 50 g aliquot from the mixed sample and place it in the tared crucible. For at least one sample of every 10, set up a sample duplicate.

13.5.1 If limited sample is available, smaller weights may be used if the aliquots are well

homogenized.

13.5.2 Using the spatula, crush any large lumps in the aliquot to be weighed and dried.

13.6 Place a batch of samples in the oven at 103 to 105°C. The samples must remain in the oven for a minimum of 8 hours and up to 24 hours. Drying times should not exceed 24 hours unless specific project instructions are being followed. .Record the drying time.

13.6.1 Remove the samples and place them in a dessicator to cool.

13.6.2 Once the samples are cool, weigh them on the balance and record the final weights. This can be done manually or electronically.

13.6.3 Then place all of the samples back in the oven for a minimum of 1 hour at 103 to 105°C. Repeat steps 13.6.1 and 13.6.2.

13.6.4 If the sample weights on the redry differ by more than 0.05 g or 4%, whichever is less, from the previous weights, then the affected sample(s) must be stirred and redried until all percent solids agree within this criteria.

13.7 Percent solids are calculated using the equation shown below.

Percent moisure = 100 – percent solids.

$$\text{Percent solids} = 100 \times \frac{(\text{final dry weight} + \text{crucible}) - \text{crucible tare weight}}{(\text{wet weight} + \text{crucible}) - \text{crucible tare weight}}$$

14.0 QC REQUIREMENTS

14.1 Each batch of 20 samples will include a duplicate sample. The exception is for sludge samples where a duplicate is required for one in 10 samples.

14.2 The limit for duplicate samples is 5 % RPD. If the RPD values are outside of this range, and all other method quality control is within limits, then sample non-homogeneity should be suspected. A description of the duplicate sample appearance should be provided for all batches where the 5% RPD is not met. In general, it is recommended that batches with samples with high RPD's be reanalyzed to confirm the original results.

The calculation for %RPD $= \frac{100 \times (\text{Sample result} - \text{Duplicate result})}{(\text{Sample result} + \text{Duplicate result})/2}$

14.3 The balance calibration must be verified at a minimum of 2 levels bracketing the range of weights measured each day before use. The calibration must meet the specifications listed in the balance logbook. If they do not, the balance must be recalibrated and rechecked before any samples can be analyzed.

14.4 For samples dried in the microwave, a minimum of 2 redries is required for every 20 samples. For solid or soil samples dried in the oven, all samples must be redried if the

original drying time is less than 12 hours. For sludge samples, all samples must be redried. Redries must match within 0.05 g or 4% of the original weight, whichever is less.

15.0 DOCUMENTATION REQUIREMENTS

15.1 All data regarding the analysis must be recorded on the data worksheet. Make sure that all sample information is included on these sheets. Any unusual characteristics of the samples should be noted in the comment section. This can be done electronically or manually. Make sure to double check crucible and sample ID's when they are recorded.

15.2 Initial balance calibrations must be recorded in the balance calibration check log for each balance.

16.0 DATA REVIEW AND REPORTING

16.1 All samples should be updated to QC batches in the LIMS system. The analyst is responsible for reviewing all data for compliance with the QC outlined in this SOP. They are responsible for making sure that the raw data is fully documented.

16.2 After the analyst review is completed, the supervisor or a designated reviewer shall review the run for technical compliance to the SOP. The supervisor is also responsible for making sure that the QC calculations are done correctly and responsible for reviewing the data entry into the LIMS. No LIMS entry review is necessary when the data is electronically transferred.

16.3 After the supervisor or designated reviewer completes their review, the data is released for client access in the LIMS. The raw data is submitted to the area manager. The manager will periodically review data for technical completeness. The raw data is then transferred to the report generation department.

17.0 POLLUTION PREVENTION & WASTE MANAGEMENT

17.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in the waste management SOP.

17.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

17.2.1 Non hazardous aqueous wastes.

17.2.2 Hazardous aqueous wastes

17.2.3 Chlorinated organic solvents

17.2.4 Non-chlorinated organic solvents

17.2.5 Hazardous solid wastes

17.2.6 Non-hazardous solid wastes

Lab Manager:_____

QA Manager:_____

Effective Date:_____

TITLE: DOCUMENTATION OF EQUIPMENT MAINTENANCE

Revised Sections*: *Modified 7.4, 10.1.1, 10.6 & 11.1

1.0 Scope & Application

1.1 Documenting procedures for the preventive maintenance, repair, and instrument removal from operation. Documentation occurs in individual instrument maintenance logbooks and on instruments as in or out of service.

2.0 Summary

2.1 Laboratory equipment requires routine preventive maintenance, repair by technical staff or outside vendors, and removal from service when the equipment is deemed inadequate to perform to a method specification. Proper documentation and/or labeling are required for these processes. With accurate and concise documentation, the maintenance history of an instrument can aid in future troubleshooting, prevention, and tracking repetitive problems.

3.0 Method Detection Limit: N/A

4.0 Definitions

DAILY MAINTENANCE. Procedures performed by the instrument operator prior to use of the instrument to assure that the equipment is in acceptable operating conditions.

LOCK OUT-TAG OUT. Safety procedures used by individuals performing instrument maintenance to assure that equipment being serviced does not inadvertently energize and cause injury to any individual within proximity of the equipment.

LOGBOOK. A bound series of forms used to record daily maintenance and non-routine maintenance activities.

NON-ROUTINE MAINTENANCE. Repair procedures that cannot be performed by in-house staff, which require the assistance of external service professionals.

5.0 Health & Safety

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual and Accutest Safety Policy, which includes the use of safety glasses and lab coats. Handle all acids, which are corrosive with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 Safety glasses must be worn when servicing any equipment.

5.3 Solvent resistant gloves when handling solvents for cleaning or rinsing components.

5.4 Power plugs must be unplugged when servicing electronic components.

5.5 Lock-out and Tag-out hardware must be used when equipment is hard wired for power.

6.0 Collection, Preservation, & Holding Times: N/A

7.0 Apparatus and Materials

7.1 Organics - GC/GCMS

- 7.1.1 Septum
- 7.1.2 Injection liners
- 7.1.3 Column ferrules
- 7.1.4 Injection port disc
- 7.1.5 Injection port Welman Assembly
- 7.1.6 Jet separator
- 7.1.7 Columns
- 7.1.8 FID jet
- 7.1.9 Mass Spec source parts

7.2 Organics - Concentrators

- 7.2.1 Purge tubes
- 7.2.2 Traps
- 7.2.3 Transfer lines
- 7.2.4 Syringe

7.3 Organics - ASE

- 7.3.1 Frits and O-rings (clog)

7.4 Inorganics - Metals

- 7.4.1 ICP/ICPMS torches
- 7.4.2 ICP/ICPMS nebulizers
- 7.4.3 ICP/ICPMS injector tubes
- 7.4.4 ICP/ICPMS pump tubings
- 7.4.5 ICP/ICPMS rinse lines
- 7.4.6 ICP/ICPMS internal standard mixing coils
- 7.4.7 ICP/ICPMS autosamplers
- 7.4.8 ICPMS cone
- 7.4.9 ICPMS skimmer cone

7.5 Inorganics - Wet chemistry

- 7.5.1 Lachat tubing
- 7.5.2 Lachat UV lamps
- 7.5.3 IC columns
- 7.5.4 IC tubing

7.5.5 TOX quartz wool
7.5.6 TOX pyrolysis
7.5.7 TOX electrodes
7.5.8 TOX O-rings
7.5.9 TOC Column catalyst
7.5.10 TOC needle
7.5.11 TOC O-rings
7.5.12 HG permeation tube
7.5.13 HG vapor/ liquid separator
7.5.14 HG Pump tubing
7.5.15 HG Lamp
7.5.16 HG Cell

7.6 General

7.6.1 Out of Service Labels
7.6.2 Lock-in Lock-out tags

8.0 Standards & Reagents

8.1 Polishing compound for GC/MS source cleaning.

8.2 Purge and trap grade methanol for source rinsing and transfer line flushing.

9.0 Interferences: N/A

10.0 Procedure – Modifications to instrument hardware during the performance of instrument maintenance procedures must be documented in the instrument maintenance logbook.

10.1 Organics - GC/GCMS. The following items must be checked and/or replaced at the specified frequency. GC/MS maintenance activities are performed daily.

10.1.1 Septum - replace daily or after 30 to 50 injections for semivolatile instruments; replace monthly or after 30 to 50 injections for volatile instruments.

10.1.2 Injection liners - replace or clean daily for semivolatile instruments.

10.1.3 Column ferrules - Replace when re-installing used columns or new columns.

10.1.4 Injection port disc - Clean with methanol and/or polishing compounds when buildup of sample residue or active sites affecting chromatography performance.

10.1.5 Injection port Welman Assembly - replace when leaks. Clean purge line when buildup of heavy boiling extract residues.

10.1.6 Jet separator - Clean when clogged.

10.1.7 Columns - Clip injection port side to eliminate active sites or residue buildup. Symptom of compound breakdown and sensitivity loss.

10.1.8 FID jet - Clean with methanol when clogged or loss of sensitivity.

10.1.9 Mass Spec source parts - Clean with polishing compound and methanol rinse when dirty. Symptom of sensitivity loss, high mass loss, and compound stability problems.

10.2 Organics - Concentrators. The following items must be checked and/or replaced at the specified frequency. Concentrator maintenance activities are performed daily.

10.2.1 Purge tubes - Rinse when sample residue is evident.

10.2.2 Traps - Bake out to remove residual contaminants. Replace when over contaminated as in sample foaming or performance degradation.

10.3 Transfer lines - rinse with methanol when contaminated or clogged.

10.4 Syringe - Clean with methanol and bake when sample residue evident. Perform daily.

10.5 Organics - ASE. The following items must be checked and/or replaced at the specified frequency.

10.5.1 Frits and O-rings - Clean with methanol when clogged or replace.

10.6 Inorganics - Metals. The following items must be checked and/or replaced at the specified frequency.

10.6.1 ICP/ICPMS torches - Clean or replace when loss in sensitivity.

10.6.2 ICP/ICPMS nebulizers - Clean or replace when clogged or contaminated affecting sensitivity.

10.6.3 ICP/ICPMS injector tubes - Clean or replace when clogged or contaminated affecting sensitivity.

10.6.4 ICP/ICPMS pump tubings - Clean or replace when clogged or contaminated.

10.6.5 ICP/ICPMS rinse lines - Clean or replace when clogged or contaminated.

10.6.6 ICP/ICPMS internal standard mixing coils - Clean or replace when clogged or contaminated.

10.6.7 ICP/ICPMS autosamplers - Clean the slides on the autosampler with methanol and wipe them with a KimWipe saturated with Teflon spray once per day.

10.6.8 ICPMS cone and skimmer cone - Clean when clogged or contaminated.

10.6.9 ICP vacuum instrument - Check and change the oil on the vacuum pump at a minimum of once every 6 months or whenever the vacuum is greater than 25 to 30.

10.7 Inorganics - Wet chemistry. The following items must be checked and/or replaced at the specified frequency.

10.7.1 Lachat tubing - Clean or replace when clogged or contaminated.

10.7.2 Lachat UV lamps - Replace when loss of sensitivity.

10.7.3 IC columns - Rinse or replace when chromatography is affected as in peak shape, peak separation and sensitivity variations.

10.7.4 IC tubing - Replace when clogged or contaminated.

10.7.5 TOX quartz wool - Replace when low recoveries observed.

10.7.6 TOX pyrolysis - Replace when badly devitrified or cracked.

10.7.7 TOX electrodes - Clean when oxidation coating is evident or loss in sensitivity on cell checks.

10.7.8 TOX O-rings - Replace when worn or leaking.

10.7.9 TOC Column catalyst - Replace when flow is reduced or particulate buildup.

10.7.10 TOC needle - Replace when worn.

10.7.11 TOC O-rings - Replace when worn or leaking.

10.7.12 HG permeation tube - Replace when loss of sensitivity.

10.7.13 HG vapor/ liquid separator - Replace when loss of sensitivity.

10.7.14 HG Pump tubing - Clean or replace when clogged or contaminated.

10.7.15 HG Lamp - Replace when loss of sensitivity.

10.7.16 HG Cell - Clean with residue buildup or replace when cracked.

10.8 General

10.8.1 Out of Service Labels - Any equipment that is deemed unusable or out of service for long term repair must be tagged with an out of service label until repaired or disposed of.

10.8.2 Lock-out Tag-out hardware - Any equipment that is hard wired must have Lock out tag on instrument along with circuit breaker panel to eliminate risk of injury of a premature power up by unaware personnel. This also applies to outside contractors servicing equipment.

10.8.3 Outside contractor service - Equipment being repaired by outside service personnel must also be documented in the instrument maintenance logbooks.

11.0 Quality Assurance

11.1 Supervisors must review and sign instrument maintenance logbooks quarterly.

12.0 Documentation

12.1 Documentation of all preventive maintenance and instrument repair must be recorded in individual instrument maintenance logbooks. Most of the routine preventive maintenance items are listed in the logbooks and checked off with date performed. Outside contractor repair is documented in detail under non-routine maintenance.

13.0 Data Review & Reporting

13.1 Supervisors must regularly review and sign instrument maintenance logbooks.

14.0 Pollution Prevention and Waste Management

14.1 Any solvents used for cleaning must be disposed in properly labeled container.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, ESM003. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non hazardous aqueous wastes.

14.2.2 Hazardous aqueous wastes

14.2.3 Chlorinated organic solvents

14.2.4 Non-chlorinated organic solvents

14.2.5 Hazardous solid wastes

14.2.6 Non-hazardous solid wastes

15.0 References: None

ATTACHMENT E - New York State Department Of Environmental Conservation
Analytical Service Protocol

NEW YORK STATE

DEPARTMENT OF ENVIRONMENTAL CONSERVATION

ANALYTICAL SERVICE PROTOCOL

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

July 2005

EXHIBIT B

TABLE OF CONTENTS

PART I -- CONTRACT REPORTS/DELIVERABLES SCHEDULE AND DISTRIBUTION

1.0 Summary Table

The following table details the Protocol reporting and deliverable requirements, their schedule, and the distribution that is required for each. Detailed requirements for each lettered "Item" listed in the chart are given in Part II of this Exhibit.

ITEM	DESCRIPTION	# of COPIES[1]	DELIVERY SCHEDULE	DISTRIBUTION		
				1	2	3
A	Standard Operating Procedures (SOPs)	1	60 days after notification of contract award, and as required in Exhibit E.	X		
B	Quality Assurance Management Plan (QAMP)	1	60 days after notification of contract award, and as required in Exhibit E.	X		
C	Weekly Sample Receipt Summary	1	The Wednesday following the calender week samples are received.	X		
D[2]	Sample Data Summary Package	2	30 days after the VTSR[3] of the last sample in the Sample Delivery Group (SDG[4]).	As Directed		
E[2]	Sample Data Package (.PDF)	1	30 days after the VTSR[3] of the last sample in the SDG[4].	X		X
F[2]	Electronic Data Deliverables (EDD)	1	30 days after the VTSR[3] of the last sample in the SDG[4].	X		X
G	Electronic Instrument Data	1	Retain for 3 years after data submission, submit within 7 days of receipt of written request from BWAM.	As Directed		
H	Samples and Extracts[5]	N/A	Retain for 365 days after data submission, submit within 7 days of receipt of written request from BWAM.	As Directed		
I	Full Verification of Instrument Parameters	1	Retain for 3 years after data submission, submit within 7 days of receipt of written request from BWAM.	As Directed		
J	Preliminary Results[6,7]	2	When requested, within 72 hours after receipt of designated samples.		X	X
K	Results of PE sample(s)	1	30 days after receipt of such Performance Evaluation (PE) sample(s).	X		

Notes (for Summary Table)

[1] The number of copies specified is the number of copies required to be delivered to each recipient, for that item.

[2] Deliverables for Items D, E, and F are to be reported total and complete. Concurrent delivery is required. Delivery shall be made such that all designated recipients receive all the items they are scheduled to receive on the same calendar day. If a deliverable item due on the same date as other deliverable items is late, all items scheduled to be due on that day shall be considered late as well. If the deliverables are due on a Saturday, Sunday, or State holiday, then they shall be delivered on the next business day.

[3] Validated Time of Sample Receipt (VTSR) is the date of sample receipt at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report/Chain of Custody Record. Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less with the same laboratory turnaround and not exceeding 20 samples [excluding performance Evaluation (PE) Samples]. Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received. See Exhibit A for further description.

[4] Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less and not exceeding 20 samples [excluding Performance Evaluation (PE) samples]. Note that preliminary results have no impact on defining the SDG. Data for all samples in the SDG are due concurrently, unless specified otherwise in a project work plan. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received.

[5] Actual unused samples and extracts are not considered a reportable item, and their return to NYSDEC, if requested, is not billable. Unused portions or samples and extracts are considered to be a deliverable only when their return is requested in writing by NYSDEC. As specified in the Protocol, and unless otherwise instructed by the BWAM, the Laboratory shall dispose of unused sample/extract volume and used sample bottles/containers no earlier than ninety (90) days following submission of analytical data in the form of the Sample Data Package. Until these ninety days have expired, NYSDEC samples and sample extracts are the exclusive property of NYSDEC and cannot be experimented upon, disposed of, or relinquished to third parties without written permission from NYSDEC.

[6] If requested at the time of sample scheduling the contractor shall provide preliminary results, consisting of Form I and Form I TIC analytical results, by fraction, for field and quality control (QC) sample analysis via telefacsimile (fax) or electronic mail, and Form X for Pesticides and Form X for Aroclors. The Contractor will be notified of the fax number or email address at the time of the sample scheduling. Chain of Custody (COC) Records and SDG Cover Sheets shall be submitted with the Preliminary Results. The contractor shall contact the Project Officer after confirming transmission. The Contractor shall document all communication in a telephone contact log.

[7] If a sample requiring Preliminary Results arrives before 5 p.m. (Contractor's local time), the Preliminary Results are due within the required turnaround time. If a sample requiring Preliminary Results is received after 5 p.m., the Preliminary Results are due within the required turnaround time beginning at 8 a.m. the following day.

Distribution Addresses:

1. Quality Standards and Analytical Management Section
The Bureau of Watershed Assessment and Management
Division of Water
NYS Department of Environmental Conservation
625 Broadway, 4th Floor
Albany, New York 12233-3502

2. NYSDEC Sample Submitters

3. NYSDEC Project Officers

The BWAM acting on behalf of the Project Officer will provide the Laboratory with the list of addressees for the nine NYSDEC Regions. BWAM will provide the Laboratory with updated Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

***NOTE**: Specific recipient names and addresses are subject to change during the term of the contract. The Bureau of Watershed Assessment and Management (BWAM) will notify the Laboratory in writing of such changes when they occur.*

PART II -- REPORT DESCRIPTIONS AND ORDER OF DATA DELIVERABLES

1.0 Overview

The Laboratory shall provide reports and other deliverables as specified by the schedule in Part I of this Exhibit. The required content and assembly of each deliverable is described in Part II of this Exhibit.

Descriptions of the requirements for each deliverable "Item" listed in the chart in Part I, are specified in sections A-G of this Part. Items submitted concurrently MUST BE arranged in the order listed. Additionally, the components of each item MUST BE arranged in the order presented in this Section when the item is submitted.

Examples of specific data deliverables not included herein may be obtained by submitting a written request to The Bureau of Watershed Assessment and Management clearly stating the information requested and signed and dated by the Laboratory Manager.

1.1 All deliverables MUST BE as follows:

- Legible, as specified in Section V,
- Clearly labeled and completed in accordance with instructions in this Exhibit,
- Arranged in the order specified in this Exhibit, and
- Paginated sequentially according to instructions in this Exhibit, starting from the SDG Narrative.
- Information reported on the CLP Forms or CLP-type Forms listed in this exhibit must either be typewritten or computer-generated. Handwritten corrections to the information on the CLP Forms and CLP-type Forms are not permitted. Notes or handwritten corrections on the hardcopy instrument output files must be legible, signed, and dated. Raw data consisting of handwritten worksheets should be completed in a legible fashion.
- Extraneous information should be kept to a minimum. Raw data pages, which contain no information pertaining to NYSDEC samples or QC relating to NYSDEC samples, should be excluded from the sample data package.
- Do not include redundant copies of the same supporting data in the data package. For example, if different sets of raw data reference the same standard prep log pages, include only one copy of the pages and link to it from the appropriate sections.

1.2 The contractor shall use NYSDEC Case Numbers, SDG Numbers, and NYSDEC Sample Numbers to identify samples received under this

contract, both verbally and in reports and correspondence. The Contract number shall be specified in all correspondence.

1.3 Sections III and IV of this Exhibit contain instruction for the required data reporting forms in CLP-specified formats, along with examples and templates for certain NYSDEC specific forms. Section V of this Exhibit contains the specifications for the .PDF file created for the data package. The format for electronic data deliverables (EDD) or other database compatible files are contained in Exhibit H.

1.4 In subsequent Sections of this document the words "copy" and "copies" are used when describing elements used to construct the Sample Data Package and Sample Data Summary Package. The terms "copy" and "copies", when used in this context, refer to Adobe .PDF pages produced from the original documents and included in the main .PDF file for the Package.

1.5 In all instances where a method detection limit (MDL), practical quantitation limit (PQL), or other detection limit (DL) must be reported along with the sample result, the appropriate limit should be adjusted based on the individual sample amount (mass or volume), dilution, and any additional factors they influence the limit being reported. This is referred to as the "sample specific detection limit". A sample specific detection limit should be reported along with all NYSDEC sample results, for all NYSDEC requested analytes to which a MDL, PQL, or DL applies. The only instance where the Laboratory may omit reporting of the sample specific detection limit is when a positive result is being reported for a specific analyte and the CLP/ASP Form I being used does not allow space for reporting of both a positive result and the sample specific detection limit.

1.6 Where applicable, the Laboratory shall include examples of the calculations used to arrive at the reported results. These sample calculations shall use the raw numbers from an actual sample (non-U flagged) in the data package, and show how the final reported result was arrived at for a randomly selected analyte. One sample calculation shall be included for each method used for reporting data in the SDG.

2.0 Resubmission of Data

2.1 If submitted documentation does not conform to the above criteria Section 1.1-1.4), the Laboratory will be required to resubmit such documentation with the deficiencies corrected within 6 business days, at no additional cost to NYSDEC.

2.2 Whenever the Laboratory is required to submit or resubmit data as a result of an on-site laboratory evaluation or through a Bureau of Watershed Assessment and Management (BWAM) action, or through a Project Officer's request, the data must be clearly marked as "ADDITIONAL DATA" and distributed to the specified data recipients. A cover letter must be included which describes what data is being delivered, to which NYSDEC sample(s) it pertains, and who requested the data.

2.3 Whenever the Contractor is required to submit or resubmit data as a result of Contact Compliance Screening (CCS) review by BWAM, the data shall be sent to the two contractual data recipients (BWAM and Region) and to NYSDEC's designated recipient when a written request for Sample Data Package has been made. In all instances the Contractor shall include a color-coded cover sheet (Laboratory Response to Results of Contract Compliance Screening) provided by BWAM. Electronic deliverable should be submitted or resubmitted to BWAM and the Region.

A. – Standard Operating Procedures

See Exhibits E and F for requirements

B. – Quality Assurance Management Plan

See Exhibits E and F for requirements

C. – Weekly Sample Receipt Summary

1.0 Weekly Sample Receipt Summaries shall be submitted by the Wednesday following the calender week (Sunday through Saturday) for which samples are submitted. This information must be transmitted electronically (emailed) as a Microsoft Excel compatible file. NYSDEC will provide the Excel file structure and all appropriate fields in the Excel file should be completed prior to submission.

1.1 The Weekly Sample Receipt Summary shall contain the following items:

- Lab name
- Contract number
- NYSDEC Case #
- NYSDEC SDG #
- NYSDEC Sample ID #
- Lab ID #
- Name of NYSDEC Sample Submitter
- Code numbers for requested analyses from Contract Laboratory Sample Information Sheet
- Sample Analysis Price – full sample price from contract for each sample # reported.
- List of NYSDEC sample numbers of all samples in the SDG, identifying the first and last samples received, and their dates of receipt.

***Note**: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the lowest sample number (considering both alpha and numeric designations); the "last" sample received would be the highest sample number (considering both alpha and numeric designations).*

1.2 The NYSDEC SDG# is found on the Contract Laboratory Sample Information Sheet. The SDG number is also reported on all data reporting forms.

D. – Sample Data Summary Package

As specified in the Delivery Schedule, one Sample Data Summary Package CD-ROM each shall be delivered to the project officer and the sample collector concurrently with delivery of other required sample data. The Sample Data Summary Package consists of Adobe .PDF copies of specified items from the Sample Data Package. These items are listed below and described in detail under part E, Sample Data Package.

The Sample Data Summary Package shall be ordered as follows and shall be submitted separately either as a separate .PDF file or clearly separated by a bookmark in the Sample Data Package .PDF directly preceding the Sample Data Package. Sample data forms shall be arranged by fraction, in increasing NYSDEC sample number order, considering both letters and numbers. E400 is a lower sample number than RH100, as E precedes R in the alphabet.

Specifications for the book marking of electronic (.PDF) data packages are given in Section V of this Exhibit. Sections that must be bookmarked are annotated with **"<B-*X*>**", where *X* is the numeric level of the bookmark required for the given Section or subsection. For further information on bookmarking requirements see Part V, Section 1.3.6.

The Sample Data Summary Package shall contain all data for all samples within one Sample Delivery Group of the Case as follows:

1. NYSDEC Data Package Summary Forms **<B-1>**

2. SDG Narrative **<B-1>**

3. By fraction (VOA, SV, PEST, ARO, IN, WC) and by sample within each fraction – tabulated target compound results (FORM I-*XXXX*) and tentatively identified compounds (FORM I-*XXXX*-TIC) (VOA and BNA only). (**<B-1>** for the "Sample Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the results for each separate fraction and/or analysis method)

   ***Note:** "XXXX" represents the code for the appropriate organic data reporting form.*

4. By fraction (VOA, SV, PEST, and ARO) – surrogate spike analysis results (FORM II-*XXXX*) by matrix (water and/or soil) and for soil, by concentration (low or medium). (**<B-1>** for the "Surrogate Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the surrogate results for each separate fraction and/or analysis method)

5. By fraction (VOA, SV, PEST, and ARO) – matrix spike/matrix spike duplicate/matrix spike blank results (FORM III-*XXXX*) – as required by method. (**<B-1>** for the "MS/MSD Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the MS/MSD results for each separate fraction and/or analysis method)

6. By fraction (VOA, SV, PEST, and ARO) – QC Check Sample/Standard Recovery Summary – If required by method. (**<B-1>** for the "Check Sample/Standard Recovery" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the check standard results for each separate fraction and/or analysis method)

7. By fraction (IN and WC only) – duplicate sample results (FORM VI-IN). (**<B-1>** for the "Duplicate Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the duplicate results for each separate fraction and/or analysis method)

8. By fraction (IN and WC only) – spike sample results (FORM V-IN). (**<B-1>** for the "Spike Sample Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the spike results for each separate fraction and/or analysis method)

9. By fraction (VOA, SV, PEST, ARO, IN, WC) – blank data (FORM IV-*XXXX* (for organics) and Form III-IN) and tabulated results (FORM I-*XXXX* (for organics) and FORM I-IN) including tentatively identified compounds (FORM I-*XXXX*-TIC)(VOA and BNA only). (**<B-1>** for the "Blank Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the blank results for each separate fraction and/or analysis method)

10. By fraction (VOA and SV only) – internal standard area data (FORM VIII-*XXXX*). (**<B-1>** for the "Internal Standard Recovery" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the internal standard recovery for each separate fraction and/or analysis method)

E. – Sample Data Package

The Sample Data Package is divided into the eight major units described below. The last six units are each specific to an analytical fraction (volatiles, semivolatiles, pesticides/Aroclors, GC organics, inorganics, and conventional wet-chemistry). If the analysis of a fraction is not required, then that fraction-specific unit is not required as a deliverable.

The Sample Data Package shall include data for analyses of all samples in one Sample Delivery Group, including field samples, re-analyses, blanks, duplicates, control spikes, matrix spikes, matrix spike duplicates, and matrix spike blanks. In addition, the package will also include the results of Method Detection Limit studies and reports establishing interelement correction factors for ICP-AES.

All data produced in support of Superfund investigation/remediation as identified by checked boxes under the Contract Laboratory Section of the Contract Laboratory Sample Information Sheet (CLSIS) (See Exhibit A) shall be reported as specified for the Superfund Category/CLP (Section 1.0 below). All data generated in support of the

SPDES program as identified by a CASE # beginning with the letter "E" shall be reported using ASP Category B (Section 3.0 below). All other samples shall be reported using either ASP Category A or ASP Category B described in Section 2.0 and 3.0 below. The specific reporting level to be used shall be specified by the CLSIS, unless otherwise specified in a project work plan.

The Laboratory shall retain a CD-ROM/.PDF copy of the Sample Data Package for 3 years after final acceptance of data. See Section V for a detailed explanation of these requirements. After this time, the Laboratory may dispose of/delete the package.

Specifications for the book marking of electronic (.PDF) data packages are given in Section V of this Exhibit. Sections that must be bookmarked are annotated with **"<B-*X*>**", where *X* is the numeric level of the bookmark required for the given Section.

1.0 Superfund Category/CLP

1.1 Cover Documentation **<B-1>**

Cover Page for the Data Package shall include: laboratory name; laboratory code; contract number; Case number; SDG number; and NYSDEC sample numbers in alphanumeric order.

1.2 SDG Narrative **<B-1>**

1.2.1 This document shall be clearly labeled "SDG Narrative" and shall contain: Laboratory name; Case number; Sample Delivery Group number (SDG); NYSDEC sample numbers in the SDG, differentiating between initial analyses and re-analyses; Contract number; and detailed documentation of any quality control, sample, shipment and/or analytical problems encountered in processing the samples reported in the data package. For soil samples collected and pre-weighed in the field the laboratory shall document all discrepancies between sample weights determined in the field and in the laboratory in the SDG Narrative. A statement on the use of background and interelement corrections performed for the samples should be included for inorganic analysis, if applicable.

1.2.2 The Laboratory shall document, in the SDG Narrative, the alternative technique used to determine cooler temperature if a temperature indicator bottle is not present in the cooler. The Laboratory shall also provide, in the SDG Narrative, sufficient information, including equations or curves (at least on equation or curve per method), to allow the recalculation of sample results from raw instrument output. The Laboratory shall also include a discussion of any performance-based modifications performed on the Protocol requirements or on published methods. If modifications are reoccurring, the laboratory may provide separate documentation of the modifications and reference such modifications in the SDG Narrative. Additionally, the Laboratory shall also identify and explain any differences that exist between the Form Is and the supporting documentation provided in the

data package and those previously provided as preliminary results.

1.2.3 The Contractor shall also provide, in the SDG Narrative or as attachments referenced in the SDG narrative, sufficient information, including copies of equations and definitions of variables (at least one equation per method), to allow the recalculation of sample results from raw instrument output.

1.2.4 All Gas Chromatography (GC) columns used for analysis should be documented in the SDG Narrative, by fraction. List the GC column identification—brand name, the internal diameter (in millimeters), and the length (in meters), packing/coating material, and film thickness. The trap used for volatile analysis shall be described here. List trap name, when denoted by the manufacturer, its composition (packing material/brand name, amount of packing material, in length). The Laboratory shall include any technical and administrative problems encountered, the corrective action taken, the resolution, and an explanation for all flagged edits (e.g. manual edits) on quantitation lists. The Laboratory shall document in the SDG Narrative all instances of manual integration.

1.2.5 Whenever data from sample re-analysis are submitted, the Laboratory shall state in the SDG Narrative for each re-analysis, whether it considers the re-analysis to be billable, and if so, why.

1.2.6 The Laboratory shall list the pH determined for each water sample submitted for volatile analysis. This information may appear as a simple list or table in the SDG Narrative. The purpose of this pH determination is to ensure that all water volatiles samples were acidified in the field. No pH adjustment is to be performed by the Laboratory on water samples for volatiles analysis. The SDG Narrative shall conclude with the following statement, verbatim: *"I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this Sample Data Package and in the electronic data deliverables has been authorized by the Laboratory Manager or his/her designee, as verified by the following signature."* This statement shall be directly followed by signature of the Laboratory Manager or his designee with a typed line below it containing the signer's name and title, and the date of signature.

1.3 Sample Log-In Sheet [FORM DC-1] **<B-1>**

NOTE: *Example copies of the DC-1 form can be found in CLP Exhibit B. Use the DC-1 Form in OLM04.2 for organic samples and the DC-1 Form in ILM05.3 for inorganics/conventional samples.*

In addition to the DC-1 Form, the contractor must include a listing showing NYSDEC sample numbers, in alphanumeric order, cross-referenced with laboratory Sample ID numbers.

1.4 Contract Lab Sample Information Sheets **<B-1>**

A copy of the Contract Lab Sample Information Sheets (CLSIS) for all of the samples in the SDG. The CLSIS shall be arranged in increasing NYSDEC sample number order, considering both letters and numbering in ordering samples.

1.5 Chain-of-Custody Forms **<B-1>**

Copies of both the external and internal chain-of-custody sheets for all samples within the SDG.

1.6 Superfund-CLP Volatiles Data **<B-1>**

1.6.1 QC Summary **<B-2>**

1.6.1.1 System Monitoring Compound or Deuterated Monitoring Compound Recovery Reports (FORM II VOA-1, VOA-2, VOA-3, VOA-4, VOA-SIM, VOA-SIM1, VOA-SIM2).

1.6.1.2 Matrix Spike/Matrix Spike Duplicate/Matrix Spike Blank Recovery Reports (FORM III VOA-1, VOA-2, VOA-SIM) – Provided when an MS/MSD analysis is requested by NYSDEC.

1.6.1.3 Method Blank Summary (FORM IV VOA, VOS-SIM) – If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank, by instrument.

1.6.1.4 GC/MS Instrument Performance Check (FORM V VOA) – If more than a single form is necessary, the forms must be arranged in chronological order, by instrument.

***Note**: This form is not required for the optical analysis when submitting data using the Selected Ion Monitoring (SIM) technique.*

1.6.1.5 Internal Standard Area and RT Summary (FORM VIII VOA, VOA-SIM) – If more than a single form is necessary, the forms must be arranged in chronological order, by instrument.

1.6.2 Volatiles Sample Data (**<B-2>** to mark Section heading, **<B-3>** to mark the beginning of each data "packet")

Sample data shall be arranged in packets with the Organic Analysis Data Sheet (FORM I VOA-1, VOA-2, including FORM I VOA-TIC), followed by the raw data for volatile samples. The sample data shall be placed in order of increasing NYSDEC sample number, considering both letters and numbers. Volatile sample data for SIM analysis must be arranged

together with the rest of the SIM Volatiles data at the end of the sub-Section.

1.6.2.1 Target Compound Results – Volatile Organics Analysis Data Sheet (FORM I VOA-1, VOA-2) – Tabulated results (identification and quantitation) of the specified Superfund-CLP target compounds (Exhibit C – Volatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 1.2). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.6.2.2 Target Compound Results – Volatile Organics Analysis Data Sheet (FORM I VOA-1, VOA-2) – Tabulated results (identification and quantitation) of the specified Superfund-CLP target compounds (Exhibit C – Volatiles) shall be included. The validation and release of the results are authorized by a specific, signed statement in the SDG Narrative (see Section 1.2). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.6.2.3 Tentatively Identified Compounds (FORM I VOA-TIC) – FORM I VOA-TIC is the tabulated list of the highest probable match for up to 10 organic compounds not system monitoring compounds and are not target compounds, system monitoring compounds, internal standard compounds, or unsubstituted alkanes, or any other compound not listed in Exhibit C – Volatiles. It including the CAS (Chemical Abstracts Registry) number, tentative identification and estimated concentrations. For estimating concentration, assume a response factor of 1, and estimate the concentration by comparison of the compound peak height or total area count to the peak height or total area count of the nearest internal standard free of interferences on the reconstructed ion chromatogram. This form must be included even if no compounds are found. If this occurs, enter a "0" in the field for "Number found" on the form.

Note: *The Laboratory must be consistent, i.e., use peak height for all comparisons or use total area count for all comparisons.*

1.6.2.4 Reconstructed Total Ion Chromatograms (RIC) (for each sample including dilutions and reanalyzes) – RICs must be normalized to the largest non-solvent component and contain the following header information:

- NYSDEC sample number;
- Date and time of analysis;
- GC/MS instrument ID;
- Lab file ID;
- Analyst ID.

Note: *Each Selected Ion Current Profile (SICP) for samples taken through the optional analysis using the SIM technique shall be labeled as in this Section.*

1.6.2.4.1 Internal standard and system monitoring compounds should be labeled with the names of compounds, either directly out from the peak, or are to be included on a printout of retention times when the retention times are directly located over the peak. Labeling of the compounds is not required and should not detract from the legibility of the required labels.

1.6.2.4.2 If automated system procedures are used for preliminary identification and/or quantification of the Superfund Target Compound List (Superfund-TCL) compounds, the complete data system report must be included in all Sample Data Packages, in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories that do not use the automated data system procedures, a laboratory "raw data sheet", which contains the following information, must be included in the sample data package in addition to the chromatogram.

- NYSDEC sample number;
- Date and time of analysis;
- RT or scan number of identified target compounds;
- Ion used for quantitation with measured area;
- Copy of area table from data system;
- On column concentration/amount, including units;
- GC/MS instrument ID;

- Lab file ID;
- Analyst ID.

1.6.2.4.3 In all instances where the data system report has been edited, or where manual integration or manual quantitation has been performed, the GC/MS operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integrated area with the letter "m" on the quantitation report. In addition, a hardcopy printout of the Extracted Ion Current Profile (EICP) of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C – Volatiles, internal standards, and system monitoring compounds.

1.6.2.5 Other required Information. For each sample, by each compound identified, the following shall be included in the data package:

1.6.2.5.1 Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C – Volatiles that are identified in the sample and corresponding background-subtracted TCL standard mass spectra. Spectra must be labeled with NYSDEC sample number, lab file ID, date, and time of analysis, and GC/MS instrument ID. Compound names must be clearly marked on all spectra.

1.6.2.5.2 Copies of mass spectra of organic compounds not listed in Exhibit C (Superfund-TCL) (Tentatively Identified Compounds), with associated best-match spectra (the three best matches), as labeled in 1.6.2.4 above.

1.6.3 Standards Data **<B-2>**

1.6.3.1 Initial Calibration Data (FORM VI VOA-1, VOA-2, VOA-3, VOA-SIM) – shall be included in order by instrument, if more than one instrument used. **<B-3>**

1.6.3.1.1 Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five-point) calibration, as labeled in 1.6.2.4 above. Spectra are not required.

1.6.3.1.2 All initial calibration data that pertain to samples in the data package must be included, regardless of when it was performed and for which Case. When more than one initial calibration is performed, the data must be put in chronological order, by instrument.

1.6.3.1.3 Labels for standards shall be descriptive of the concentrations of the non-ketone (majority) analytes in µg/L.

1.6.3.1.4 EICPs displaying each manual integration.

1.6.3.2 Continuing Calibration (FORM VII VOA-1, VOA-2, VOA-3, VOA-SIM) – shall be included in order by instrument, if more than one instrument used. **<B-3>**

1.6.3.2.1 Volatile standard(s) reconstructed ion chromatograms and quantitation reports for all continuing (12-hour) calibration verifications, as labeled in 1.6.2.4. Spectra are not required.

1.6.3.2.2 When more than one Continuing Calibration Verification is performed, forms must be in chronological order, by instrument.

1.6.3.2.3 EICPs displaying each manual integration.

1.6.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS Operator shall identify such edits or manual procedures by initializing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integration area with the letter "m" on the quantitation report. In addition a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C – Volatiles, internal standards, and system monitoring compounds.

1.6.4 Volatiles Raw QC Data **<B-2>**

1.6.4.1 4-Bromofluorobenzene (BFB) shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized. **<B-3>**

1.6.4.1.1 Bar graph spectrum, as labeled in 1.6.2.4.

1.6.4.1.2 Mass listing, as labeled in 1.6.2.4.

1.6.4.1.3 Reconstructed total ion chromatogram (RIC), labeled as in 1.6.2.4.

1.6.4.2 Blank Data shall be arranged by type of blank (method, storage, instrument) and shall be in chronological order, by instrument. **<B-3>**

***Note**: This order is different from that used for sample data (Section 1.6.2).*

1.6.4.2.1 Tabulated results (FORM I VOA-1, VOA-2, VOA-SIM).

1.6.4.2.2 Tentatively Identified Compounds (FORM I-TIC) – even if none are found.

1.6.4.2.3 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), as labeled as in 1.6.2.4.

1.6.4.2.4 Target compound spectra with laboratory-generated standard, labeled as in 1.6.2.4. Data systems that are incapable of dual display shall provide spectra in the following order:

- Raw target compound spectra;
- Enhanced or background-subtracted spectra;
- Laboratory generated standard spectra.

1.6.4.2.5 GC/MS library search spectra for Tentatively Identified Compounds (TIC), labeled as in 1.6.2.4.

1.6.4.2.6 Quantitation/calculation of TIC concentrations.

1.6.4.3 Matrix Spike Blank Data **<B-3>**

1.6.4.3.1 Tabulated results (FORM I VOA-1, VOA-2, VOA-SIM) of all target compounds. Form I VOA-TIC is <u>not</u> required.

1.6.4.3.2 Reconstructed ion chromatogram(s) and quantitation report(s), as labeled in 1.6.2.4. Spectra are <u>not</u> required.

1.6.4.4 Matrix Spike Data **<B-3>**

1.6.4.4.1 Tabulated results (FORM I VOA-1, VOA-2) of all target compounds. FORM I VOA-TIC is <u>not</u> required.

1.6.4.4.2 Reconstructed ion chromatogram(s) and quantitation report(s), as labeled in 1.6.2.4. Spectra are not required.

1.6.4.5 Matrix Spike Duplicate Data **<B-3>**

1.6.4.5.1 Tabulated results (FORM I VOA) of all target compounds. FORM I VOA-TIC is not required.

1.6.4.5.2 Reconstructed ion chromatogram(s) and quantitation report(s), as labeled in 1.6.2.4. Spectra are not required.

1.6.5 Copy of Calculations **<B-2>**

The Laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the quantitation report. If manipulations are performed by a software package, a copy of the formula used must be supplied, as well as, values for all terms in the formula.

Note: *All correction factors and equations utilized must be indicated on the work sheet.*

1.6.6 Copy of Extraction Logs **<B-2>**

These logs must be legible and include: (1) date, (2) sample weights and volumes, (3) sufficient information to unequivocally identify which QC samples (i.e. matrix spike, matrix spike duplicate, matrix spike blank) correspond to each batch extracted, (4) comments describing any significant sample changes or reactions which occur during preparation, and (5) final volumes and vial identification numbers.

1.7 Semivolatiles Data **<B-1>**

1.7.1 Semivolatiles QC Summary **<B-2>**

1.7.1.1 System Monitoring Compound Percent Recovery Summary (FORM II SV-1, SV-2, SV-3, SV-4, SV-SIM).

1.7.1.2 Matrix Spike/Matrix Spike Duplicate Summary (FORM III SV-1, SV-2, SV-SIM) - Provided when an MS/MSD analysis is requested by NYS DEC.

1.7.1.3 Method Blank Summary (FORM IV SV, SV-SIM) – If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.

1.7.1.4 GC/MS Instrument Performance Check (FORM V SV) – If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

Note: *This form is not required when submitting data for the analysis of Polynuclear Aromatic Hydrocarbons (PAHs)/phenols using the SIM technique.*

1.7.1.5 Internal Standard Area and RT Summary (FORM VIII SV-1, SV-2) – If more than a single form is necessary, the forms shall be arranged in chronological order, by instrument.

1.7.1.6 Instrument Detection Limits.

1.7.2 Semivolatile Sample Data (**<B-2>** to mark Section heading, **<B-3>** to mark the beginning of each data "packet")

Sample data shall be arranged in packets with the Semivolatile Organics Analysis Data Sheet (FORM I SV-1, SV-2, including FORM I SV-TIC), followed by the raw data for semivolatile samples. These sample packets should then be placed in increasing DEC sample number, considering both letters and numbers in ordering samples.

1.7.2.1 Target Compound Results, Semivolatiles Organics Analysis Data Sheet (FORM I SV-1, SV-2) – Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C – CLP Semivolatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 1.2). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.7.2.2 Semivolatile Tentatively Identified Compounds (FORM I SV-TIC) – Form I SV-TIC is the tabulated list of the highest probable match for up to 20 organic compounds that are not target compounds, system monitoring compound, internal standard compounds, and are not listed in Exhibit C – CLP Volatiles and Semivolatiles. It includes the CAS number (if applicable), tentative identification, and estimated concentration. For estimating concentration, assume a response factor of 1, and estimate the concentration by comparison of the compound peak height or total area count to the peak height or total area count of the nearest internal standard free of interferences on the reconstructed ion chromatogram. This form must be included even if no compounds are found. If this occurs, enter a "0" in the field for "Number found" on the form.

Note: *This form is not required when submitting data for the optional analysis of PAHs/phenols using the SIM technique.*

Note*: The Laboratory must be consistent, i.e., use peak height for all comparisons or use total area count for all comparisons.*

1.7.2.3 PAHs/Phenols Analysis Data Sheet (FORM I SV-SIM) – This data form shall be submitted upon the NYS DEC's request for optional analysis of PAHs/phenols using the SIM technique. The specific target PAHs/phenols listed in Exhibit C – CLP Semivolatiles shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 1.2). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.7.2.4 Reconstructed Total Ion Chromatograms (RICs) (for each sample, including dilutions and reanalyzes). RICs must be normalized to the largest non-solvent component, and must contain the following header information:

- NYSDEC sample number;
- Date and time of analysis;
- GC/MS instrument ID;
- Lab file ID; and
- Analyst ID.

1.7.2.4.1 Internal standards and system monitoring compounds are to be labeled on RICs or SICPs with the names of compounds, either directly out from the peak, or are to be included on a printout of retention times if the retention times are printed directly over the peak.

1.7.2.4.2 If automated data system procedures are used for preliminary identification and/or quantification of the target compound, the complete data system report shall be included in all Sample Data Packages, in addition to the reconstructed ion chromatogram or SICP for optional PAHs/phenols analysis. The complete data system report shall include all of the information listed below. For laboratories that do not use the automated data system procedures, a laboratory "raw data sheet," containing the following information, shall be included in the Sample Data Package, in addition to the chromatogram.

- NYSDEC sample number

- Date and time of analysis
- RT or scan number of identified Superfund-TCL compounds
- Ion used for quantitation with measured area
- Copy of area table from data system
- GC/MS instrument ID
- Lab file ID

1.7.2.4.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS operator shall also mark each integrated area with the letter "m" on the quantitation report. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C – CLP Semivolatiles, internal standards, and system monitoring compounds.

1.7.2.5 Other Required Information – For each sample, by each compound identified, the following shall be included in the data package:

1.7.2.5.1 Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C – CLP Semivolatiles that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. This includes PAH/phenol target compounds that are identified during the optional analysis using the SIM technique. Spectra shall be labeled with NYS DEC sample number, laboratory file ID, date, and time of analysis, and GC/MS instrument ID. Compound names must be clearly marked on all spectra.

1.7.2.5.2 Copies of mass spectra of non-system monitoring/non-internal standard organic compounds not listed in Exhibit C – CLP Semivolatiles with associated best-match spectra (maximum of three best matches). This

includes the mass spectra for tentatively identified alkanes. Spectra shall be labeled with NYS DEC Sample Number, laboratory file ID, date and time of analysis, and GC/MS instrument ID. Compound names shall be clearly marked on all spectra.

1.7.3 Semivolatiles Standards Data **<B-2>**

1.7.3.1 Initial Calibration Data (FORM VI SV-1, SV-2, SV-3) or FORM VI SV-SIM (when optional analysis of PAHs/phenols is performed) shall be included in order by instrument, if more than one instrument used. **<B-3>**

1.7.3.1.1 Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for the initial (five-point) calibration, labeled in 1.7.2.4. Spectra are not required.

1.7.3.1.2 When optional analysis of PAHs/phenols is requested, then SICPs and quantitation reports for the initial calibration standards (five-point), labeled as in Section 1.7.2.4, shall be submitted. Spectra are not required.

1.7.3.1.3 All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed and for which SDG. When more than one initial calibration is performed, the data must be put in chronological order, by instrument.

1.7.3.1.4 Labels for standards shall reflect the concentrations of the majority of the analytes in µg/L.

1.7.3.1.5 EICPs displaying each manual integration.

1.7.3.2 Continuing Calibration Verification Data (FORM VII SV-1, SV-2, SV-3) or FORM VII SV-SIM (when optional analysis of PAHs/phenols is performed) shall be included in order by instrument, if more than one instrument used. **<B-3>**

1.7.3.2.1 Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for all opening, closing, and continuing calibrations verifications, as labeled in Section 1.7.2.4. Spectra are not required.

1.7.3.2.2 When optional analysis of PAHs/phenols is requested, then SICPs and quantitation reports

for all opening, closing, and CCVs, labeled as in Section 1.7.2.4. Spectra are not required.

1.7.3.2.3 When more than one continuing calibration is performed, forms must be in chronological order, by instrument.

1.7.3.2.4 EICPs displaying each manual integration.

1.7.3.3 In all instances where the data system report has been edited, or where the manual integration or quantitation has been performed, the GC/MS Operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integration area with the letter "m" on the quantitation report. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C – CLP Semivolatiles, internal standards, and system monitoring compounds.

1.7.4 Semivolatiles Raw Quality Control (QC) Data **<B-2>**

1.7.4.1 Decafluorotriphenylphosphine (DFTPP) data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized. **<B-3>**

1.7.4.1.1 Bar graph spectrum, as labeled in 1.7.2.4.

1.7.4.1.2 Mass listing, as labeled in 1.7.2.4.

1.7.4.1.3 Reconstructed total ion chromatogram (RIC), labeled as in 1.7.2.4.

1.7.4.2 Blank Data shall be in chronological order by extraction date. **<B-3>**

Note: *This order is different from that used for samples.*

1.7.4.2.1 Tabulated results (FORM I SV-1, SV-2, SV-SIM).

1.7.4.2.2 Tentatively Identified Compounds (FORM I SV-TIC) – even if none found.

1.7.4.2.3 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), as labeled in 1.7.2.4.

1.7.4.2.4 Target compound spectra with laboratory-generated standard, as labeled in 1.7.2.4. Data

systems that are incapable of dual display shall provide spectra in the following order:

- Raw target compound spectra;
- Enhanced or background-subtracted spectra;
- Laboratory-generated standard spectra.

1.7.4.2.5 GC/MS library search spectra for Tentatively Identified Compounds (TICs), as labeled in 1.7.2.4.

1.7.4.2.6 Quantitation/Calculation of TIC concentrations.

1.7.4.3 Semivolatiles Matrix Spike Blank Data **<B-3>**

1.7.4.3.1 Tabulated results (FORM I SV) of all target compounds. Form I SV-TIC not required.

1.7.4.3.2 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), as labeled in 1.7.2.4. Spectra are required.

1.7.4.4 Semivolatiles Matrix Spike Duplicate Data **<B-3>**

1.7.4.4.1 Tabulated results (FORM I SV-1, SV-2) of all target compounds. FORM I SV-TIC is not required.

1.7.4.4.2 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), as labeled in 1.7.2.4. Spectra are not required.

1.7.4.5 Semivolatile Gel Permeation Chromatography (GPC) Data – The two most recent Ultra Violet (UV) traces of the (GPC) calibration solution, and the reconstructed ion chromatogram and data system reports for the GPC blank shall be arranged in chronological order by GPC for the GPC calibration. **<B-3>**

1.7.4.5.1 Traces must be labeled with GPC column identifier, date of calibration, and with compound names labeled either directly out from the peak, or on a printout of retention times, if retention times are printed over the peak.

1.7.4.5.2 Reconstructed ion chromatogram and data system report(s) labeled as specified in Section 1.7.2.4 for the GPC blank analysis.

1.7.4.5.3 Reconstructed ion chromatogram and data system report(s) for all standards used to quantify compounds in the GPC blank, labeled, as specified in section 1.7.2.4.

1.7.5 Copy of Calculations **<B-2>**

The Laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the quantitation report. If manipulations are performed by a software package, a copy of the formula used must be supplied as well as values for all terms in the formula.

Note: *All correction factors and equations utilized must be indicated on the work sheet.*

1.7.6 Copy of Extraction Logs **<B-2>**

These logs must be legible and include: (1) date, (2) sample weights and volumes, (3) sufficient information to unequivocally identify which QC samples (i.e. matrix spike, matrix spike duplicate, matrix spike blank) correspond to each batch extracted, (4) comments describing any significant sample changes or reactions which occur during preparation, and (5) final volumes and vial identification numbers.

1.8 Pesticide Data **<B-1>**

1.8.1 Pesticide QC Summary **<B-2>**

1.8.1.1 Surrogate Recovery (FORM II PEST-1, PEST-2)

1.8.1.2 Matrix Spike/Matrix Spike Duplicate/Matrix Spike Blank Recovery (FORM III PEST-1, PEST-2): MS/MSD is required for the Pesticide fraction of an SDG, unless otherwise specified by the NYS DEC.

1.8.1.3 Laboratory Control Sample Recovery (FORM III PEST-1, PEST-2).

1.8.1.4 Method Blank Summary (FORM IV PEST): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank.

1.8.2 Pesticide Sample Data (**<B-2>** to mark Section heading, **<B-3>** to mark the beginning of each data "packet")

Sample data shall be arranged in packets with the Pesticide Organic Analysis Data Sheet (FORM I PEST), followed by the raw data for pesticide samples. These sample packets should then be

placed in increasing NYSDEC sample number order, considering both letters and numbers in ordering samples.

1.8.2.1 Target Compound Results, Pesticide Organics Analysis Data Sheet (FORM I PEST). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C – CLP Pesticides) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (see Section 1.2). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.8.2.2 Copies of Pesticide Chromatograms. Positively identified compounds shall be labeled with the names of compounds, either directly out from the peak on the chromatogram, or on a printout of RTs on the data system printout if RTs are printed over the peak on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D, and shall be labeled with the following information:

- NYSDEC sample number;
- Volume injected (µL);
- Date and time of injection;
- On column concentration/ amount including units;
- GC column identifier (by stationary phase and internal diameter);
- GC instrument identifier; and
- Scaling factor (label the x and y axes using a numerical scale).

1.8.2.3 Copies of pesticide chromatograms from second GC column shall be included and labeled as in Section 1.8.2.2.

1.8.2.4 Data System Printout. A printout of RT, corresponding peak height or peak area, and on the column amount shall accompany each chromatogram. The printout shall be labeled with the NYS DEC sample number. In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the Gas Chromatograph/Electron Capture Detector (GC/ECD) Operator shall identify all such edits or manual procedures by initialing and dating the

changes made to the report, and shall include the integration time range. The GC/MS Operator shall also mark each integration area with the letter "m" on the quantitation report.

1.8.2.5 All manual worksheets shall be included in the Sample Data Package.

1.8.2.6 Other Required Information. If pesticides are confirmed by GC/MS, the Laboratory shall submit copies of reconstructed ion chromatograms, raw spectra, and background-subtracted mass spectra of target compounds listed in Exhibit C – CLP Pesticides that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. Compound names shall be clearly marked on all spectra. For Toxaphene confirmed by GC/MS, the Laboratory shall submit mass spectra of 3 major peaks from samples and standards.

1.8.3 Pesticides Standards Data **<B-2>**

1.8.3.1 Initial Calibration of Single Component Analytes (FORM VI PEST-1, PEST-2): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.2 Initial Calibration of Multicomponent Analytes (Toxaphene, etc.) (FORM VI PEST-3, PEST-4): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.3 Analyte Resolution Check Summary (FORM VI PEST-5): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.4 Performance Evaluation Mixture (PEM) (FORM VI PEST-6): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.5 Individual Standard Mixture A (FORM VI PEST-7): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.6 Individual Standard Mixture B (FORM VI PEST-8): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.7 Individual Standard Mixture C (FORM VI PEST-9, PEST-10): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.8 Calibration Verification Summary (FORM VII PEST-1): For all mid-point concentrations of Individual Standard Mixtures A and B or C and instrument blanks used for calibration verification, on all GC columns and instruments, in chronological order by GC column and instruments. **<B-3>**

1.8.3.9 Calibration Verification Summary (FORM VII Pest-2, Pest-3): For all mid-point concentrations of Individual Standard Mixtures A and B or C and instrument blanks used for calibration verification, on all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.10 Analytical Sequence (FORM VIII PEST): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.11 Florisil Cartridge Check (FORM IX PEST-1): For all lots of cartridges used to process samples in the SDG, using Individual Standard Mixtures A or C. **<B-3>**

1.8.3.12 GPC Calibration Verification (FORM IX PEST-2): For all GPC columns, in chronological order by calibration verification date. **<B-3>**

1.8.3.13 Identification Summary for Single Component Analytes (FORM X PEST): For all samples with positively identified single component analytes, in order by increasing NYSDEC Sample Number. **<B-3>**

1.8.3.14 Chromatograms and data system printouts are required for all standards including the following: **<B-3>**

- Resolution Check Mixture.
- Performance Evaluation (PE) mixtures, all.
- Individual Standard Mixture A and B, both at five concentrations, for each initial calibration and Individual Standard Mixture B, at five concentrations, for each initial calibration.

Or

- Individual Standard Mixture C, at five concentrations, each initial calibration.
- Toxaphene, at five concentrations, each initial calibration.

- All mid-point concentrations of Individual Standard Mixtures A and B or C used for calibration verification.
- All toxaphene standards analyzed for confirmation.
- All lots of Florisil cartridge check solution
- Pesticide GPC Calibration Check Solution, all calibrations relating to samples in the SDG.
- All multicomponent analyte standards analyzed for confirmation.

1.8.3.15 A printout of RT and corresponding peak height or peak areas shall accompany each chromatogram. The printout shall be labeled with the NYSDEC Sample Number. In addition, all chromatograms shall meet the acceptance criteria in Exhibit D, and shall be labeled with the following: **<B-3>**

- NYSDEC Sample Number for the standard (e.g., INDA10K, INDA20K, etc., See Forms Instructions for details);
- Label all standard peaks for all individual compounds either directly out from the peak or on the printout of retention times if retention times are labeled over the peak;
- Total nanograms injected for each standard. When total nanograms injected appear on the printout, it is not necessary to include them on the chromatogram;
- Date and time of injection;
- GC column identifier (by stationary phase and internal diameter);
- GC instrument identifier; and
- Scaling factor (label the x and y axes using a numerical scale).

Note: *In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/ECD Operator shall identify such edits or manual procedures by initialing and changes made to the report, shall include the integration time range. The GC/MS Operator shall also mark each integrated area with the letter "m" on the quantitation report.*

1.8.4 Pesticides Raw Quality Control (QC) Data **<B-2>**

1.8.4.1 Blank Data shall be arranged by type of blank (method, instrument, sulfur cleanup) and shall be in chronological order by instrument. **<B-3>**

1.8.4.1.1 Tabulated results (FORM I PEST).

1.8.4.1.2 Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, as labeled in 1.8.2.2 and 1.8.2.4 above.

1.8.4.2 Pesticide LCS Data **<B-3>**

1.8.4.2.1 Tabulated results (FORM I PEST) of target compounds for both GC columns.

1.8.4.2.2 Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, as labeled in 1.8.2.2 and 1.8.2.4 above.

1.8.4.3 Pesticides Matrix Spike Data **<B-3>**

1.8.4.3.1 Tabulated results (FORM I PEST) of target compounds for both GC columns.

1.8.4.3.2 Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, as labeled in 1.8.2.2 and 1.8.2.4 above.

1.8.4.4 Pesticides Matrix Spike Duplicate Data **<B-3>**

1.8.4.4.1 Tabulated results (FORM I PEST) of target compounds for both GC columns.

1.8.4.4.2 Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, as labeled in 1.8.2.2 and 1.8.2.4 above.

1.8.4.5 Matrix Spike Blank Data **<B-3>**

1.8.4.5.1 Tabulated results (FORM I-CLP-PEST) of all Superfund-TCL compounds.

1.8.4.5.1.1 Chromatogram(s) and data system printout(s) (GC), as labeled in 1.8.2.2 and 1.8.2.4 above.

1.8.5 Raw Gel Permeation Chromatograph (GPC) Data **<B-2>**

1.8.5.1 GPC Calibration. The UV traces for the GPC calibration solution, chromatograms, and the data system reports for the GPC blank shall be arranged in chronological order for the GPC calibration.

1.8.5.1.1 UV traces labeled with the GPC column identifier, date of calibration, and compound names. Compound names shall be placed directly out from the peak, or on the printout of the RTs when the RTs are printed directly over the peak.

1.8.5.1.2 Chromatograms and data system report(s) labeled as specified in Sections 1.8.2.2 and 1.8.2.4 above.

1.8.5.1.3 Chromatograms and data system report(s) for all standards used to identify compounds in the GPC blank labeled as specified in Section 1.8.3.14 and 1.8.3.15 (i.e., Individual Standard Mixture A, Individual Standard Mixture B, Individual Standard Mixture C, and the Toxaphene standards).

1.8.5.2 GPC Calibration Verification. The Chromatogram and the data system report(s) shall be arranged in chronological order for the GPC calibration check.

1.8.5.2.1 Chromatograms and data system printouts labeled as specified in Sections 1.8.2.2 and 1.8.2.4 for the GPC calibration verification solution analyses.

1.8.5.2.2 Chromatogram and the data system report(s) for the standards used to quantify compounds in the GPC calibration verification solution labeled as specified in Section 1.8.3.14 and 1.8.3.15 (i.e., Individual Standard Mixtures A and B or C from the initial calibration sequence).

1.8.6 Raw Florisil Data **<B-2>**

1.8.6.1 The chromatogram and the data system report(s) shall be arranged in chronological order by Florisil cartridge performance check analysis.

1.8.6.1.1 Chromatograms and data system reports, labeled as specified in Sections 1.8.2.2 and 1.8.2.4 for the Florisil cartridge performance check analysis.

1.8.6.1.2 Chromatograms and data system reports for standard analyses used to quantify compounds in the Florisil cartridge performance check analysis, labeled as specified in Section 1.8.3.14 and 1.8.3.15 (i.e., Individual Standard Mixture A, Individual Standard Mixture B, Individual Standard Mixture C, and the 2,4,5-Trichlorophenol solution).

1.8.7 Copy of Calculations **<B-2>**

The Laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the quantitation report. If manipulations are performed by a software package, a copy of the formula used must be supplied as well as values for all terms in the formula.

Note: *All correction factors and equations utilized must be indicated on the work sheet.*

1.8.8 Copy of Extraction Logs **<B-2>**

These logs must be legible and include: (1) date, (2) sample weights and volumes, (3) sufficient information to unequivocally identify which QC samples (i.e. matrix spike, matrix spike duplicate, matrix spike blank) correspond to each batch extracted, (4) comments describing any significant sample changes or reactions which occur during preparation, and (5) final volumes and vial identification numbers.

1.9 Aroclor Data **<B-1>**

1.9.1 Aroclor QC Summary **<B-2>**

1.9.1.1 Surrogate Recovery (FORM II ARO-1, ARO-2).

1.9.1.2 Matrix Spike/Matrix Spike Duplicate Recovery (FORM III ARO-1, ARO-2): MS/MSD is required for the Aroclor fraction, unless otherwise specified by NYSDEC. One MS/MSD set is required per SDG.

1.9.1.3 LCS Recovery (FORM III ARO-3, ARO-4).

1.9.1.4 Method Blank Summary (FORM IV ARO): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank.

1.9.2 Aroclor Sample Data (**<B-2>** to mark Section heading, **<B-3>** to mark the beginning of each data "packet")

Sample data shall be arranged in packets with Aroclors Organics Analysis Data Sheet (FORM 1 ARO), followed by the raw data for Aroclor samples. These sample packets should then be placed in

order of increasing NYSDEC Sample Number, considering both letters and numbers.

Note: *For a Sample analysis in which "S" flags are reported a FORM I ARO is required for the original analysis (NYSDEC Sample Number = XXXXX) in which the "S" flags are reported, and a FORM I ARO is required for the billable reanalysis (NYSDEC Sample Number = XXXXXRE) of the sample performed after a valid 5-point calibration of the detected Aroclor. An additional FORM I ARO is required for any necessary dilutions (NYSDEC Sample Number = XXXXXDL).*

1.9.2.1 Target Compound Results, Aroclors Organics Analysis Data Sheet (FORM I ARO). Tabulated results (identification and quantification) of the specified target compounds (Exhibit C – Aroclors) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (Section 1.2). In the event that the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.9.2.2 Copies of Aroclor Chromatograms. Positively identified compounds shall be labeled with the names of compounds, either directly out from the peak on the chromatogram, or on a printout of the RTs on the data system printout if the RTs are printed over the peak on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D, and shall be labeled with the following information:

- EPA Sample Number;
- Volume injected (µL);
- Date and time of injections;
- On column concentration/amount including units;
- GC column identifier (by stationary phase and internal diameter);
- GC instrument identifier; and
- Scaling factor (label the x and y axes using a numerical scale).

1.9.2.3 Copies of Aroclor chromatograms for the second GC column shall be included and labeled as in Section 1.9.2.2.

1.9.2.4 Data System Printout

A printout of RT, corresponding peak height or peak area, and the on column amount shall accompany each

chromatogram. The printout shall be labeled with the EPA Sample Number and standard concentration level. In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/ECD Operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range. The GC/MS Operator shall also mark each integrated area with the letter "m" in the quantitation report.

1.9.2.5 All manual worksheets shall be included in the Sample Data Package.

1.9.2.6 Other Required Information. If Aroclors are confirmed by GC/MS, the Contractor shall submit copies of reconstructed ion chromatograms. Raw spectra and background-subtracted mass spectra must be submitted for at least three major peaks of Aroclor target compounds (see Exhibit C – Aroclors) that are identified in the sample and corresponding standard mass spectra. Compound names shall be clearly marked on all spectra.

1.9.3 Aroclor Standard Data **<B-2>**

1.9.3.1 Initial Calibration of Aroclors (FORM VI ARO-1, ARO-2, and ARO-3): For all GC columns, all instruments, in chronological order by GC column and instrument. **<B-3>**

1.9.3.2 Calibration Verification Summary (FORM VII ARO): For all calibration verification standards on all GC columns and instruments, in chronological order by GC column and instruments. **<B-3>**

1.9.3.3 Analytical Sequence (FORM VIII ARO): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.9.3.4 Identification Summary for Multicomponent Analytes (FORM X ARO): For all samples with positively identified Aroclors, in order by increasing EPA Sample Number. **<B-3>**

1.9.3.5 Chromatograms and data system printouts shall be included for all standards, including the following:

- All Aroclor standards used for initial calibration on each column and instrument.
- All Aroclor standards used for calibration verification on each GC column and instrument.

- All Aroclor standards analyzed for confirmation.

1.9.3.6 A printout of RT and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA Sample Number. In addition, all chromatograms shall meet the acceptance criteria in Exhibit D, and shall be labeled with the following:

- NYSDEC Sample Number for the standard (e.g., AR10161OK, AR12601OK).
- Label all standard peaks with the compound name, either directly out from the peak on the chromatogram, or on the printout of RTs on the data system printout, if RTs are printed over the peak on the chromatogram.
- Total nanograms injected for each standard. When total nanograms injected appear on the printout, it is not necessary to include them on the chromatogram.
- Date and time of injection.
- GC column identifier (by stationary phase and internal diameter).
- GC instrument identifier.
- Scaling factor (label the x and y axes using a numerical scale).

Note: *In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/ECD Operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range. The GC/MS Operator shall also mark each integrated area with the letter "m" on the quantitation report.*

1.9.4 Aroclor Raw Quality Control (QC) Data **<B-2>**

1.9.4.1 Blank data shall be arranged in chronological order by extraction data. **<B-3>**

Note: *This order is different from that used for samples.*

- Tabulated results (FORM I ARO).
- Chromatogram(s) and data system printout(s) for each GC column and instrument used for analysis, labeled as in Sections 1.9.2.2 and 1.9.2.4.

1.9.4.2 Aroclor Laboratory Control Sample (LCS) Data **<B-3>**

- Tabulated results (FORM I ARO) of target compounds for both GC columns.

- Chromatograms and data system printouts for both GC columns, labeled as in Sections 1.9.2.2 and 1.9.2.4.

1.9.4.3 Aroclors Matrix Spike Data **<B-3>**

- Tabulated results (FORM I ARO) of target compounds for both GC columns.

- Chromatograms and data system printouts for both GC columns, labeled as in Sections 1.9.2.2 and 1.9.2.4.

1.9.4.4 Aroclors Matrix Spike Duplicate Data **<B-3>**

- Tabulated results (FORM I ARO) of target compounds for both GC columns.

- Chromatograms and data system printouts for both GC columns, labeled as in Sections 1.9.2.2 and 1.9.2.4.

1.9.5 Raw Gel Permeation Chromatography (GPC) Data **<B-2>**

1.9.5.1 GPC Calibration. The UV traces for the GPC calibration solution, chromatograms, and the data system reports for the GPC blank shall be arranged in chronological order for the GPC calibration.

- UV traces labeled with the GPC column identifier, date of calibration, and compound names. Compound names shall be placed directly out from the peak, or on the printout of RTs when the RTs are printed directly over the peak.

- Chromatograms and data system report(s) labeled as specified in Sections 1.9.2.2 and 1.9.2.4 for the GPC blank analyses.

- Chromatogram and data system report(s) for all standards used to assess the Aroclor pattern, labeled as specified in Section 1.9.2.2 and 1.9.2.4 (i.e., AR10161OK, AR12601OK from the initial calibration).

1.9.5.2 GPC Calibration Verification. The chromatogram and the data system reports(s) shall be arranged in chronological order for the GPC calibration check.

- Chromatograms and data system report(s) for standards used to assess the Aroclor pattern, labeled as specified in Sections 1.9.2.2 and 1.9.2.4 (i.e., Aroclor Standard Mixture 1016/1260 from the initial calibration sequence).

1.9.6 Copy of Calculations **<B-2>**

The Laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the quantitation report. If manipulations are performed by a software package, a copy of the formula used must be supplied as well as values for all terms in the formula.

Note: *All correction factors and equations utilized must be indicated on the work sheet.*

1.9.7 Copy of Extraction Logs **<B-2>**

These logs must be legible and include: (1) date, (2) sample weights and volumes, (3) sufficient information to unequivocally identify which QC samples (i.e. matrix spike, matrix spike duplicate, matrix spike blank) correspond to each batch extracted, (4) comments describing any significant sample changes or reactions which occur during preparation, and (5) final volumes and vial identification numbers.

1.10 Inorganic Data **<B-1>**

Sample data shall be submitted with the Inorganic Analysis Data Reporting Forms for all samples in the SDG, arranged in increasing alphanumeric NYSDEC sample number order, followed by the QC analyses data, quarterly and annual verification of method and instrument parameter forms, raw data, and copies of the digestion and distillation logs.

1.10.1 Results – Inorganic Analysis Data Sheet [FORM IA-IN and FORM IB-IN] – Tabulated analytical results (identification and quantitation) of the requested analytes (Exhibit C) must be accompanied by a signed statement in the SDG narrative. This signature validates and allows for the release the results. If the Laboratory Manager cannot validate all data reported for each sample, he/she must provide a detailed description of the problems associated with the sample(s) on the Cover Page. (**<B-2>** marking the beginning of results from each new fraction and/or analysis method)

1.10.1.1 Appropriate concentration units must be specified and entered on FORM IA-IN and FORM IB-IN. The quantitative values shall be reported in units of micrograms per liter (µg/L) for aqueous samples and milligrams per kilogram (mg/kg) for solid samples. Other units are acceptable only for trace level analyses. Results for solid sample must be reported

on a dry weight basis. Analytical results must be reported to two significant figures if the result value is less than 10 and to three significant figures if the value is greater than or equal to 10. Results for percent solids must be reported to one decimal place. The preceding discussion concerning significant numbers applies to FORM IA-IN, IB-IN, and IX-IN only. For the other forms, follow the Reporting Requirements and Order of Data Deliverables (Con't) instructions specific to those forms as discussed in this exhibit.

1.10.2 Quality Control (QC) Data **<B-2>**

1.10.2.1 The QC Summary for inorganic analysis shall contain the forms listed below.

Note: *If more than one form is necessary, duplicate forms must be arranged in chronological order.*

1.10.2.1.1 Initial and Continuing Calibration Verification [FORM IIA-IN] **<B-3>**

1.10.2.1.2 CRQL Check Standard [FORM IIB-IN]

1.10.2.1.3 Blanks [Form III-IN] **<B-3>**

1.10.2.1.4 ICP-AES Interference Check Sample [FORM IVA-IN] **<B-3>**

1.10.2.1.5 ICP-MS Interference Check Sample [FORM IVB-IN] **<B-3>**

1.10.2.1.6 Matrix Spike Sample Recovery [FORM VA-IN] **<B-3>**

1.10.2.1.7 Post-Digestion Spike Sample Recovery [FORM VB-IN] **<B-3>**

1.10.2.1.8 Duplicates [FORM VI-IN] **<B-3>**

1.10.2.1.9 Laboratory Control Sample [FORM VII-IN] **<B-3>**

1.10.2.1.10 ICP-AES and ICP-MS Serial Dilutions [FORM VIII-IN] **<B-3>**

1.10.2.1.11 Method Detection Limits (Annually) [FORM IX-IN] **<B-3>**

1.10.2.1.12 ICP-AES Interelement Correction Factors (Quarterly) [FORM XA-IN] **<B-3>**

1.10.2.1.13 ICP-AES Interelement Correction Factors (Quarterly) [FORM XB-IN] **<B-3>**

1.10.2.1.14 ICP-AES and ICP-MS Linear Ranges (Quarterly) [FORM XI-IN] **<B-3>**

1.10.2.1.15 Preparation Log [FORM XII-IN] **<B-3>**

1.10.2.1.16 Analysis Run Log [FORM XIII-IN] **<B-3>**

1.10.2.1.17 ICP-MS Tune [FORM XIV-IN] **<B-3>**

1.10.2.1.18 ICP-MS Internal Standards Relative Intensity Summary [FORM XV-IN] **<B-3>**

***Note**: Copies of Verification of Instrument Parameters forms for the current quarter must be submitted with each data package*.

1.10.3 Raw Data **<B-2>**

For each reported value, the Laboratory shall include in the Sample Data Package all raw data from the instrument used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample results. This statement does not apply to the quarterly and annual Verifications of Instrument Parameters submitted as part of each Sample Data Package. When analysis of the ICP-AES or ICP-MS target analytes listed in Exhibit C (or any subset or additional analytes) is requested, the raw data shall include, for all samples, not only the results for the requested analytes(s), but also those for all the interferents. The raw data shall also contain the results of any other analyte(s), which have been determined to interfere with the requested analyte(s).

1.10.3.1 Raw data must contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Batch Sheets) used for the sample results. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the Method Detection Limit (MDL). Raw data shall not be corrected for dilutions or volume adjustments. All Atomic Absorption (AA), Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES), and Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) instruments shall provide a legible hardcopy of the direct real-time instrument readout (i.e., strip charts, printer tapes, etc.) or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included. A hardcopy of the instrument's direct sequential readout shall be included for cyanide if the instrument has the capability.

1.10.3.2 The order of raw data in the Sample Data Package for inorganic analyses shall be: ICP-AES, Graphite Furnace Atomic Adsorption (GFAA), ICP-MS, Mercury, and Cyanide. All raw data shall include concentration units for ICP and absorbance or concentration units for AA, Mercury, and Cyanide. (**<B-3>** marking the beginning of raw data for each separate method)

1.10.3.3 The ICP-MS raw data shall also contain the turbidity measurement results [in Nephelometric Turbidity Units (NTU)] for the field samples.

1.10.3.4 Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

1.10.3.5 Raw data shall be labeled with NYSDEC sample number and appropriate codes, as shown in Exhibit B, "Table 2 – Codes for Labeling Data", to unequivocally identify:

- Calibration standards, including source and preparation date. Standard preparation logbooks can be submitted if they contain this information;
- Initial and Continuing Calibration Blanks (ICBs/CCBs) and Preparation Blanks (PBs).
- Initial and Continuing Calibration Verification (ICV/CCV) standards, Interference Check Samples, serial dilution samples, Contract Required Quantitation Limit (CRQL), Check Standard (CRI), Laboratory Control Sample (LCS), and Post Digestion Spike;
- Diluted and undiluted samples (by NYSDEC sample number) and all weights, dilutions, and volumes used to obtain the reported values (if the volumes, weights and dilutions are consistent for all samples in a given SDG, a general statement outlining these parameters is sufficient);
- Duplicates;
- Spikes (indicating standard solutions used, final spike concentrations, and volumes involved). If spike information (source, concentration, volume) is consistent for a given SDG, a general statement outlining these parameters is sufficient;

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

- Time and date of each analysis. Instrument run logs can also be submitted if they contain time and date of analysis. If the instrument does not automatically provide times of analysis, these shall be manually entered on all raw data (e.g., ICV/CCV, blanks, and the CRQL check standard.

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

- Integration times for AA analyses.

1.10.3.6 Digestion and Distillation Logs. The following logs shall be submitted as appropriate for each preparation procedure: digestion logs for ICP-AES, ICP-MS, mercury preparations, and cyanide. These logs shall include: (1) date; (2) sample weights and volumes, with initial sample weight/volume and final volume clearly indicated; (3) sufficient information to unequivocally identify which QC samples (i.e., LCS, PB) correspond to each batch digested; (4) comments describing any sufficient sample changes or reactions which occur during preparation shall be entered in the log and noted in the SDG Narrative; (5) indication of pH less than 2 or greater than 12, as applicable; and (6) identification of the sample preparer(s) [signature(s)]. **<B-3>**

1.10.4 Copy of Calculations – The Laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the instrument output report. If manipulations are performed by a software package, a copy of the formula used must be supplied, as well as, values for all terms in the formula. **<B-2>**

Note: *All correction factors and equations utilized must be indicated on the work sheet.*

2.0 ASP Category A

2.1 Cover Documentation **<B-1>** - See Requirements listed in Section 1.1 above.

2.2 SDG Narrative **<B-1>** - See Requirements listed in Section 1.2 above.

2.2.1 In addition to the requirements listed in Section 1.2, the Laboratory shall also document any out of range QC parameters associated with the data. Indicate what QC parameters were out of control, the limit that was exceeded, the result of the QC in exceedance, what samples are associated with that QC item, and how the results of those samples may be affected by the out of range QC.

2.3 Contract Lab Sample Information Sheets **<B-2>** - See Requirements listed in Section 1.4 above.

2.4 Chain-of-Custody Forms **<B-1>** - See Requirements listed in Section 1.5 above.

2.5 NYSDEC Data Package Summary Forms **<B-1>** - Requirements and Instructions for these forms are listed in Section IV of this Exhibit.

2.6 GC/MS Volatiles Data **<B-1>**

2.6.1 Sample Data

Sample data shall be arranged in packets consisting of the respective "Organic Analysis Data Sheet" (FORM I VOA-1, VOA-2) followed by the FORM I VOA-TIC for that sample. These packets shall be arranged in order of increasing NYSDEC sample number, considering both numbers and letters. For a detailed explanation of the Volatile FORM I requirements, see Sections 1.6.2.1 and 1.6.2.2 above.

2.7 GC/MS Semivolatiles Data **<B-1>**

2.7.1 Sample Data

Sample data shall be arranged in packets consisting of the respective "Organic Analysis Data Sheet" (FORM I SV-1, SV-2, SV-SIM) followed by the FORM I SV-TIC for that sample. These packets shall be arranged in order of increasing NYSDEC sample number, considering both numbers and letters. For a detailed explanation of the Semivolatile FORM I requirements, see Sections 1.7.2.1, 1.7.2.2, and 1.7.2.3 above.

2.8 Pesticide Data **<B-1>**

2.8.1 Sample Data

Sample data shall be reported on individual "Organic Analysis Data Sheet(s)" (FORM I PEST). These forms shall be arranged in order of increasing NYSDEC sample number, considering both

numbers and letters. For a detailed explanation of the Pesticide FORM I requirements, see Sections 1.8.2.1, above.

2.9 Aroclor Data **<B-1>**

2.9.1 Sample Data

Sample data shall be reported on individual "Organic Analysis Data Sheet(s)" (FORM I ARO). These forms shall be arranged in order of increasing NYSDEC sample number, considering both numbers and letters. For a detailed explanation of the Aroclor FORM I requirements, see Sections 1.9.2.1, above.

2.10 GC Organic Data (Includes all Organic data generated using a GC or GC-type instrument that does not fit into any of the categories listed in Sections 2.6-2.9.) **<B-1>**

2.10.1 Sample Data

Sample data should be reported using modified versions of the "FORM I" used in the above organic categories. Questions regarding the modification of the FORM I's for this data should be directed to the NYSDEC Quality Standards and Analytical Management Section. See also Section 3.10 for further explanation.

2.11 Inorganic Data **<B-1>**

2.11.1 Sample Data

Sample data shall be submitted with the "Inorganic Analysis Data Reporting Forms" (FORM IA-IN and FORM IB-IN) for all samples in the SDG, arranged in increasing alphanumeric NYSDEC sample number order. For a detailed explanation of the Inorganic FORM I requirements, see Sections 1.10.2, above.

2.12 Toxicity Characteristic Leaching Procedure (TCLP) Data **<B-1>**

2.12.1 Sample Data (**<B-2>** the beginning of data for each unique analysis fraction)

Sample data shall be submitted on modified reporting forms based on the reporting forms used in Sections 2.6-2.11. The analysis specific FORM I's should be modified to include the following TCLP specific information, either in the footer or the header of the form:

- Matrix of Original Sample
- % Solid content of the sample, if the sample was a filterable liquid please fill this field with "<0.5%".
- Start date/time of TCLP extraction

- End date/time of TCLP extraction
- Start Temperature of TCLP extraction room
- End Temperature of TCLP extraction room.
- TCLP Fluid used (#1 or #2)
- Sample pH
- Ending extract pH

3.0 ASP Category B

3.1 Cover Documentation **<B-1>** - See Requirements listed in Section 1.1 above.

3.2 SDG Narrative **<B-1>** - See Requirements listed in Section 1.2 above.

3.3 Contract Lab Sample Information Sheets **<B-1>** - See Requirements listed in Section 1.4 above.

3.4 Chain-of-Custody Forms **<B-1>** - See Requirements listed in Section 1.5 above.

3.5 NYSDEC Data Package Summary Forms **<B-1>** - Requirements and Instructions for these forms are listed in Section IV of this Exhibit.

3.6 GC/MS Volatiles Data **<B-1>**

3.6.1 Volatiles QC Summary **<B-2>**

3.6.1.1 System Monitoring Compound Summary – See requirements listed in Section 1.6.1.1.

3.6.1.2 Matrix Spike/Matrix Spike Duplicate Summary – See requirements listed in Section 1.6.1.2.

3.6.1.3 QC Check Sample/Standard – (If Applicable) Reported on a modified version of FORM I VOA-1, VOA-2. The form should be modified in such a way that the header clearly states that the results being reported are from a "QC Check Sample/Standard".

3.6.1.4 Method Blank Summary – See requirements listed in Section 1.6.1.3.

3.6.1.5 GC/MS Instrument Performance Check – See requirements listed in Section 1.6.1.4.

3.6.1.6 Internal Standard Area and RT Summary – See requirements listed in Section 1.6.1.5.

3.6.1.7 Instrument Detection Limits – Reported on a modified version of FORM I VOA-1, VOA-2. The form should be modified in such a way that the header clearly states that the results being reported are the statistically determined detection limits for a given instrument using a given method. Detection limits should be determined annually. The "Q" column on the FORM I's should not be used.

3.6.2 Sample Data **<B-2>**

Sample Data should be reported in the same format and order as detailed in Section 1.6.2.

3.6.3 Standards Data **<B-2>**

Standard Data should be reported in the same format and order as detailed in Section 1.6.3.

3.6.4 Raw QC Data **<B-2>**

Raw QC Data should be reported in the same format and order as detailed in Section 1.6.4. In addition to the requirements listed in Section 1.6.4, the raw data for "QC Check Sample/Standard" should be reported following the raw data for "Matrix Spike Duplicate Data" as follows:

3.6.4.1 QC Check Sample/Standard **<B-3>**

3.6.4.1.1 Tabulated results (FORM I-VOA) of <u>all</u> target compounds. FORM I-VOA-TIC is <u>not</u> required.

3.6.4.1.2 Reconstructed ion chromatograms(s) and quantitation reports(s) or legible (GC/MS), labeled as in Section 1.6.2.4. Spectra are <u>not</u> required.

3.6.5 Copy of Calculations **<B-2>**

Please provide copies of calculations as specified in Section 1.6.5.

3.6.6 Copy of Extraction Logs **<B-2>**

Please provide copies of extraction logs as specified in Section 1.6.6.

3.7 GC/MS Semivolatiles Data **<B-1>**

3.7.1 QC Summary **<B-2>**

3.7.1.1 System Monitoring Compound Summary – See requirements listed in Section 1.7.1.1.

3.7.1.2 Matrix Spike/Matrix Spike Duplicate Summary – See requirements listed in Section 1.7.1.2.

3.7.1.3 QC Check Sample/Standard – (If Applicable) Reported on a modified version of FORM I SV-1, SV-2. The form should be modified in such a way that the header clearly states that the results being reported are from a QC Check Sample/Standard.

3.7.1.4 Method Blank Summary – See requirements listed in Section 1.7.1.3.

3.7.1.5 GC/MS Instrument Performance Check – See requirements listed in Section 1.7.1.4.

3.7.1.6 Internal Standard Area and RT Summary – See requirements listed in Section 1.7.1.5.

3.7.1.7 Instrument Detection Limits – Reported on a modified version of FORM I SV-1, SV-2. The form should be modified in such a way that the header clearly states that the results being reported are the statistically determined detection limits for a given instrument using a given method. Detection limits should be determined annually. The "Q" column on the Form Is should not be used.

3.7.2 Sample Data **<B-2>**

Sample Data should be reported in the same format and order as detailed in Section 1.7.2. In addition to all the requirements listed under Section 1.7.2, any GPC Chromatograms produced during the analysis of the samples should be included at the end of Section 3.7.2.

3.7.3 Standards Data **<B-2>**

Standard Data should be reported in the same format and order as detailed in Section 1.7.3. In addition to all the requirements listed under Section 1.7.3, data for "Semivolatile GPC Calibration Data" should be listed as follows:

3.7.3.1 Semivolatile GPC Calibration Data – UV detector traces showing peaks that correspond to the compounds in the semivolatile GPC calibration mixture. Traces must be labeled with GPC column identifier, date of calibration, and with compound names labeled either directly out from the peak, or on a printout of retention times, if retention times are printed over the peak. Do not include FORM IX Pest-2, as the compounds used on that form

are not appropriate for semivolatile sample extracts. **<B-3>**

3.7.4 Raw QC Data **<B-2>**

Raw QC Data should be reported in the same format and order as detailed in Section 1.7.4. In addition to the requirements listed in Section 1.7.4, the following should be added directly after the raw data for "Matrix Spike Duplicate Data" but before the GPC Raw QC data:

3.7.4.1 QC Check Sample/Standard **<B-3>**

3.7.4.1.1 Tabulated results (FORM I-SV) of all target compounds. FORM I-SV-TIC is not required.

3.7.4.1.2 Reconstructed ion chromatograms(s) and quantitation reports(s) or legible (GC/MS), labeled as in Section 1.7.2.4. Spectra are not required.

3.7.5 Copy of Calculations **<B-2>**

Please provide copies of calculations as specified in Section 1.7.5.

3.7.6 Copy of Extraction Logs **<B-2>**

Please provide copies of extraction logs as specified in Section 1.7.6.

3.8 GC/ECD and GC/MS Pesticide Data **<B-1>**

3.8.1 QC Summary **<B-2>**

3.8.1.1 System Monitoring Compound Summary – See requirements listed in Section 1.8.1.1.

3.8.1.2 Matrix Spike/Matrix Spike Duplicate Summary – See requirements listed in Section 1.8.1.2.

3.8.1.3 Laboratory Control Sample Recovery – See requirements listed in Section 1.8.1.3.

3.8.1.4 QC Check Sample/Standard – (If Applicable) Reported on a modified version of FORM I PEST-1. The form should be modified in such a way that the header clearly states that the results being reported are from a QC Check Sample/Standard.

3.8.1.5 Method Blank Summary – See requirements listed in Section 1.8.1.4.

3.8.1.6 GC/MS Instrument Performance Check – (if Applicable) No Form exists for this requirement. A Narrative statement should be included for GC/MS pesticide data. The narrative should document the following.

- Frequency at which instrument performance checks were performed. Include the date and time the check was run and the sample runs (file IDs) associated with the check.
- The results of the Instrument Performance Check (Pass or Fail).
- The criteria used to evaluate the acceptance of the check.

3.8.1.7 Instrument Detection Limits – Reported on a modified version of FORM I PEST-1. The form should be modified in such a way that the header clearly states that the results being reported are the statistically determined detection limits for a given instrument using a given method. Detection limits should be determined annually. The "Q" column on the Form Is should not be used.

3.8.2 Sample Data **<B-2>**

Sample Data should be reported in the same format and order as detailed in Section 1.8.2, up to and including Section 1.8.2.5 (omit 1.8.2.6). In addition to all the requirements listed under Section 1.8.2, please include the following:

3.8.2.1 UV traces from GPC (if GPC performed).

3.8.2.2 If pesticides are confirmed by GC/MS or run solely via GC/MS, the Laboratory shall submit copies of reconstructed ion chromatograms, raw spectra and copies of background-subtracted mass spectra of Pesticide target compounds listed in Exhibit C that are identified in the sample and corresponding background-subtracted Superfund-TCL standard mass spectra. Compound names must be clearly marked on all spectra. For multi-component pesticides/Aroclors confirmed by GC/MS, the Laboratory shall submit mass spectra of 3 major peaks of multi-component compounds from samples and standards.

3.8.3 Standards Data **<B-2>**

Standard Data should be reported in the same format and order as detailed in Section 1.8.3. For the purposes of NYSDEC ASP Category B reporting the requirements of Section 1.8.3.4-7 may be omitted. In addition to the requirements of Section 1.8.3, please include the following:

3.8.3.1 Pesticide GPC Calibration Data – UV detector traces showing peaks that correspond to the compounds in the pesticide GPC calibration mixture. Traces must be labeled with GPC column identifier, date of calibration, and with compound names labeled either directly out from the peak, or on a printout of retention times, if retention times are printed over the peak. **<B-3>**

3.8.4 Raw QC Data **<B-2>**

Raw QC Data should be reported in the same format and order as detailed in Section 1.8.4. In addition to the requirements listed in Section 1.8.4, the following should be added directly after the raw data for "Matrix Spike Duplicate Data":

3.8.4.1 QC Check Sample/Standard **<B-3>**

3.8.4.1.1 Tabulated results (FORM I-PEST) of all target compounds.

3.8.4.1.2 Chromatogram(s) and data system printout(s) (GC), as labeled in Section 1.8.2.2.

3.8.5 Copy of Calculations **<B-2>**

Please provide copies of calculations as specified in Section 1.8.5.

3.8.6 Copy of Extraction Logs **<B-2>**

Please provide copies of extraction logs as specified in Section 1.8.6.

3.9 GC/ECD and GC/MS Aroclor Data **<B-1>**

3.9.1 QC Summary **<B-2>**

3.9.1.1 System Monitoring Compound Summary – See requirements listed in Section 1.9.1.1.

3.9.1.2 Matrix Spike/Matrix Spike Duplicate Summary – See requirements listed in Section 1.9.1.2.

3.9.1.3 Laboratory Control Sample Recovery – See requirements listed in Section 1.9.1.3.

3.9.1.4 QC Check Sample/Standard – (If applicable) Reported on a modified version of FORM I ARO. The form should be modified in such a way that the header clearly states that the results being reported are from a QC Check Sample/Standard.

3.9.1.5 Method Blank Summary – See requirements listed in Section 1.9.1.4.

3.9.1.6 GC/MS Instrument Performance Check – (if Applicable) No Form exists for this requirement. A Narrative statement should be included for GC/MS Aroclor data. The narrative should document the following.

- Frequency at which instrument performance checks were performed. Include the date and time the check was run and the sample runs (file IDs) associated with the check.
- The results of the Instrument Performance Check (Pass or Fail)
- The criteria used to evaluate the acceptance of the check.

3.9.1.7 Instrument Detection Limits – Reported on a modified version of FORM I ARO. The form should be modified in such a way that the header clearly states that the results being reported are the statistically determined detection limits for a given instrument using a given method. Detection limits should be determined annually. The "Q" column on the Form Is should not be used.

3.9.2 Sample Data **<B-2>**

Sample Data should be reported in the same format and order as detailed in Section 1.9.2. In addition to all the requirements listed under Section 1.9.2, please include the following:

3.9.2.1 UV traces from GPC (if GPC performed).

3.9.2.2 If pesticides are confirmed by GC/MS or run solely via GC/MS, the Laboratory shall submit copies of reconstructed ion chromatograms, raw spectra and copies of background-subtracted mass spectra of Pesticide target compounds listed in Exhibit C that are identified in the sample and corresponding background-subtracted Superfund-TCL standard mass spectra. Compound names must be clearly marked on all spectra. For multi-component pesticides/Aroclors confirmed by GC/MS, the Laboratory shall submit mass spectra of 3 major peaks of multi-component compounds from samples and standards.

3.9.3 Standards Data **<B-2>**

Standard Data should be reported in the same format and order as detailed in Section 1.9.3. In addition to the requirements of Section 1.9.3, please include the following:

3.9.3.1 Pesticide GPC Calibration Data – UV detector traces showing peaks that correspond to the compounds in the pesticide GPC calibration mixture. Traces must be labeled with GPC column identifier, date of calibration, and with compound names labeled either directly out from the peak, or on a printout of retention times, if retention times are printed over the peak. **<B-3>**

3.9.4 Raw QC Data **<B-2>**

Raw QC Data should be reported in the same format and order as detailed in Section 1.9.4. In addition to the requirements listed in Section 1.9.4, the following should be added directly after the raw data for "Matrix Spike Duplicate Data":

3.9.4.1 QC Check Sample/Standard **<B-3>**

3.9.4.1.1 Tabulated results (FORM I ARO) of all target compounds.

3.9.4.1.2 Chromatogram(s) and data system printout(s) (GC), as labeled in Section 1.9.2.2.

3.9.5 Copy of Calculations **<B-2>**

Please provide copies of calculations as specified in Section 1.9.5.

3.9.6 Copy of Extraction Logs **<B-2>**

Please provide copies of extraction logs as specified in Section 1.9.6.

3.10 GC Organic Data **<B-1>**

On occasion NYSDEC may require samples to be analyzed by various GC methods for organic analytes. The reporting of these analytes represents a challenge because no EPA CLP forms exist to report this data. Since most environmental reporting software packages are very rigid in their output formats, it is prohibitive for NYSDEC to develop specialized reporting forms for GC organic data. NYSDEC recognizes that some software venders have created "CLP-like" reporting for GC organic data, and when feasible NYSDEC recommends the use of such software for this data. If such software is not available or unobtainable to the laboratory, the laboratory should modify and use the reporting formats and reports specified in Sections 1.6, 1.7, 1.8, and 1.9. The order of the reporting elements should be unaltered from the original Section being modified. If the reporting software package allows, the identifier for the Forms should be changed to "GC" (i.e. FORM I GC, FORM II GC, etc.). The basic structure of this reporting section should be as follows:

3.10.1 QC Summary **<B-2>**

3.10.1.1 Surrogate/System Monitoring Compounds Recovery Reports (FORM II GC)

3.10.1.2 Matrix Spike/Matrix Spike Duplicate Summary (FORM III GC)

3.10.1.3 QC Check Sample/Standard (FORM I GC + Raw Data)

3.10.1.4 Method Blank Summary (FORM IV GC)

3.10.1.5 Instrument Detection Limits (Performed annually)

3.10.2 Sample Data **<B-2>**

3.10.2.1 Results and raw data for each individual sample should be assembled in packets as follows, and placed in order according to NYSDEC Sample ID, from lowest to highest:

3.10.2.1.1 Target Compound Results (FORM I GC)

3.10.2.1.2 Manual calculation worksheets, if applicable,

3.10.2.1.3 Appropriate raw instrument data**,**

3.10.2.1.4 GPC chromatograms or other qualitative sample specific clean-up data, if applicable.

3.10.3 Standards Data **<B-2>**

3.10.3.1 Initial Calibration Data

3.10.3.2 Continuing Calibration Data

3.10.3.3 Standard chromatograms and data system printouts for all standards.

3.10.4 Copy of Calculations **<B-2>**

3.10.5 Copy of Extraction Logs **<B-2>**

3.11 Inorganic Data **<B-1>**

Sample data shall be submitted with the Inorganic Analysis Data Reporting Forms for all samples in the SDG, arranged in increasing alphanumeric DEC sample number order, followed by the QC analysis data, Quarterly Verification of Instrument Parameter forms, raw data, and copies of the digestion and distillation logs.

3.11.1 Results – Should be reported on FORM IA-IN and FORM IB-IN, and reported according to the specifications in Section 1.10.1. **<B-2>**

3.11.2 Quality Control Data – Should be reported and ordered per the specifications listed above in Section 1.10.2. Verification of Instrument Parameters should also be reported in this Section. Frequency of verifications is unmodified from the CLP requirements. **<B-2>**

3.11.3 Raw Data – Should be reported and ordered per the specifications listed above in Section 1.10.3. **<B-2>**

3.11.4 Digestion and Prep Logs – Should be reported and ordered per the specifications listed above in Section 1.10.4. **<B-2>**

3.12 Wet Chemistry Data **<B-1>**

On occasion NYSDEC may require samples to be analyzed by wet chemistry methods for "conventional" analytes. The reporting of these analytes represents a challenge because no EPA CLP forms exist to report such data. Since most environmental reporting software packages are very rigid in their output formats, it is prohibitive for NYSDEC to develop specialized reporting forms for wet chemistry analysis data. NYSDEC recognizes that some software venders have created "CLP-like" reporting for wet chemistry parameters, and when feasible NYSDEC recommends the use of such software for this data. If such software is not available or unobtainable to the laboratory, the laboratory should modify and use the reporting formats and reports specified in Sections 1.10 (Inorganics). The order of the reporting elements should be unaltered from the original Section being modified. If the reporting software package allows, the identifier for the Forms should be changed to "WC" (i.e. FORM I-WC, FORM II-WC, etc.). The basic structure of this reporting section should be as follows:

3.12.1 Results – Modified Inorganic Analysis Data Sheet **<B-2>**

Tabulated analytical results (identification and quantitation) of the specified analytes (Exhibit C) must be accompanied by a specific, signed statement in the SDG Narrative, which authorizes the validation and release of analytical results (Section 1.2). If the Laboratory Manager cannot validate all data reported for each sample, he/she must provide a detailed description of the problems associated with the sample(s) on the Cover Page.

Appropriate concentration units must be specified and entered on FORM I-WC. The quantitative values shall be reported in units of micrograms per liter (µg/L) for aqueous samples and milligrams per kilogram (mg/kg) for solid samples. Units may be adjusted in order to make excessively large or small concentration numbers more manageable. Results for solid samples must be reported on a dry weight basis. Analytical results must be reported to two significant figures if the result value is less than 10; to three significant figures if the value is greater than or equal to 10. Results for percent solids must be

reported to one decimal place. Data qualifiers should be added according to Table 2.

3.12.2 Quality Control Data – include each only when applicable to the parameter being analyzed. **<B-2>**

3.12.2.1 Initial and Continuing Calibration Verification

3.12.2.2 CRQL Standard for Wet-Chemistry Analysis

3.12.2.3 Blanks

3.12.2.4 Spike Sample Recovery

3.12.2.5 Post Digest Spike Sample Recovery

3.12.2.6 Duplicates

3.12.2.7 Laboratory Control Sample

3.12.2.8 Holding Times

3.12.3 Raw Data **<B-2>**

For each reported value, the Laboratory shall include in the data package all raw data from the instrument used to obtain that value and the QA/QC values reported (except for raw data for Verifications of Instrument Parameters). Raw data must contain all instrument readouts used for the sample results, including those readouts that may fall below the IDG. ALL instruments must provide a legible hard copy of the direct real-time instrument readout (i.e., stripcharts, printer tapes, etc.). A photocopy of the direct sequential instrument readout must be included. A hardcopy of the direct instrument readout for cyanide must be included if the instrumentation has the capability. All raw data shall include absorbance values with concentration units (unless instrument direct readout is in concentration units). A photocopy of manual worksheets used must be included for all non-instrumental parameters. Raw data must be labeled with NYSDEC sample number or be associated to a group of NYSDEC sample numbers for the following:

3.12.3.1 Calibration standards, including source and prep date.

3.12.3.2 Initial and continuing calibration blanks and preparation blanks.

3.12.3.3 Initial and continuing calibration verification standards.

3.12.3.4 Diluted and undiluted samples (by NYSDEC sample number) and all weights, dilutions and volumes used to obtain the reported values. (If the volumes, weights, and dilutions are consistent for all samples in a given

SDG, a general statement outlining these parameters is sufficient).

3.12.3.5 Duplicates.

3.12.3.6 Spikes (indicating standard solutions used, final spike concentrations, volumes involved). If spike information (source, concentration, volume) is consistent for a given SDG, a general statement outlining these parameters is sufficient.

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

3.12.3.8 Time and date of each analysis. Instrument run logs can be submitted if they contain this information. If the instrument does not automatically provide times of analysis, these must be manually entered on all raw data for initial and continuing calibration verification and blanks, as well as, interference check samples and linear range analysis.

3.12.4 Digestion and Distillation Logs **<B-2>**

These logs must include: (1) date, (2) sample weights and volumes, (3) sufficient information to unequivocally identify which QC samples (i.e., laboratory control sample, preparation blank) correspond to each batch digested, (4) comments describing any significant sample changes or reactions which occur during preparation, and (5) indication of pH <2 or >12, as applicable.

3.13 Toxicity Characteristic Leaching Procedure (TCLP) Data **<B-1>**

Sample data shall be submitted with the Toxicity Characteristic Leaching Procedure Analysis Data Reporting Forms for all samples in the SDG, arranged in packets by analysis fraction. The packets shall consist of the sample results in increasing alphanumeric DEC sample number order, followed by the QC analyses data, Verification of Instrument Parameters forms, raw data, and copies of the digestion and distillation logs pertaining to that analysis fraction. The logbook page or pages dedicated to the TCLP extraction procedure should be included at the end of all the packets for the applicable analysis fractions.

Neither NYSDEC nor EPA CLP have created specific forms for reporting the results of TCLP extracted analytes. Due to the lack of any standardized forms for this data, it is unlikely that any commercial software would be or will be available to report TCLP analysis data. NYSDEC requests that the laboratory report TCLP analysis results on the analogous FORM *X* reports for each analysis and/or QC procedure

performed on the TCLP extraction fluid. The only modification to the traditional CLP-type Forms specified for use in the NYSDEC ASP is that these forms clearly be marked either in the header or in the footer comments that the results being reported on the form are from the analysis of a TCLP extract. If feasible the codes for the forms should be modified and a final suffix of "-TCLP" should be added. For example a "FORM 1 VOA-1" reported for the analysis of a TCLP extract would be "FORM 1 VOA-1-TCLP".

Note: *Data for every separate analysis performed on a TCLP extract should be separated and marked with a second level bookmark* ***(<B-2>)****.*

3.13.1 Results – Toxicity Characteristic Leaching Procedure (TCLP) Analysis Data Sheet (TCLP Modified FORM Is) **<B-3>**

Tabulated analytical results (identification and quantitation) of the specified analytes (Exhibit C) must be accompanied by a specific, signed statement in the SDG Narrative, which authorizes the validation and release of analytical results (Section 3.1). If the Laboratory Manager cannot validate all data reported for each sample, he/she must provide a detailed description of the problems associated with the sample(s) on the Cover Page.

Appropriate concentration units must be specified and entered on TCLP Modified FORM Is. The quantitative values shall be reported in units of milligrams per liter (mg/L). No other units are acceptable. Analytical results must be reported to two significant figures if the result value is less than 10; to three significant figures if the value is greater than or equal to 10. Results for percent solids must be reported to one decimal place. Qualifiers are to be added according to Table 1 and Table 2.

3.13.1.1 Organic Data Results – Should be reported in order by NYSDEC Sample ID, with the raw data and TIC's (if applicable) directly following the modified FORM I from the sample. See specifications in Sections 1.6.2, 1.7.2, 1.8.2, and 1.9.2 for instructions of reporting sample result for TCLP Organics

3.13.1.2 Inorganic Data Results – Should be reported according to the specifications listed in Section 1.10.1. Raw data will not be assembled directly after the sample data, but included later in Section 3.14.4.

3.13.2 TCLP Quality Control Data – quality control reporting should be accomplished in a manner similar to that used to report sample data on the modified FORM I's above. The key features of all CLP or CLP-like reporting forms should be retained, while notation should be added to denote that the results being reported are from the analysis of a TCLP extract. **<B-3>**

3.13.2.1 Organic Analysis of TCLP Extracts

3.13.2.1.1 Report all QC data according to the specifications listed in Sections 1.6.1, 1.7.1, 1.8.1, and 1.9.1.

3.13.2.2 Inorganic analysis of TCLP Extracts

3.13.2.2.1 Report all QC data according to the specifications listed in Section 1.10.2.

3.13.3 Verification of Instrument Parameters **<B-3>**

3.13.3.1 Organic Analysis of TCLP Extracts – Not required to be included in data package.

3.13.3.2 Inorganic analysis of TCLP Extracts – Data pertaining to the verification of inorganic instrument parameters relative to TCLP extract analysis should be reported according to the specifications in Section 1.10.3.

Note: *Copies of Verification of Instrument Parameters forms for the current quarter must be submitted with each data package.*

3.13.4 Raw Data **<B-3>**

3.13.4.1 Organic Raw Data – Raw data supporting sample results should be included in Section 3.13.1.

3.13.4.1.1 Standards Data – This section should include the raw data for calibration and calibration verifications run to support the analysis of the TCLP extract. See Sections 1.6.3, 1.7.3, 1.8.3, and 1.9.3 for instructions and specifications.

3.13.4.1.2 Raw QC Data – This section should include the raw data need to support the QC results reported in Section 3.13.2.1. The data should be presented and arranged according to the specifications in Sections 1.6.5, 1.7.5, 1.8.5, and 1.9.5.

3.13.4.2 Inorganic Raw Data – Raw data supporting the results reported in Section 3.13.1 and Section 3.13.2 should be included in this section. The raw data should follow the order and format specified in section 1.10.3.

3.13.5 Prep/Digestion Logs (Analysis Specific) – Directly following the Forms and raw data for a fraction packet, all applicable preparation and digestion logs should be included that are relevant to that analysis fraction. **<B-3>**

3.13.6 Prep Logs (TCLP Specific) – A report or copy of the logbook for the TCLP extraction process is required. If multiple TCLP extraction batches were performed within the SDG, a report or logbook page per TCLP batch is required. This report should include the following information: **<B-2>**

- NYSDEC Sample IDs
- Laboratory Sample IDs
- Sample Matrix
- % Total Solids for Sample
- Extract Filterable or Non-filterable
- Average Particle Size in Sample
    - Was Sample Particle Size Reduced?
- Data on Extraction Fluid Determination
    - Initial pH of Sample
    - pH of Sample after Addition of Acid
    - Extraction Fluid Used (Type 1 or Type 2)
- Data on the Extraction Fluid
    - Extraction Fluid Type
    - Extraction Fluid Batch ID
    - Initial pH of Fluid
- Amount (grams) of Sample Extracted
- TCLP Extraction Start Date and Time
- Temperature of TCLP Extraction Room at Start Time
- TCLP Extraction End Date and Time
- Temperature of TCLP Extraction Room at End Time
- pH of TCLP Extract at End Time

F. – Data In Computer Readable Form

Exhibit H details the requirements for electronic data deliverables (EDDs) and any other sample data submissions required to comply with NYSDEC database requirements.

For the purposes of this Protocol, and specifically Exhibit H, Sample Data Packages and Sample Summary Data Packages in the form of .PDF files are not considered “Data In

Computer Readable Form". Requirements for .PDF files are given in this Exhibit, under Section V.

G. – Electronic Instrument Data

The Laboratory must archive all raw and processed instrument data on portable electronic storage media, in the format specified by the instrument manufacturer. Portable electronic storage media can be any of the following: magnetic tapes, CD-ROM, DVD-ROM, DAT, ZIP Disks, or any other portable storage media meeting the following requirements: must be "locked, read only" after the initial "write" to the media, stable over time, easily stored on site. Data may be archived to a non-portable media such as an auxiliary hard drive, but the capability must exist to extract data upon request from NYSDEC. Data archived to an auxiliary hard drive must meet the following criteria: (a) the capability must exist to migrate the files back into the instruments data system in order to generate/regenerate appropriate analysis data and (b) the capability must exist to transfer archived files to portable storage media in order to ship the raw data to NYSDEC. This storage media must contain all instrument files used directly or indirectly to construct the NYSDEC Sample Data Packages. NYSDEC related instrument files do not need to be archived separately if the lab uses an all-inclusive archive technique for instrument data. Output files subject to this archive requirement include, but are not limited to, samples, blanks, spikes, matrix spikes, matrix spike duplicates, calibration standards, continuing calibrations, instrument tunes, as well as all laboratory-generated spectral libraries and quantitation reports required to generate the data package. The Laboratory shall maintain a written reference logbook of stored files to NYSDEC sample number, calibration data, standards, blanks, matrix spikes, and matrix spike duplicates. The logbook should include NYSDEC sample numbers and standard and blank ID's, identified by Case and Sample Delivery Group.

The Laboratory is required to retain the stored files for 3 years after data submission. During that time, the Laboratory shall submit copies of archived files and associated logbook pages within seven days after receipt of a written request from the Bureau of Watershed Assessment and Management.

H. – Samples and Extracts

1.0 Unused and Excess Sample Amounts

After the required sample aliquot has been successfully analyzed and reported, the Laboratory shall preserve any unused and excess sample amounts at the required storage temperature and conditions as specified in Exhibit I. Samples should be stored in their original containers, clearly lableled with their NYSDEC Sample Numbers and associated Case and SDG numbers. The Laboratory is required to retain samples for 365 days following data submission. During that time, the Laboratory shall submit samples and associated custody documents within seven days following receipt of a written request from the Bureau Watershed Assessment and Management or the Project Officer.

2.0 Sample Extracts (Organincs only)

The Laboratory shall preserve sample extracts at a temperature less than 4°C in bottles/vials with Teflon-lined septa. Extract bottles/vials shall be labeled with

NYSDEC sample number, Case number, and Sample Delivery Group (SDG) number. The Contractor shall maintain a logbook of stored extracts, listing NYSDEC Sample Numbers and associated Case and SDG numbers. The Laboratory is required to retain extracts for 365 days following data submission. During that time, the Laboratory shall submit extracts and associated logbook pages within seven days following receipt of a written request from the Bureau Watershed Assessment and Management or the Project Officer.

I. – Verification of Instrument Parameters

1.0 Organic Verifications

The contractor shall perform and report annual verification of MDLs by the technique specified in 40 CFR Part 136 using the analytical methods specified in Exhibit D (by type, matrix, and model for each instrument used on the contract) to the Bureau of Watershed Assessment and Management. All the MDLs shall meet the CRQLs specified in Exhibit C.

2.0 Inorganic Verifications

The Laboratory shall perform verification of instrument detection limits, method detection limits, correction factors, and linear ranges for those instrument-types specified in Exhibit E. The methods and frequency for such verifications are detailed in Exhibit E. For the ICP instrumentation and methods, the Laboratory shall also report annually interelement correction factors (including method of determination), wavelengths used, and integration times. Verification of Instrument Parameters forms for the current period shall be submitted in each Sample Delivery Group data package, using Forms X, XI, and XII. Submission of Full Verification of Instrument Parameters shall include the raw data used to determine those values reported.

3.0 All Analyses

Method Detection Limit (MDL) Study is to be performed at minimum annually, or for each new instrument brought into service, whichever is more frequent. Some analyses and methods may require more frequent running of the MDL study. If a method requires more frequent running of the MDL study, that requirement supercedes the annual requirement set herein. The information on current and past MDL studies should be maintained on file at the laboratory. The Laboratory shall maintain records for any and all instrument performance verifications performed for a period of 3 years. During that time, the Laboratory shall submit copies of such records within seven days following receipt of a written request from the Bureau Watershed Assessment and Management or the Project Officer.

J. – Preliminary Results

1.0 Organic Preliminary Results

The FORM I data results shall be submitted for all samples in one SDG of a Case. This includes tabulated target compound results (FORM I *XXXX-X*) for the volatile, semivolatile, pesticide, and Aroclor fractions, and Tentatively Identified

Compounds (FORM I *XXXX*-TIC) for the volatile and semivolatile fractions. The contractor shall clearly identify the Preliminary Results by labeling each FORM I and FORM I TIC as "Preliminary Results" under each form title (e.g., under "Volatile Organics Analysis Data Sheet", "Volatile Organics Analysis Data Sheet Tentatively Identified Compounds").

2.0 Inorganic Preliminary Results

The FORM I IN data results (including all appropriate qualifiers and flags) shall be submitted for all samples in one SDG of a Case. Sample analysis shall follow all requirements stipulated in the Method, Exhibit D. The Contractor shall clearly identify the Preliminary Results by labeling each FORM I as "Preliminary Results" under the form title (e.g., under "Inorganic Analysis Data Sheet"). The Contractor shall also include a disclaimer in the "Comments" field on all Form Is stating that the "Data results contained on the Form I are for scanning purposes only, and may not have been validated for CLP/ASP criteria." Copies of Sample Traffic Reports/Chain of Custody Records shall be submitted with the Preliminary Results.

3.0 All Preliminary Results (Organic and Inorganic)

Copies of Sample Traffic Reports/Chain of Custody Records shall be submitted with the Preliminary Results. The Contractor shall also submit a Cover Page following the specifications in Exhibit B, Part E, Section 1.1. In addition, the Cover Page shall be clearly labeled to indicate that the data being reported are Preliminary Results. The Cover Page shall contain the following statement, (usually included in the SDG Narrative) verbatim: **"I certify that these Preliminary Results are in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or the Manager's designee, as a verified by the following signature."** This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.

K. – Results of PE Samples

Results of Performance Evaluation (PE) Samples should be reported similar to a standard environmental sample with deliverables as specified in Items E and F (Sample Data Package (.PDF) and Electronic Data Deliverables (EDD)).

Table 1

List of Organic Method Qualifiers

Qualifier (Q)	Description
B	Entered if the analyte is found in the associated blank as well as the sample.
C	Applied to pesticide results when the identification has been confirmed by GC/MS.
D	Included when the all identified compounds in the analysis are at the secondary dilution factor.
E	Identified compounds whose concentrations exceed the calibration range of the instrument for that specific analysis.
J	Indicates an estimated value, may indicate one of the following, depending on the situation: (1) The reported value is estimated and below the MDL. (2) Used when estimating a concentration for TIC where a 1:1 response is assumed or when the result indicates the presence of a compound that meets the identification criteria, but the results is less than the quantitation limit, but greater than zero. (3) QC associated with this analyte is within warning limits.
N	Included for TIC that indicate presumptive evidence of a compound.
U	Entered if the analyte was analyzed for, but not detected.
P	Used for a pesticide/Aroclor target analyte when the concentration difference between 2 GC columns is greater than 25%; the lower value is flagged with a "P".
EMPC	"Estimated Maximum Possible Concentration" – The amount of analyte cannot be accurately quantified, so a maximum concentration has been estimated for the compound.
"XYZ"	"Wildcard" or Laboratory defined qualifier.

Note: *Form I allows only one character in each qualifier column. If multiple qualifiers are applicable, please assess qualifier priority in the following order: U, E, J, B, D, C, P, N. Reporting done in the EDD may include multiple qualifiers when applicable, separated by a single space.*

Table 2

List of Inorganic Method Qualifiers

Qualifier	Column[1]	Description
Concentration qualifiers		
B	C	Entered if the reported value was less than the CRDL, but greater than the IDL.
U	C	Entered if the analyte was analyzed for, but not detected.
J	C	Entered if the reported value is estimated and below the MDL.
*	C	Duplicate precision exceeds RPD limit.
M	C	Replicate precision exceeds RPD limit.
"XYZ"	C	"Wildcard" or Laboratory defined qualifier.
Qualifier specific entries		
E	Q	Entered if the reported value is estimated because of the presence of interferences.
Method qualifiers		
A	M	Flame atomic absorption
AS	M	Semi-automated spectrophotometric
AV	M	Automated cold vapor atomic absorption
C	M	Manual spectrophotometric
F	M	Furnace atomic absorption
MS	M	Mass spectrometry (ICP-MS)
NR	M	Analyte is not required to be analyzed
P	M	Inductively coupled plasma (ICP)
" "	M	No data have been entered

[1] The term "Column" is used to indicate under which column heading in the reporting forms that the qualifier will be found under.

Note: *Form I allows only one character in each qualifier column. If multiple qualifiers are applicable to column C, please assess qualifier priority in the following order: U, J, B. Reporting done in the EDD may include multiple qualifiers when applicable, separated by a single space.*

PART III -- CLP REPORTING FORMS AND INSTRUCTION GUIDE

1.0 NYSDEC has not created any specific reporting forms for the purpose of ASP reporting. Since most data is now reported using software formatted to produce data in the EPA CLP or EPA CLP-Like Forms, the ASP relies on the forms and instructions specified by the EPA in the CLP. Copies of the CLP SOWs, containing the required Organic and Inorganic Reporting forms and their instructions, can be found in ASP Exhibit D, in the CLP folder.

2.0 The Exhibit B forms and instructions contained in the CLP SOWs can be followed verbatim in most cases. Please note that the following exceptions and modifications to the CLP Forms and Form Instructions should be made.

2.1 Substitutions, General

- All references to "USEPA" or "EPA" should be substituted with "NYSDEC".
- All references to "EPA Sample Number" should be substituted with "NYSDEC Sample Number".
- All references to the "CLP SOW" or "SOW" should be substituted with "NYSDEC ASP" or "ASP", respectively.
- All references to "USEPA Regional Contract Laboratory Program Project Officer (CLP PO)", "USEPA OERR Analytical Operations/Data Quality Center (AOC)" and "Inorganic Program Manager (AOC PM)" should be substituted with "NYSDEC Bureau of Watershed Assessment and Management".
- The "Laboratory Code" to be used on all reporting documents should be the NYSDOH ELAP code assigned to the laboratory.

2.2 All references to the following can be disregarded:

- Non-Routine Analytical Services (NRAS)
- Sample Traffic Reports

2.3 The Forms and Instructions for Organic Data Reporting should follow CLP, Draft SOM01.X, Exhibit B with the following exceptions:

- References to "Modification Reference Number" or "Mod. Ref. Num." can be omitted or ignored in ASP reporting.

2.4 The Forms and Instructions for Inorganic Data Reporting should follow CLP, ILM05.3, Exhibit B, Section 3 with the following exceptions (All Section Numbers refer to directly to the CLP documents):

- The items under Section 3.3.5 may be disregarded.

- The requirement listed in Section 3.4.1.2.1 requiring the entry of the Statement of Work as “ILM05.3” should be modified and the label “ASP2004” should be inserted in the field for the SOW.

- Section 3.6 (CSF Instructions) may be disregarded.

PART IV -- NYSDEC DATA PACKAGE SUMMARY FORMS

The completion of Data Package Summary Forms is no longer a standard requirement for NYSDEC sample data or sample data packages. However for a small portion of NYSDEC Projects, completion of summary forms will be requested and required. These requests will be dependent upon the needs of the data users at NYSDEC. NYSDEC may also request changes in the style and content of the summary forms from those given herein.

The Data Package Summary Forms provided in this Exhibit are similar to the summary forms requested by NYSDEC in the past. If summary forms are requested and no specific template or blank forms have been provided to the laboratory, the following forms should be considered the default format. If custom forms are requested, the laboratory must report the summary data in the format requested. When summary data is requested in a non-standard format, the Laboratory should anticipate that the amount of information required in the summary forms would be similar to the amount of data required to complete the standard summary forms.

Instructions for NYSDEC Data Package Summary Forms

I. Sample Identification and Analytical Requirement Summary (Form S-I)

A. NYSDEC Sample ID/Code

Sample code number or ID assigned to the sample by NYSDEC personnel.

B. Laboratory Sample ID/Code

Code number given to respective sample by the laboratory and used for identification throughout analysis.

C. Analytical Requirements

This column is broken down into 6 sub-columns. The heading of each sub-column is an analytical parameter group. If the sample listed in a row is being analyzed for the parameter group listed at the top of the sub-column, complete the box below with the method number being used to analyze that sample for that parameter group. If no analysis is being performed in that parameter group, the space should be left blank.

II. Sample Preparation and Analysis Summary - Semivolatile (BNA), Volatile (VOA), and Pesticides/PCB's (Form S-IIa/b/c)

A. Laboratory Sample ID

The sample code number that the laboratory will use throughout the analysis for a specific sample.

B. Matrix

Label the sample with matrix indicated as water, soil, oil, grease, or drum solvent, etc.

C. Date Collected

Record the date that sample was collected on site.

D. Date Received at Laboratory

Record the date the Laboratory received the sample. (Validated Time of Sample Receipt - VTSR)

E. Date Extracted

Record the date the sample was extracted. This field should be left blank for aqueous VOA samples.

F. Date Analyzed

Record the date the sample was analyzed.

III. Sample Preparation and Analysis Summary – Miscellaneous Organics (Form S-III)

A. Laboratory Sample ID

The sample code number that the laboratory will use throughout analysis for a specific sample.

B. Matrix

Label the sample with matrix indicated as water, soil, oil, grease, or drum solvent, etc.

C. Analytical Protocol

Record the number of the method used to analyze the sample.

D. Extraction Method

Write the method used for sample extraction.

E. Auxiliary Clean-Up

If cleanup was done on sample, record the method or methods used.

F. Dil/Con Factor

If sample was diluted, record the final (just prior to analysis) dilution factor, or if concentrated, record also.

IV. Sample Preparation and Analysis Summary - Inorganics Analysis

A. Laboratory Sample ID

The sample code number that the laboratory will use throughout analysis for a specific sample.

B. Matrix

Label the sample with matrix indicated as water, soil, oil, grease, or drum solvent, etc.

C. Metals Requested

List metals that are to be analyzed. If for NYSDEC ASP, write full TCL in column, or more individual metals required.

C. Date Received at Laboratory

Record the date the Laboratory received the sample. (Validated Time of Sample Receipt - VTSR).

D. Date Digested

Date the sample was digested or otherwise prepared for analysis.

E. Date Analyzed

Date sample was analyzed on instrument.

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-I

SAMPLE IDENTIFICATION AND ANALYTICAL REQUIREMENT SUMMARY

NYSDEC Sample ID/Code	Laboratory Sample ID/Code	Analytical Requirements					
		VOA GC/MS (Method #)	BNA GC/MS (Method #)	VOA GC (Method #)	Pest PCBs (Method #)	Metals (Method #)	Other (Method #)

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-IIa

SAMPLE PREPARATION AND ANALYSIS SUMMARY
SEMIVOLATILE (BNA)
ANALYSES

Laboratory Sample ID	Matrix	Date Collected	Date Rec'd at Lab	Date Extracted	Date Analyzed

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-IIb

SAMPLE PREPARATION AND ANALYSIS SUMMARY
VOLATILE (VOA)
ANALYSES

Laboratory Sample ID	Matrix	Date Collected	Date Rec'd at Lab	Date Extracted	Date Analyzed

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-IIc

SAMPLE PREPARATION AND ANALYSIS SUMMARY
PESTICIDE/PCB
ANALYSES

Laboratory Sample ID	Matrix	Date Collected	Date Rec'd at Lab	Date Extracted	Date Analyzed

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-III

SAMPLE PREPARATION AND ANALYSIS SUMMARY
MISCELLANEOUS ORGANIC
ANALYSES

Laboratory Sample ID	Matrix	Analytical Protocol	Extraction Method	Auxiliary Cleanup	Dil/Conc Factor

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-IV

SAMPLE PREPARATION AND ANALYSIS SUMMARY
INORGANIC ANALYSES

Laboratory Sample ID	Matrix	Metals Requested	Date Rec'd at Lab	Date Digested	Date Analyzed

PART V – NYSDEC ACROBAT DOCUMNENT REQUIREMENTS

1.0 Sample Data Package .PDF File

In order to comply with the Paperless Office requirements being implemented by various New York State government organization, the Department of Environmental Conservation requires that all data packages be submited as Adobe Acrobat .PDF files on a CD-ROM. The following steps must be followed for the submission of Sample Data Packages and other related documents in .PDF format to insure that all data received by NYS DEC can be easily read, understood, and used for Department decision making.

1.1 CD-ROM Requirements

1.1.1 The CD-ROM containing the sample data package must be of the CD-R media type. Use of CD-RW media type is strictly prohibited for the submittal of NYSDEC Sample Data Packages.

1.1.2 The Laboratory is required to produce an additional copy of the Data Package CD-ROM submitted to NYSDEC and retain it for their records, stored for a minimum period of 3 years. This archive copy of the Sample Data Package and accociated SDG submitted files should be stored on CD-R type media. Use of CD-RW media is not permitted.

1.2 Sample Data Package Hardcopy Requirements

1.2.1 Generation of a hardcopy original Sample Data Package for storage at the Laboratory facility is no longer required.

1.2.1.1 Two (2) hardcopies of the SDG Cover Page and SDG Narrative from the Sample Data Package must be generated and signed by the appropriate Laboratory representative. One set of copies must be submitted to NYSDEC with the Sample Data Package CD-ROM. The second set of copies must be kept on file at the laboratory for a minimum period of 3 years from the date of sample receipt.

1.2.2 At the request of NYSDEC the lab should be prepared to generate a hardcopy of the full Sample Data Package, certifiy the newly generated hardcopy with the appropriate signatures, and submit the entire certified Sample Data Package to NYSDEC within 7 business days.

1.2.2.1 The associated computer files required to produce a hardcopy data package should be archived and stored at the laboratory for a minimum of 3 years from the date of sample receipt.

1.3 .PDF File Requirements

Sample Data Packages submitted to NYS DEC in .PDF file format should be of the "Formatted Text and Graphics" .PDF-type. Sample Data Package .PDFs should not be "Image Based" documents. This format allows .PDF documents to be searched for specific text strings within the data package. It also prevents poor integrity of original documents and poor scan qaulity from affecting the overall legibility of the data package.

1.3.1 File to .PDF Conversion – Whenever possible data packages should be constructed from instrument output files and report generator output files converted to .PDF format by processing the files through Adobe Acrobat Writer. When output files are converted into .PDF, the .PDF files created are searchable and the characters/fonts tend to be more legible. Care must be taken to insure that the fonts contained in output files are recognized by Acrobat and are properly converted. Converted files should also be checked to insure formatting (spacing, margins, etc.) and graphics are preserved from the original.

1.3.2 Hardcopy to .PDF Conversion - In some cases output files cannot be used and hard copy data must be scanned to create an image file (non-.PDF) and then converted into .PDF format. In these cases the integrity of the scanned document and the quality of the scan must be closely monitored to insure to overall legibility of the data package. The following requirements should be adhered to when creating .PDF files from hardcopy data.

1.3.2.1 The document should be scanned at 300 dpi or greater.

1.3.2.2 The document should be scanned at a speed slow enough not to distort the fonts or images in the rusultant image file.

1.3.2.3 NYS DEC requires that all scanned image files be processed through the Adobe Acrobat Capture Utility to convert the image file into a Formatted Text and Graphics .PDF. Whenever possible, original hardcopy documents should have no smaller than an 8 pt. font.

Note: *All text of 8 pt. size and greater, orientated along the horizontal axis of the page, should be recognizeable and convertable when processed through ScanSoft OmniPage or a similar Optical Charact Recognition (OCR) software engine. The OCR conversion should produce a .rtf document with an accuracy of 99% or greater when compared to the .PDF original. Text smaller than 8 pt. size or text not oriented along the horizontal axis of the document is not subjuect to the 99% accuracy requirement.*

1.3.3 Cropping of Pages - The pages in the .PDF file should be completely viewable to the reader, with a minimum margin width, on the left, right, top, and bottom of the document, of 0.5 inches when printed on a standard 8.5 by 11 inch piece of paper,. No part of an original image "page" shall be cropped in order to fit the document into a single .PDF "page". If necessary an original document may be proportionally reduced in size by 78%. If a document requires reduction greater than 78% in order to fit on a

single page, the document should be cafefully divided into equally sized parts and a .PDF page created for each part. An 8.5 by 14 inch legal sized document reduced by 78% will fit into a standard page by this requirement.

1.3.4 Page Orientation – Every effort should be made to have pages in the .PDF pages oriented in a consistant manner. NYS DEC prefers all pages to be in the portrait orientation when feasible. If the data system allows for the format of instrument output to be programmed between portrait and landscape, the output should be set to the portrait mode. If landscape is the only output mode possible, or in the case of the NYS Sample Summarry Forms, .PDF pages with landscape orientation should be inserted into the .PDF rotated counter-clockwise 90°. Landscape pages setup with this orientation would be displayed normally after a 90° clockwise rotation by the reader. If, due to the unprogrammable format of instrument data systems or report generation software, the majority of the pages are converted into .PDF in landscape orientation, they may remain in landscape orientation. If landscape is the majority orientation of the pages, portrait pages should be rotated counter-clockwise 90°, so that a clockwise rotation of 90° by the reader will orientate the image properly.

1.3.5 Linked Table of Contents – NYS DEC requuires that all Sample Data Packages include a Table of Content. The Table of Contents in the .PDF file should provide clickable links to the various sections and sub-sections listed in the Table.

1.3.6 Bookmarks – The Sample Dat Package shall contain bookmarks within the Adobe Acrobat file, arranged in the following manner:

1.3.6.1 The Sample Data Package .PDF should contain bookmarks to separate individual sections and the subsections within. All sections and subsections requiring bookmarks are marked in this Exhibit with a "**<B-*X*>**".

1.3.6.1.1 Sections marked with "**<B-1>**" should be bookmarked with a level one bookmark. Level one is the hightest level of bookmarking in the data package.

1.3.6.1.2 Sections marked with "**<B-2>**" should be bookmarked with a level two bookmark. Level two bookmarks are sub-bookmarkes to the parent level one bookmarks.

1.3.6.1.3 Sections marked with "**<B-3>**" should be bookmarked with a level three bookmark. Level three bookmarks are sub-bookmarkes to the parent level two bookmarks.

1.3.6.2 All items listed in the table of contents should be bookmarked within the .PDF and accessable from the bookmark navigation panel in Acrobat Reader.

1.3.6.3 Sample Data Packages should be further bookmarked when either one of the following conditions are met.

1.3.6.3.1 In cases when sample data exceeds more than 5 pages per sample data "packet", in either a "Sample Results" Section or a "Raw Data" Section, the beginning of each data "packet" must be bookmarked with the appropriate level bookmark **<B-(*X*+1)>**. Where *X* is the level of the parent bookmark for the Section in which the data is being placed in.

1.3.6.3.2 In cases when the total amount of data in any of the Sample Data Package sections designated for either a "Sample Results" or "Raw Data" exceeds 40 pages, the beginning of each data "packet" must be bookmarked with the appropriate level bookmark **<B-(*X*+1)>**. Where *X* is the level of the parent bookmark for the Section in which the data is being placed in.